"Semi-wet" unconventional electrolysis series

can

2018-11-23

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| Experiments performed under the common theme of providing ou |

Experiments performed under the common theme of providing only slight amounts of water between two electrodes instead of immersing them in water. The experiments listed here have been performed before acquiring KOH and K_2CO_3 electrolyte.

1. "Semi-wet" unconventional electrolysis of 2018-11-18

1.1. First round

Trying to replicate the first experiments I reported, which caused crackling noise emission from the cathode. They did not use electrodes immersed in water, but just water at the interface and an almost nonexisting electrode gap. They used to get very hot.

The basic procedure starts with the top electrode getting "dropped" on the anode. This allows for conduction to somehow ramp up gradually until a *cushion* composed of cavitating water, hydrogen and oxygen gases and steam prevents it from making direct contact with the bottom electrode and shorting out.

Since I need both hands to operate the device and that it needs constant adjusting, writing notes as the experiment proceeds will not be as easy as with other experiment types.

- <09:55> Starting to setup experiment
 - Magnet at the bottom of metal holder (a ferromagnetic wire stripper)
 - Anode (+) at the bottom
 - The initial idea behind this is that having the Cathode (-) at the top, the presumably active electrode, allowed for rapid inspection of the deposition layer formed
 - 5V will be applied
 - Gap will be formed by the oxide layer at the interface
 - Might take a while until it forms
 - Electrolyte will be used to add a temporary (soluble) low-conductivity barrier until proper conditions are set
- <10:05> Started with no electrolyte
 - Line voltage 5V
- <10:15> Red iron oxide layer forming on anode
- <10:23> Suddenly much easier to pass a current
 - AM radio noise also appeared
- <10:33> The process can be replicated
 - I keep the cathode moving or rotating slightly to avoid shorting as much as possible
- <10:40> Voltage drops down to 4.92V, then the electrodes eventually short
 - Water at the interface becomes foamy in the process
 - Electrodes become very difficult to separate, but apparently not (just?) because of welding
- <10:49> Experiment paused

- Mostly the anode appears to have been affected
 - Its surface turned rusty red, but for the most part on the edges that had a shorter gap relatively to each other
 - Reason being that the center part of the electrodes got eroded over time due to various processes
- No significant deposition observed on cathode, which is smooth and grayblack, seemingly oxide-free
 - No crackling noise heard nor expected because of this
- This testing round was performed differently than past experiments



Figure 1.1.1: Ferritic steel washers as electrodes; top cathode, bottom anode. No electrolyte used.



Figure 1.1.2: The electrode interface. When it was first disassembled about 90 minutes earlier, the cathode did not look oxidized like this, but more gray-black looking.

1.2. Second testing round

I plan to add electrolyte powder to the coin, then water and start with 12V, then eventually switch to 5V. This would better replicate what I used to do prior to acquiring the Geiger counter with this experiment type.

- <12:30> Cathode appears slightly rusted too, upon disassembing
- <12:32> Added slight amounts of Na₂CO₃ electrolyte
- <12:36> Current appears at 5V to be more intense
 - Voltage dropped to about 4.5V
 - Note that voltage is used as a proxy for PSU load here
 - For this reason I haven't applied 12V as it would have meant immediate PSU shutdown
- <12:37> Anode became cleaned of the previously formed oxides, while the cathode became black



Figure 1.2.1: Electrodes after using a Na_2CO_3 electrolyte solution. After a while of operation, the anode got partially cleaned from its surface red iron oxide layer and the cathode became dark black from presumably partially oxidized iron (for the most part).



Figure 1.2.2: Electrodes after a longer period of operation using an electrolyte solution instead of just tap water.

- <12:43> Changed anode plate to a large coin
- <12:54> I'm having problems avoiding shorting the electrodes out
- <13:00> After a few unsuccessful tries I flipped coin to the other side, which is smoother due to lack of arc discharge craters formed in previous experiments
 - Also added electrolyte and water



Figure 1.2.3: New electrode arrangement using a 100 Lire coin (SS430) as the anode.



Figure 1.2.4: Conditions of electrodes at the interface after a period of operation. All red iron oxides got removed, cathode became dark black. This arrangement did not prove to be reliable due to the ruined anode surface from previous testing.

- <13:02> I find it's working much better now and that I can apply a current for much longer periods of time and at a higher level
- <13:05> Reaction steady but messy
 - Red iron oxide splatters all around
 - Bubbling also intense
- <13:10> At this point I find I only need to add water through the top opening to keep the reaction going
 - No particular noise from the AM radio is noticed throughout this latter part of the experiment
- <13:12> Experiment manually terminated due to rusty mess and no further change observed
 - In retrospect I could have tried adding an electrolyte solution instead of plain (tap) water to check out for changes in the residue layer formed



Figure 1.2.5: New arrangement with flipped coin after a while of operation. Rust production was intense and bubbling caused the top part to also become partially covered with it. Electrolyte was only minimally initially added.



Figure 1.2.6: Both electrodes at the interface. The anode has been cleaned and eroded by the presumably intense cavitating action and gas formation. Red iron oxide is not sticking to the cathode, but the previously formed black oxide (?) layer has remained.



Figure 1.2.7: Electrodes after cleaning in tap water. It's clear that the anode got eroded during the previous process. The cathode got an impression of the anode marking on its surface, and the red iron oxide did not stick.

1.3. Conclusions and observations for the day

- I could replicate the previous black coating finding once I added Na₂CO₃, but didn't reproduce the coarse coating which originally showed a crackling noise (due to embrittlement?)
 - Adding electrolyte appears to be important for this. Without it, red iron oxide that does not seem to stick to the cathode gets produced instead
- When conditions and materials are right, there's only need to add water to keep the process going
- I'm assuming that under steady state conditions the gap between both electrodes is minimal and that electrical conduction occurs mostly through the wet iron oxide layer
 - The cathode got a partial impression of the coin anode
- Red iron oxide production intense today
 - I've never witnessed this in prior testing

- This is probably thanks to the favorable electrode configuration this time
- The anode got visibly eroded in the process
- Fe_2O_3 is often used as a dehydrogenation catalyst in petrochemical processes, even on its own
 - Having obtained it should be a good thing, in theory
- That so much iron is getting oxidized at the temperatures involved should mean that most of it is from the dissociated water rather than ambiental oxygen
 - This might imply that gas evolution is hydrogen-biased
- No clear changes in Geiger counts associated with the experiment have been observed
 - However the logging cart is still about 3 meters away from the testing area

2. "Semi-wet" unconventional electrolysis of 2018-11-19 (Day 2)

2.1. Experimental notes

- <09:40> Setting up the experiment again, as previously done
 - This time I'm using using a slightly lower strength hard disk Nd magnet on the bottom of the ferromagnetic support
 - I plan using Na₂CO₃ electrolyte solution
 - But I will start with plain tap water first
 - A 28mm diameter Italian 100 Lire coin will be the bottom anode (+), a stainless steel washer will be the top cathode (-)
 - Both presumably made of SS430 alloy
 - The top part of the washer has been sanded down with 1000 grit sandpaper to clean it up and to improve conductivity with the electrical wire
 - I plan to use 5V straight away
 - Keep in mind that from the photos the wire color is inverted (black for positive, yellow for negative)
- <09:57> Finished setting up the experiment



Figure 2.1.1: Newly set up experiment, similar to that performed the day before.

- <10:01> Line voltage: 5.00V
- <10:02> Added tiny amounts of electrolyte through the top opening
 - Electrical conduction almost immediately appears to be more intense
- <10:04> Line voltage: 4.88V
 - Because of gas production causing splatters when there is too much water, I'm trying to apply power impulsively
- <10:05> Added more electrolyte through the top opening
- <10:06> Aaded more electrolyte and water
- <10:09> The water seeping off the electrodes looks rusty
 - Line voltage: 4.76V under steady state conditions
 - Added more electrolyte
- <10:12> Added electrolyte in the water solution directly
- <10:17> Added more electrolyte in the solution
- <10:18> Inspected the cathode: it looks still black



Figure 2.1.2: Electrodes after inspection. At this point there are still relatively large amounts of hematite being produced.

- <10:21> Added more water after a continous period of operation
- <10:23> Allowed the interface to dry almost completely
 - Voltage eventually increased to 5.00V
- <10:24> Added more electrolyte in the solution
- <10:29> Allowed a period of continuous operation with several water refills through the top opening of the cathode
- <10:30> Inspected the cathode
 - Initially was deep black, then rather quickly turned reddish upon air exposure
 - Was this sodium ferrite? (NaFeO₂) Investigation required



Figure 2.1.3: The cathode quickly turned red after disassembling inspection. Sodium ferrite (NaFeO₂) production suspected.

- <10:31> Added more electrolyte in the solution
- <10:35> Allowed a period of continuous operation
 - Less hematite/rust getting produced than before, visually
 - This seems consistent with previous observations



Figure 2.1.4: Red iron oxide (hematite) production decreasing, electrodes kind of getting cleaned after increasing amount of electrolyte in the water solution. It still appears as if upon exposure the cathode turns more reddish.

- <10:37> Added more electrolyte in the solution
- <10:41> The electrode assembly at this point appears to be running slightly hotter than earlier
 - Also cleaner with significantly less hematite production than before
- <10:45> Experiment terminated
 - Will not wash the electrodes right away

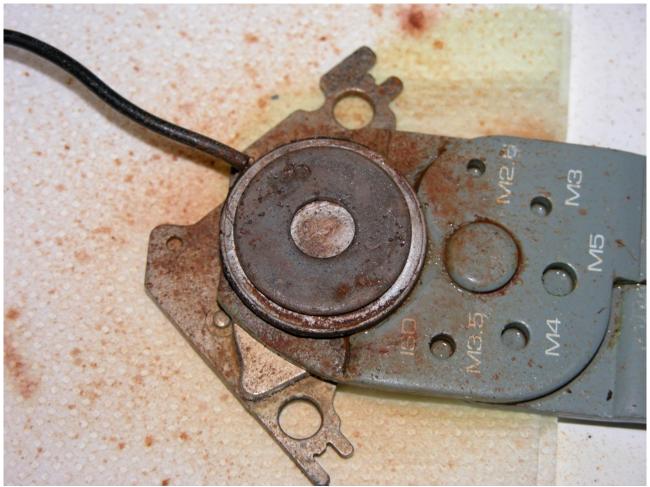


Figure 2.1.5: Electrodes after the experiment. Red iron oxide almost disappeared after adding progressively increasing amounts of electrolyte in the water solution.



Figure 2.1.6: Situation at the electrode interface. Similar observations as above.

2.2. Observations for the day

- All went smoothly, without many particular surprises
- The electrodes did not slide from each other, yet no serious short occurred
 - This might be thanks to the oxide layer formed, which is high friction and has a sufficiently high resistivity
 - It only occurred once that I had to reposition the cathode in order to restore a shorting condition. Rotating it on its place seemed sufficient
- I allowed water to evaporate on purpose
 - A strangely fine mist was visible at times getting emitted from the electrode assembly
 - If we are to assume that some special water form is getting produced or special hydrogen is getting entrained into it, it's possible that it could decay later on with emission of unusual radiation, causing an increase in background readings
 - However, this might not happen right away, but need some time first, perhaps 12-24 hours

- For the same reason I haven't washed thoroughly the materials used this time, to the extents of what was practical
- There's the possibility that sodium ferrite could be produced under these electrolytic conditions
 - Interesting if confirmed, as it would likely be a catalytically active compound similar to $KFeO_2$ in standard iron oxide-based industrial catalysts
 - I would expect this to be more stable than KFeO₂

2.3. Thoughts on radiation apparent base-level increase

Also posted <u>on LENR-Forum</u>.

Experiments (if so they can be defined) continue with [the equipment] I already have, which might possibly be sufficient to test the concepts and hypotheses presented. Earlier on I've written that I have a slight suspicion that certain testing might be increasing base-level background readings in my environment. Over the past two days of testing the "unconventional electrolysis" of this thread (rather than other experiments or other variations) I think I am seeing some changes overall; if they're actually due to the tests, they could imply the presence of something that is "decaying" after formation and not the direct emission of radiation from them.

3. "Semi-wet" unconventional electrolysis of 2018-11-20 (Day 3)

3.1. Experimental notes

- <09:45> Started setting up the experiment as usual
 - Previously used and now somewhat worn SS430 alloy coin and washer as electrodes (respectively anode and cathode)
 - Weaker hard disk Nd magnet of the two I have placed below ferromagnetic assembly
 - Planning to use 5V
 - Electrolyte solution prepared from previous day
 - Wire color still inverted
- <09:47> Immersed electrodes in water to wash them from excess carbonate and debris
- <09:49> Took electrodes out of water and dried them with paper towel
- <09:50> The surface of the cathode still looks black
 - It's not clear if this dark layer is still from the first usage period or if it got regenerated over time

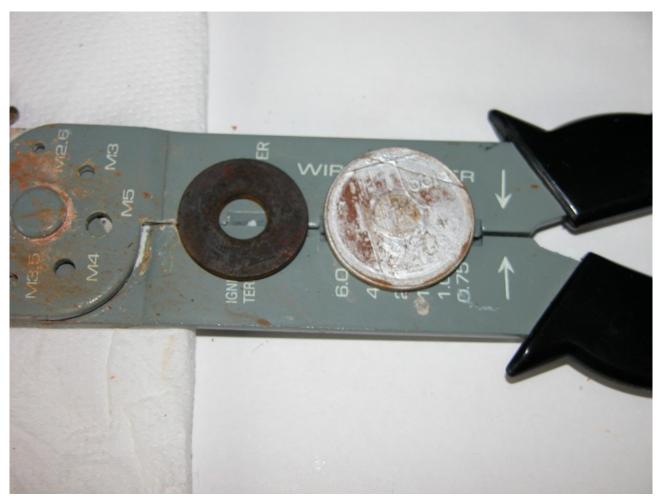


Figure 3.1.1: The active surface of the electrodes after washing in clean water and drying with a paper towel.



Figure 3.1.2: Experiment ready to start.

- <09:58> Finished setting up the experiment
 - Voltage: 5.17V
- <10:00> <u>Experiment started</u>
- <10:02> Voltage: 4.89V
 - Having some issues keeping the electrodes aligned and avoiding to short them out
- <10:04> Cathode slides around too much due to the applied current
 - Using a non-conductive support to hold it in place
- <10:07> Voltage: 4.82V
 - Sometimes I get brief shorts which cause visible arc discharges especially on the rim of the cathode
- <10:10> Allowed conduction until the electrodes got almost completely dried out of the water solution
- <10:15> Same as above, after refilling water from the top opening a few times
- <10:16> Inspected interface



Figure 3.1.3: Inner surfaces getting inspected.

- <10:17> Rotated cathode on its spot to mitigate shorting issues
- <10:20> Allowed conduction until completely dry
 - Inspected cathode afterwards
- <10:26> Continuous operation period
 - I find that with time the electrode assembly gets cleaner and less hematite is visible
 - Voltage: 4.70V (down peak)
- <10:27> Allowed the electrodes to dry up
- <10:35> Continuous operation period with water solution refills
 - Voltage: 4.64V (down peak)
 - Vibration from cavitation perceived from the cathode wire feels intense at times
- <10:37> Conducted electricity until the electrodes dried up
 - However, I find that drying them up completely is very difficult with this method



- A boiling sound continues at low level and keeps going, while voltage remains at $5.00\mathrm{V}$

Figure 3.1.4: The exposed electrode surface after attempting to dry up their inner surface (by Joule heating, essentially) does not look substantially different bar for more evident hematite formation.



Figure 3.1.5: Inside surface after drying.

- <10:40> Sandpapered electrodes onto each other before final run
 - Hematite is a ceramic material and is relatively hard (5.5-6.5 on the Mohs scale according to data <u>from Wikipedia</u>). The electrodes did feel grainy when slid onto each other
 - Hopefully this will lead to less shorting events



Figure 3.1.6: Inner electrode surface after dry abrasion.

- <10:43> Final test started
- <10:46> Shorting event
- <10:48> Current passed until electrodes dried up
 - They got cleaned somewhat in the process
 - Voltage: 4.76V (down peak), then up to 5.00V
- <10:49> <u>Experiment terminated</u>
 - I will leave it in place as it is until after lunch
 - Will avoid cleaning materials too much in order to test the hypothesis where dense hydrogen or other unusual molecules are getting ab/adsorbed in the materials and decaying later on



Figure 3.1.7: Electrode assembly after the experiment was terminated. Except for splatters around the testing area (which might be a problem with hydroxides) the electrodes themselves seem relatively clean compared to the first experiments in this series where I only used tap water.

3.2. Observations for the day

- I find that the experiment goes more smoothly when the electrodes dry up at the interface
 - Shorting events after this seem less frequent
 - The electrodes don't slide around as much
 - All of this probably due to the oxide layer formed
- As the experiment tends to produce splatters all around, this could be a problem with KOH, which is caustic
- In retrospect, this method of continuously introducing electrolyte solution from the top opening might not be ideal
 - On the long term the concentration of salts at the interface and on the electrodes in general will tend to increase as water evaporates and more water is added



Figure 3.2.1: White sodium carbonates on the top cathode. Photo inadvertently blurred.

- Some time after I performed the experiment I noticed that reflective speckles appeared on the surface of the cathode
 - This is similar to what I used to observe in early experiments where crackling sounds occurred after electrolysis
 - It might imply that this kind of surface starts appearing only after the concentration of alkali salts on the cathode is high enough



Figure 3.2.2: Reflective speckles barely visible on the inner surface of the cathode after disassembling.



Figure 3.2.3: Some time after the experiment was terminated. This short image sequence shows that there are tiny bits which are reflecting light randomly.



Figure 3.2.4: Second and last image in the series.

4. "Semi-wet" unconventional electrolysis of 2018-11-21 (Day 4)

4.1. Experimental notes

Today's testing will use much less sodium carbonate electrolyte (if any at all) to test the hypothesis where for the actually desired anomalous effects (increased background radiation) too much is actually harmful. My expectation is that more Fe_2O_3 (hematite) will be produced as a result, with the experiment being messier in general. Hematite on its own is regarded as being mildly catalytic in dehydrogenation reactions and when calcined at low temperatures it can have a specific surface as high as 160 g/cm² before sintering occurs¹. Furthermore, its presence implies that oxygen is gettered in the process.

- <09:05> Started setting up the experiment.
 - The actual experiment will start later in the morning
- 1 Emerson H. Lee (1974) Iron Oxide Catalysts for Dehydrogenation of Ethylbenzene in the Presence of Steam, Catalysis Reviews, 8:1, 285-305, DOI: <u>10.1080/01614947408071864</u>

- The usual ferritic (thus ferromagnetic) steel 100 Lire coin and steel washer used respectively as anode and cathode.
 - Yesterday the surface deposition layer at the interface of both electrodes was partially abraded after the testing, so conditions are different today
- The electrodes are washed in clean warm water and then placed onto the ferromagnetic holder (a repurposed wire stripper)
- Using the slightly stronger hard disk Nd magnets of the two I have



Figure 4.1.1: The electrodes after washing in clean warm water.



Figure 4.1.2: Electrodes in place and experiment ready to start.

- <09:45> Finished setting up the experiment
 - The plan is adding electrolyte initially, then will continue with plain tap water
- <09:48> Voltage: 5.15V
- <09:52> <u>Experiment started</u>
 - Having very hard time making proper conduction: short circuiting occurs
- <09:54> Attempting rotating the cathode, adding electrolyte and various ways of dropping it onto the anode
- <09:59> After finally obtaining normal conduction the experiment manages to actually start
 - Hematite (red iron oxide) immediately produced
 - Slow mist (steam?) rising from the cathode
 - Allowed conduction until almost complete dryness
 - But couldn't dry the interface completely as a fizzling sound keeps continuing
 - Voltage: 4.76V (peak), then up to 5.00V with dried electrodes



Figure 4.1.3: Hematite formed once the electrode started conducting a current as intended.



Figure 4.1.4: Exposed surface of cathode manually cleaned to achieve better conduction with the corresponding wire and avoid shorts.

- <10:02> <u>Experiment paused</u>
 - Had other duties to perform, not related with the experiment
- <10:11> <u>Experiment resumed</u>
 - This time I only added plain water
 - To achieve a better conduction I cleaned the top (exposed) surface of the cathode
- <10:14> I find that the AM radio is unusually noisy
- <10:15> With a hematite layer in place there seem to be no problems starting and maintaining the experiment going
 - Voltage: 4.90V
- <10:17> Allowed the electrode interface to dry up, then added water
 - Voltage: 4.86V (peak), then eventually 5.00V
- <10:21> Allowed the electrode interface to dry up again
 - Voltgae: 4.86V rising to 5.00V at the end of the run

- <10:22> Added electrolyte solution
- <10:28> Conducted electricity for long continuous time, refilling water as needed to maintain voltage around 4.90V
 - The cathode slid around slightly, but this did not seem to be an issue

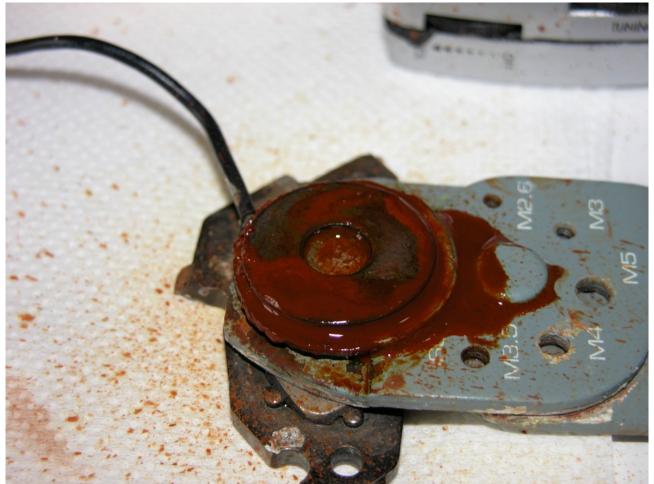


Figure 4.1.5: After a period of operation the cathode ended up sliding slightly from its centered position.

- <10:30> Cathode centered again on the anode
 - AM radio seems noisy
- <10:31> Added water and kept going on
 - Voltage: 4.97V, seems a bit low
- <10:35> Water is present but conduction feels difficult
 - Added electrolyte solution
- <10:36> Conduction visibly higher after adding electrolyte
 - Voltage: 4.80V, rising to 4.92V
- <10:39> AM radio is making fuzzy noises, but there does not seem to be any problem conducting electricity

- Voltage seemed to jump a bit, however
- Strange noise stopped after a while
- <10:43> Allowed conduction until dryness
 - This means reaching 5.00V
 - Even after keeping conduction alive, still not 100% dry
- <10:44> <u>Experiment terminated</u>
 - Will keep the setup untouched for a while and avoid excessive cleaning, but I will empty plastic container with tap water



• The container should not make any difference

Figure 4.1.6: The electrodes at the end of the experiment. No significant visual change to note.

4.2. Reheat test

About one hour later, is conduction through the dried electrodes still possible? Could they be heated this way? Let's find out.



Figure 4.2.1: One hour after the test the cathode looks dry on the surface

- <11:44> Tried applying 5V
 - Voltage: from 5.15V (no load) to 5.14V (load)
 - No apparent effect
- <11:46> Tried applying 12V
 - Voltage: from 11.7V (no load) to 11.69V (load)
 - No apparent effect
- <11:51> Tried 5V to ferromagnetic holder
 - No useful heating could be obtained
 - Mostly short circuiting
- <11:58> Electrode interface inspected
 - It seems there is only dried hematite
 - I left the entire assembly in one place afterwards



Figure 4.2.2: Upon inspection, the electrode interface also appears to be composed of dried material, mainly hematite. I have not separated the electrodes during the experiment.

4.3. Observations for the first part of the day

- During today's experiment I've never separated the electrodes except at the beginning when I was having problems achieving proper conduction
- The experiment went much better, thanks to to hematite layer formed which prevented a full short and increased friction between both plates
 - The cathode was not completely fixed in place or in any way bonded with the anode
- I have the impression that background gamma emissions have remained elevated longer than usual
 - At the time of the test I get a decrease while today they remained at the same level
 - To be fair, this could be coincidental
- The strategy of introducing electrolyte solution only when needed appears to work better in general both on theoretical and practical grounds

- It can also be argued that the formation of a hematite layer should be desirable
- Sodium carbonate should still exist in some form at the interface between both cathodes and inside the porous hematite layer formed
- At some point the AM radio became constantly noisier than it was initially, even with the cell at rest and no power applied, but it's not clear if this is directly due to the experiment or external interference
- This time I did not completely disassemble the setup after usage
 - The Nd magnet is also still in place

4.4. Extra test: higher temperatures at 12V

I am curious to check out what will happen once I start applying 12V when electrode resistivity increases high enough. The formation of a hematite layer should make this easier to test without annoying short circuits. I will first with 5V and include only water through the pre-existing oxide layer which as of now has dried up.

An obvious expectation basing on past tests is that the electrodes will dry up quicker.

- <15:05> Setup resettled
- <15:18> <u>Experiment started</u>
 - Tiny amounts of water introduced on the top opening
- <15:20> Seems difficult to conduct
 - Voltage: 4.97V (under load)
 - More water added
- <15:21> Switched to 12V
 - Voltage: 11.72v (no load)
- <15:22> The reaction appears violent
 - Voltage drops below 10V
- <15:23> I kept applying a current to complete dryness
 - Settled at 11.65V (load)
 - Portions of the anode got cleaned
 - This is where intense evaporation and electrolysis occurred
- <15:24> Refilled some water again and started applying 12V
- <15:25> Voltage drops below 10V
 - Water evaporates very quickly
 - Kept applying until dryness achieved
- <15:27> I noticed that when added in the opening, water diffuses to the top of the cathode by capillarity through the hematite deposits
 - Does not appear to be a severe problem

- <15:28> Started applying again 12V
- <15:33> Added water several times until eventually dryness was achieved
 - Fine mist quickly produced
- <15:35> Electrolyte solution added
 - Once power was applied, it dried up much quicker but there didn't seem to be a corresponding increase in PSU load
 - However this is just a subjective impression
- <15:36> Added water only
 - The effects of previous electrolyte application are still present
- <15:39> Added water several times to dryeness
- <15:40> Added some electrolyte and let the cell to soak for a minute
 - It seems that it is getting absorbed, and after a while It looks less wet than just after adding it
- <15:45> Applied 12V to the assembly several times to dryness
 - After wards I added again some electrolyte
- <15:48> After making it reach dryness from the heat several time, I think I've seen enough for the day
 - I have acquired a slight headache
 - But it could simply be CO from the improperly baked Na_2CO_3 electrolyte in the solution
- <15:49> <u>Experiment terminated</u>
 - I will briefly separate the electrodes for a photo first
 - Leaving the device assembled afterwards like I did earlier



Figure 4.4.1: State of the electrodes after the experiment, assembled. Significant hematite deposits and splatters all around. The cathode does not slide easily over the anode.



Figure 4.4.2: Electrodes disassembled. Some larger hematite pieces got stuck to the cathode presumably when I separated it from the anode.

4.5. Observations for the second part of the day

- With 12V heat is much more intense
 - This was only partially expected
 - It's only only possible to apply 12V after an oxide layer has formed
 - No short-circuit events have occurred, thanks to this layer
- I allowed significant amounts of water to evaporate in the testing environment
 - If the entrainment of unusual hydrogen or water molecule hypothesis is true, following this I should eventually observe an appreciable (not necessarily large, but visible) increase in background radiation, as such molecules eventually decay.
 - The decay hypothesis is based on the behavior of Holmlid's ultra-dense hydrogen
- I might have left out minor observations along the way. At some point the radio started emitting noises as if conduction was not perfect.

- Not clear if at the interface or at the cable (a possibility due to hematite deposits)
- No direct increase in gamma radiation from the Geiger counter appear to have occurred during the experiment.
 - The counter is still located about 3 meters away from the testing area because I have several weeks worth of recorded data from that position

5. Low-temperature heating test of 2018-11-21 | 22

5.1. 2018-11-21

In the evening I placed the assembly (not significantly altered since the unconventional electrolysis test performed in the morning) on an improvised USB heater to slowly increase temperatures above ambient, up to roughly 40-50°C. I will keep it like this until the next day.



Figure 5.1.1: The improvised USB heater with an Aluminium IR reflector.

- <17:31> Placed assembly on USB heater
- <19:10> Rotated electrodes on top of each other, abrading slightly their surface and redistributing the pulverized hematite

- <19:21> Added magnet pile on one side of the cathode
- <19:57> Added Al hood (improvised IR reflector)
- <20:25> Removed large magnet from assembly
 - A magnetic field is still present from the pile of small magnet, but more limited in extent
- <20:35> Improved reflector hood
 - Made it flatter with a wooden roller
- <21:10> Removed temporarily assembly from heater
- <21:23> Put assembly back into low temperature heater
- <23:31> Removed temporarily assembly from heater
 - Does not seem to be significantly above ambient temperature
 - Somehow I get a strange cough feeling when I am around it
 - Could be a psychosomatic reaction
- <23:33> Put a drop of electrolyte solution into the top cathode opening and let it soak
 - Solution getting absorbed, making tiny cracking noises in the process
- <23:36> Put another drop of electrolyte solution
 - Getting absorbed again
 - I find it's reaching nearby parts by capillarity
- <23:38> Put another drop
 - Even though it seems to be taking more, I will make this the last one
- <23:40> Put assembly back into low temperature heater

5.2. 2018-11-22

- <00:00> Temporarily removed assembly from heater for inspection
 - It appears to have partially dried, but hematite still looks like wet mud
 - Put assembly back into heater afterwards
- <00:59> Assembly inspected
 - Hematite layer drying up
 - Heater feels hot
- <05:20> Temporarily removed assembly from heater
 - Seems mostly dried up
- <06:17> Put assembly back into heater
- <08:53> Power removed
- <10:30> Removed assembly from the cold heater

- Added deionized water until saturation through the top opening
- <10:32> Put assembly back into the heater
 - The heater was turned on just earlier on and will slowly heat up
- <11:16> Heater warm
 - The electrode assembly appears to be on the way of drying, but still looks wet
- <12:15> Power removed from heater
 - <u>Experiment terminated</u>

6. "Semi-wet" unconventional electrolysis of 2018-11-22 (Day 5)

6.1. Experimental notes

- <12:15> Started setting up the experiment
 - Will use mostly electrolyte solution
 - Initially keeping inner rust in place
 - But cleaning top cathode surface for conductivity
 - Also added added graphite powder from HB pencil
 - Then distributed it by applying pressure on the cathode and rotating it



Figure 6.1.1: New experiment in place, with abundant hematite already present. The top of the anode was cleaned with water and a paper towel.



Figure 6.1.2: Graphite powder added, before being crushed and redistributed between both electrodes.

- <12:42> <u>Experiment started</u>
- <12:43> Starting with 5V
- <12:46> Conduction seems limited with 4.86V observed
- <12:47> Added water
- <12:48> Applied current until dryness
 - Top cathode now dark
- <12:49> Added electrolyte solution
- <12:52> Conduction visibly stronger now
 - Voltage: 4.65V (peak)
- <12:54> Applied current to dryness
- <12:55> Added electrolyte
 - Also switched to 12V
 - Voltage: 11.69V (no load)
- <12:56> Voltage: 9.70V (load)

- Applied to dryness, quickly achieved
- <12:57> Added electrolyte
 - Short circuit event causing the PSU to turn off
 - Too much current probably
- <12:58> Switched to 5V
 - Conduction seems fine
- <13:06> Applied current and added electrolyte continuously with 5V
 - Voltage: 4.70-4.68V (peak)
 - Getting cleaned, not much red iron oxide (hematite) produced anymore



Figure 6.1.3: Hematite from the electrodes slowly disappearing.



Figure 6.1.4: The electrodes at the interface upon inspection show the typical conditions of the anode getting cleaned (presumably by the evolved gases and steam/cavitation) and the cathode turning dark.



Figure 6.1.5: Cathode after cleaning again its top surface.

- <13:09> Rotated cathode and cleaned its top surface
- <13:10> Added electrolyte
- <13:16> Kept adding electrolyte while applying 5V
 - Initially hematite was produced prod
 - Then it got cleaner, with the area on the rim of the cathode getting clear
 - Refilled with electrolyte solution several times, then to dryness
- <13:17> It seems that waiting or exposing the cathode's inner surface to air causes the assembly to produce hematite in the initial stages of power application



Figure 6.1.6: Electrodes inspected again after a continuous period of operation.

- <13:24> Added graphite
 - Crushed and distributed it by applying pressure and rotating the electrode
 - Added electrolyte
- <13:25> Applied 5V
 - Dark bubble production
 - Beware of them as they can be explosive
 - Applying power impulsively to limit their production
- <13:26> Having some issues preventing shorting events
 - After rotating the cathode and applying current a few times continuously disregarding the black bubbles it looks like now it's working well
- <13:29> Applied 5V and refilled electrolyte several times in the process
 - Voltgae: 4.55V (peak)
 - Evolved particles initially black
 - Then, hematite started getting produced
 - Then, the solution got clearer

- <13:30> Added electrolyte
 - Short-circuit event after a while
 - I think I've seen enough for today
- <13:31> <u>Experiment terminated</u>
- <13:49> Put assembly onto low temperature USB heater to dry it

6.2. Evening notes

Eventually I washed coins and materials. Will continue these *unconventional electrolysis* experiments when I will have KOH and K_2CO_3 to use as electrolyte.

7. Quick Cu-Ni-Zn-Al-Sn test on 2018-11-22 | 23

7.1. 2018-11-22

For this quick test unfortunately I did not take photos or notes as I previously did

In the evening of 2018-11-22 I tried a quick test with a 2 Euro coin that had been sanded down as to obtain a smooth surface (material: <u>Nickel Silver</u>) as the bottom cathode (-) and a 10 Eurocent coin as the top anode (+), also sanded down (material: <u>Nordic Gold</u>).

Unfortunately I could not achieve good conduction and the current passed at 5V through the coins was generally low. Furthermore the lack of a top opening made refilling water not straightforward.

Another problem was that the 2 Euro coin does not show significant ferromagnetism, which complicated holding firmly together the entire assembly. Initially I tried using a top cathode composed of the usual ferritic steel washed, but I had the opposite problem (excessive attraction to the magnet) which would have made annoying shorting events a certainty.

Nickel Silver is supposed to have a composition of nominally 60% Cu, 20% Ni, 20% Zn, while Nordic Gold nominally 89% Cu, 5% Al, 5% Zn, 1% Sn. Excluding impurities at least different 5 transition metal elements are present in total.

For the most part I used the usual rather impure saturated sodium carbonate solution (exact molarity unknown). After passing a current the bottom cathode turned black, while the top anode acquired a green tinge from presumably copper.

At the end of the short test the coins were placed onto a USB device repurposed as a low temperature heater for drying. I once added a drop of electrolyte solution at the center of their interface.



Figure 7.1.1: The coins the day after before separation and after prolonged heating at low temperature with electrolyte solution inbetween.



Figure 7.1.2: Electrode interface inspected.

7.2. 2018-11-23

- <11:48> When I tried to separate the pieces for a couple photos, they appeared to be stuck onto each other. I supposed that galvanic corrosion might have occurred and cause them to bond together
- <11:51> Seeing that possibly interesting effects might have happened, I added another drop of saturated electrolyte on the coin interface and placed them on the low-temperature USB heater
 - The heater will be slowly increase its temperature to somewhat above ambient