### DOI 10.14622/Advances\_48\_2022\_14

### Printable electrolyte for printed batteries

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#### Short abstract

In this study, we focus on optimising the electrolyte layer of printed batteries. In the stack design of a printed battery, the electrolyte layer is located between the anode and cathode and is responsible for ion transport. However, the anode and cathode must not touch each other, so a material called a separator is usually used. This separator is an absorbent porous fleece-like material that is usually drenched with electrolyte. In the zinc/ manganese dioxide electrochemical system, the electrolyte is a salt dissolved in water, usually a zinc chloride solution. The solution is almost as liquid as water and therefore not printable in screen printing. In this research we used thickeners (gelling agents) to achieve printable fluid flow properties, added small rigid particles as spacers to the solution to get rid of the unnecessary fleece, and finally experimented with a mixture of ammonium chloride and zinc chloride to enhance the battery performance.

Keywords: printed batteries, screen printing, printable electrolyte, thickeners, spacers

### 1. Introduction and background

For a period of more than 15 years the research group Innovative Applications of the Printing Technologies (IAD) is working together the German battery manufacturer VARTA Microbattery GmbH. During this period very many different electrochemical systems have been investigated. However, the primary (non-rechargeable) system zinc/manganese dioxide aka zinc/carbon system is still of central importance. In the governmentally funded project OxiFlexIT (2022) this type of film batteries are employed for flexible skin patches to monitor physiological data such as blood oxygen levels. The batteries are used to drive the measurement setup and power broadcasting of data to a remote-control station. For hygienic reasons and ease of use, the batteries do not need to be rechargeable. However, the printed batteries need to be made as thin as possible to be flexible (total thickness <<1 mm). A rigid button cell (the competition) is markedly disadvantageous. Furthermore, there are serious indications and signs from industry partners that printed batteries based on this electrochemical system will be available on the mass market very soon. Therefore, the research shown here contributes significantly to improving the performance and manufacturability of thin film printed batteries. A quite similar project to OxiFlexIT, the EU funded research action called "BEWELL", also employs printed batteries (BEWELL, 2022). In a first version the same primary battery system was used and later expanded to rechargeable ones based on Li-ion technology.

The chemical systems zinc/ manganese dioxide, the printing technique, and mechanical properties used for these film batteries has been described earlier (Hübner, et.al., 2015; 2021). The design used here is the so-called stack design as shown in Figure 1.



Figure 1: Basic design of the so-called stack design of printed batteries (Hübner, 2015)

The printing process that is the most suitable for this type of application is screen printing since the mass (layer thicknesses typically ~150  $\mu$ m) of the active electrode materials determine the battery capacity and the particles used are quite coarse (10  $\mu$ m to 25  $\mu$ m). Nevertheless, with a total thickness of the printed cell of about 600  $\mu$ m it remains bendable and can snuggle to the human skin.

In comparison to the alternative co-planar design, the stack can deliver much higher peak (burst) currents. These load peaks occur in the use case because high power demand for the measurement process and the transmission of the collected data regularly occurs in the use cycle after idle periods with low power consumption. The challenge of the stack design is that a direct contact between the anode and cathode must be avoided and therefore a separator is used. In classical setup, this separator is an absorbent porous fleece-like material that is usually drenched with electrolyte. For the zinc/manganese dioxide electrochemical system that is used here, the electrolyte is a salt dissolved in water, usually a zinc chloride solution. The solution is almost as liquid as water and therefore not printable in screen printing. In this research we used thickeners (gelling agents) to achieve printable fluid flow properties, added small rigid particles as spacers to the solution to get rid of the unnecessary fleece, and finally experimented with a mixture of ammonium chloride and zinc chloride.

The conventional battery is an electrochemical cell, in which redox reactions take place. In the  $Zn/MnO_2$ -cell, the following simplified chemical reactions take place (Wikipedia, 2019):

at anode: 
$$\text{Zn} \rightarrow \text{Zn}^{2+} + 2e^-$$
 and at cathode:  $2\text{MnO}_2 + 2\text{H}_2\text{O} + 2e^- \rightarrow 2\text{MnO}(\text{OH}) + 2\text{OH}^-$  [1]

The nature of the electrolyte plays an important role in the development of the zinc/manganese dioxide cells (Wikipedia, 2022). In early times liquid ammonium chloride was used in the Leclanché cell, then in a thickened, immobilized form, the cell was called zinc-carbon cell. Later, zinc chloride was used, and the best performance shows the variant with a KOH-based electrolyte, which is called an alkaline battery. Here, the authors used either pure zinc chloride or a mixture of ammonium and zinc chloride. In other projects, a lot of experience was gained with KOH-based electrolytes, which showed that this solution creeps extremely and even attacks the seals. Therefore, the KOH-based types were not investigated here, because the usage will be on skin. Since the electrolyte must act as an ion transport system, it has to be kept moist during the whole lifetime of the battery. To prevent the battery from drying out, the cell must be tightly sealed. It is even advisable not to use substrates made of a single material through which water vapour can easily migrate, but to use composite films that enclose a barrier layer such as aluminium.

As the batteries are intended to become a mass product, some considerations about pricing and sustainability are appropriate. As discussed earlier elsewhere (Hübner, et al. 2021) the substrate plays an important role. If the tendency to dry out can be reduced by using a more hygroscopic electrolyte it could be possible to leave out the barrier layer in the substrate. Mixtures of ammonium and zinc chloride reduce the tendency to dry out. Using mono-material foils could also be helpful regarding recyclability and in the present composition there are hardly any environmentally critical materials.

If the separator (fleece-type material), which has to be inserted into the battery either manually or by pick and place, can be avoided, a big step towards simple inline manufacturing has been taken. A printable electrolyte fulfilling the separator function by itself is a big cost saving factor.

### 2. Materials and methods

Measuring instruments and techniques are described below.

# 2.1 Rheology

A Physica MCR 300 rheometer was used to evaluate the flow behaviour of the printable electrolyte paste formulations. Typically, the pastes show a pronounced shear thinning behaviour. To compare different formulations, the viscosity value was recorded at a shear rate of 50 or 100 reciprocal seconds (s<sup>-1</sup>). Because of the particles of up to 50  $\mu$ m introduced into the pastes, a parallel plate geometry was used. The more accurate cone and plate geometry could not be used because the surfaces could be damaged by the abrasive effect of the particles in the small gap in the middle.

# 2.2 Conductivity and GEIS measurements

One of the performance-determining factors of batteries is the internal resistance. Obviously, the smaller the internal resistance of the ion transport system, the better the battery performance will be. Therefore, it is very important to determine the resistance – or better the impedance – of the electrolyte solution. The conductivity as the reciprocal of the impedance of the electrolyte solutions was measured with a setup shown in Figure 2.



Figure 2: Conductivity measurement set-up

Two metal electrodes are immersed in the electrolyte, with the distance between the electrodes and the immersion depth being kept constant across all measurements. Nickel strips with a high corrosion resistance were used as electrodes and replaced after several measurements. The electrodes are connected by crocodile clips to the measurement device, a Bio-Logic SP-300 potentiostat (Biologic, 2022).

When the electrodes are immersed in the electrolyte, metal ions dissolve, leaving free electrons behind. This leads to the formation of an electrochemical double layer at the electrode/electrolyte phase boundary, which acts like a capacitor with capacitance *C*. The capacitor causes a frequency-dependent behaviour of the system. This is the reason why the conductivity of the electrolyte cannot be determined using a direct current (DC) measurement but must be determined using an alternating current (AC) impedance measurement. Figure 3 shows the (simplified) equivalent circuit diagram of the system. Detailed information on the corresponding measurement techniques and theory can be found in Knoblauch (2015), Gamry (2009), and Biologic (2022). The resistance parallel to the capacitor is called the polarization resistance  $R_{\rm p}$  (Gamry, 2009). The resistance of the electrolyte which we are interested in is represented as resistance  $R_{\rm E}$ . Both  $R_{\rm p}$  and  $R_{\rm p}$  can be regarded as frequency independent ohmic resistances.



Figure 3: Equivalent circuit diagram

The capacitor C is characterised by the capacitive reactance  $X_{c}$  shown in Equation [2].

$$X_{\rm C} = -\frac{1}{2\pi fC}$$
[2]

To determine the  $R_{\rm E}$  value, the impedance of the system is measured with a sinusoidal AC excitation at variable frequencies using the Galvanostatic Electrochemical Impedance Spectroscopy (GEIS) method (Biologic, 2022). In this work the GEIS measurements were performed with an alternating current of 200 µA amplitude and a frequency range from 1 Hz to 1 MHz.

Figure 4 shows an (idealized) result of a GEIS measurement of a system according to Figure 3 in the form of a Nyquist plot. The ohmic resistances  $R_{\rm E}$  and  $R_{\rm p}$  appear in the diagram at the points where the semicircle touches the *x*-axis, and the imaginary part of the impedance is 0. At those points the capacitive reactance is 0 (for  $f \rightarrow \infty$ ) or approaching infinity (for  $f \rightarrow 0$ ).



Figure 4: Sample Nyquist plot with impedance vector for a circuit according to Figure 3

To determine the conductivity of the electrolyte, the resistance  $R_{\rm E}$  was taken from the Nyquist diagram.

### 2.3 Measurement conditions of the cell set-up

To calculate the conductivity, the cell constant *K* of the measurement set-up must be known. The cell constant *K* is primarily dependent on the geometric arrangement and can be determined using the procedure described above with a calibration solution with a known specific conductivity  $\kappa_{ref}$  in S cm<sup>-1</sup>.

$$K = R_{\rm E} \kappa_{\rm ref}$$
[3]

After the cell constant is evaluated, the unknown specific conductivity of the electrolyte can be calculated from the measured impedance by using the same rearranged formula:

$$\kappa_{\text{sample}} = K / R_{\text{Esample}}$$
<sup>[4]</sup>

where *K* is the value obtained by the calibration and  $R_{Esample}$  is the measured resistance value.

Since this measurement of the specific conductivity depends very much on the determination of the cell constant and other parameters the accuracy can be estimated at  $\pm 2 \text{ mS cm}^{-1}$ .

### 2.4 Electrical characterisation of batteries

For characterization of the battery properties, the performance of the battery is characterized by the voltage U times the current I. The unit of electrical power P is Watt (W). The energy is power multiplied by the time the power is delivered, t.

$$E = P \cdot t = U \cdot I \cdot t \tag{5}$$

Another important characteristic is the energy capacity Q of the battery. Battery capacity is defined as the total amount of electricity generated due to electrochemical reactions in the battery and is expressed in ampere times hours. For example, if a battery has a capacity Q = 50 mAh, then a constant discharge current of 50 mA can be drawn out of the battery for 1 hour.

$$Q = I \cdot t \tag{6}$$

### 2.5 Printable electrolyte with separator function

In comprehensive preliminary experiments several thickener agents were tested to bring the zinc-chloride solution in a printable viscosity and appropriate flow behaviour range. It turned out that the best and most stable results could be achieved by using Agar-Agar. Agar-Agar belongs to the category of common gelling agents and is composed by 70 % agarose and 30 % agaropectin and it is extracted from the cell walls of different red algae (CarlRoth, 2022).

Agar-Agar should solely function as a thickener but should not play any performance influencing role in a battery. Thus, we used a laboratory grade. Maybe for a later mass production also other grades can be used.

Here, the highly pure BioScience Agar-Agar from vendor Carl Roth has been used. It is often employed in molecular- and microbiology.

### 2.5.1 Usage of spacers

Although the printable electrolyte shows quite high viscosity and thus high resistance to flow it does not have the capability to permanently separate anode from cathode. The idea adopted to overcome this problem is the addition of spacers into the solution. Just alike the usage of powder in the delivery of offset presses little bead-like particles should fulfil the purpose of spacing. Indeed, the first test was done with the starch-based particles used in the offset press. However, it turned out that these materials dissolve in the electrolytes and were not applicable.

Three different materials could be found from which appropriate beads could be sourced

- Phenolic resin Figure 5
- Glass
- PMMA (Poly methyl methacrylate) Figure 6

The phenolic resin beads that were found are hollow phenolic microspheres. Normally they are used as the performance enhancers for grinding wheels and are used as a filler material (Kremer, 2022). Their size ranges from 5  $\mu$ m up to 127  $\mu$ m and their relative density from 0.20 gcm<sup>-3</sup> to 0.80 gcm<sup>-3</sup>. They are an oligomeric reaction product of formaldehyde with phenol (CAS No. 9003-35-4; EINECS 2000052), purity  $\geq$  99.8 %. They are a red-brown granulate (Figure 5) provided by the company Kremer Pigmente GmbH & Co.



Figure 5: Phenol beads



Figure 6: PMMA beads

The glass beads that we found on the market for sandblasting did not have spherical shape. They showed irregularities and a wide distribution of particle sizes. Almost all preliminary tests showed short circuits. Thus, we did not investigate the glass material further.

The PMMA particles (Figure 6) are microspheres with the desirable spherical morphology. PMMA stands for Poly methyl methacrylate, also known as acrylic glass. The material is thermoplastic and transparent. Two sizes have been used for this work:  $20 \ \mu m$  and  $50 \ \mu m$ .

The goal is to find the optimum distance between anode and cathode. On the one hand a reliable separation must be assured on the other hand if the resistance is too high then the ions cannot move anymore between cathode and anode hindering the proper working of the battery. For that reason, the right number and size of the beads in the electrolyte must be evaluated.

To find out about the minimum separator function a simple short circuit test was carried out by putting different weights (mechanical loads) on the batteries. However, that was not a complete battery in this case. The test samples do not need to contain anode and cathode. The printable electrolyte was just sandwiched between the upper and the lower current collector making this a very harsh test since the thick-

ened electrolyte itself could contribute as well at least a bit to the separating function. During the loading with weights simply the ohmic resistance was measured using a multimeter.

# 2.5.2 Electrolyte composition and preparation

Both chemicals,  $ZnCl_2$  and  $NH_4Cl$ , are white crystalline salts that are highly soluble in water. They both are commonly used as electrolytes as they have high ionic conductivity. Distilled water was used to create a solution with the Zinc Chloride and Ammonium Chloride as well as dissolve the Agar-Agar.

The electrolyte was prepared in a plastic laboratory jar. The substances were added in the following order: first the salts to form the solution with the water. Then the thickener Agar-Agar was added up in several steps, each time small amounts were added to the system until it reached the desired amount in the desired percentage. By adding the thickener, the electrolyte was constantly mixed up to avoid inhomogeneity. After the electrolyte was ready, it was manually mixed so that the Agar-Agar could dissolve in the liquid solution.

# 2.5.3 Printing process

A semi-automatic laboratory screen-printing machine Ekra E2 was used. The following specifications have been set up: the speed of the squeegee set to 100 mm/s; pressure of the squeegee 136 N; snap-off distance 2 mm. The screen used was a PET mesh with 21-140 (n-d i.e., mesh count in threads per cm – thread diameter in  $\mu$ m). This mesh has a nominal mesh opening of 340  $\mu$ m. The rule of thumb that the mesh size should be at least 3 to 4 times the largest particle size (e.g., 50  $\mu$ m PMMA beads) in the ink is thus easily fulfilled.

A two-stroke operation was used, meaning that the flood bar and the squeegee moved two times back and forth. Figure 7 shows the placement of the pre-printed current collectors onto the vacuum table of the press. Two copies with upper and lower current collectors fit on the layout. Later, the two halves are folded over each other along the previously made perforation in the middle.



Figure 7: Pre-printed battery current collectors (two copies) placed on vacuum table of press

Figure 8 shows the simple, rectangular layout for the printing of the electrolyte. The electrolyte is printed on both the lower and the upper current collectors respectively electrodes. Since the left side of the screen was not used here it was taped with a silver blocking tape.



Figure 8: Ink (printable electrolyte) poured on the screen

### 3. Results

The standard electrolyte solution that was found to be optimal during previous preliminary tests was 40 wt.% of  $\text{ZnCl}_2$ . To this master solution different amounts of thickener (Agar-Agar) were added. Figure 9 shows the viscosity readings at a shear rate of 100 s<sup>-1</sup>. The measurements were not repeated with a statistically significant numbers of test runs. However, the accuracy can be estimated at ± 2 Pas.



Figure 9: Viscosity of the electrolytes with six different percentages of Agar-Agar

The thickener has been added with regard to the amount of water in the solution. It is evident that the higher the amount of thickener is, the higher will be the viscosity. Since the printability of the tests was satisfactory beginning with an addition of 11 % thickener or higher, we continued all further experiments with a standard of 11 % (equivalent to 7 wt.% of the total solution).

With regard to battery performance, the influence of the addition of the thickener on the conductivity is important. From Figure 10 it can be seen that there is a clear influence. However, the assumption that the addition of the thickener reduces the amount of water and salt does not correspond to the extent of the decrease in conductivity. It could be that the addition of agar forms a gel network that impedes the movement of ions (including hydrate shells) through the electrolyte. These measurements were carried out in the test setup shown in Figure 2 and do not represent the final achievable battery performance. This is the subject of ongoing research.



Figure 10: Conductivity measurements electrolyte solutions at frequency 1 MHz; the ZnCl, content was kept constant at 40 % with respect to water

### 3.1 Usage of spacers

Although results with the addition of phenolic resin beads to the standard formulation (40 % ZnCl<sub>2</sub> based on water content, 11 % Agar based also on water content) at the first glance looked promising

- Increase of viscosity (at 50 s<sup>-1</sup>) from 35.5 Pas to 38.4 Pas
- Decrease of conductivity from 73.1 mS cm<sup>-1</sup> to 71.5 mS cm<sup>-1</sup>

the investigation was not continued because over time the properties changed significantly, and it is assumed that due the hollowness of the beads the electrolyte penetrates into the inner body and conversely that means that the resin somehow is softened or even dissolved. Further, a reaction could not to be excluded. As can be seen in Figure 11, the prints show a foam effect (trapped air escaping?) and a very inhomogeneous surface. The unevenness decreases a little over time, but too many imponderables remain, so this approach was not pursued further.



Figure 11: Inhomogeneous surface with foam like structures appeared after printing the standard electrolyte with phenolic resin hollow beads

The continuation of the experiments with PMMA beads turned out to be more successful. In Table 1 the results of the short circuit test (printable electrolyte with beads added sandwiched between lower and upper current collectors) are listed. Short circuit as a result means that there is no separation function achieved, infinite, however, is the desirable outcome.

PMMA 20 µm beads	Amount of beads						
	5 %	8 %	14 %	17 %	18 %	19 %	
Weight 330 g	circuit	uit	circuit	circuit	>600 kΩ	nite	
Weight 600 g		circ			~300 Ω		
Weight 850 g	ort	ort	ort	ort	~70 Ω	infi	
Weight 1 100 g	Sh	Sh	Sh	Sh	Short circuit		

Table 1: Results of the short circuit test with different weights (PMMA 20 μm beads)

The results of the short circuit test with the 50  $\mu m$  PMMA beads summarized in Table 2 are much more promising.

PMMA 50 µm beads	Amount of beads				
	1%	2 %	3 %	5 %	
Weight 330 g	uit	uit	infinite		
Weight 600 g	circı	ort circ	~70 Ω	nite	
Weight 850 g	ort		Short circuit	infi	
Weight 1 100 g	Sh	Sh	Short circuit		

Table 2: Results of the short circuit test with different weights (PMMA 50 µm beads)

Based on this result for further testing PMMA 50  $\mu m$  beads were used with 4 wt.% in the electrolyte formulation.

In parallel work was done on finding most promising mixtures of  $\text{ZnCl}_2$  and  $\text{NH}_4\text{Cl}$  and further investigations focused on the optimum mixture of these two components. A series of measurements with different mixture ratios was carried out. Figure 12 shows the increase of conductivity measured with the different ratios. The components are abbreviated. "A" stands for  $\text{NH}_4\text{Cl}$ , and "Z" for  $\text{ZnCl}_2$ . The sum of the components was always kept at 40 %.



Figure 12: Conductivity of ZnCl<sub>2</sub> and NH<sub>4</sub>Cl, solution mixtures

The rheological measurements with these different ratios showed no change in behaviour, at all.

To find out what is the optimum ratio between "A" and "Z" batteries were fully assembled and tested. Figure 13 shows the result in terms of energy.



Figure 13: Performance of test cells with different ZnCl<sub>2</sub>/NH<sub>4</sub>Cl mixtures

As a result from this test, the optimum ratio was assumed to be Z 25 % + A 15 %. In the further investigation this ratio is called "S".

Figure 14 shows a comparison of the measured conductivity between the new standard formulation "S" with different levels of Agar and the starting point "Z" 40 % + 11 % Agar. It is evident that the lowest amount of addition of any material is desirable.



Figure 14: Conductivity of different electrolytes with 4 % PMMA 50 beads at f = 1 MHz

The addition of beads, however, changes the viscosity readings significantly. As can be seen in Figure 15 the values increase by factors between 25 and 4.



Figure 15: 2 viscosity of different electrolytes with and without 50 µm PMMA beads

Figure 16 shows some print results. The spacer functionality can be seen clearly. The  $\text{ZnCl}_2$  electrolyte with the highest amount of Agar shows the smoothest surface. However, this might not be desirable since the electrolyte and the anode and cathode electrodes should have a high common surface to amplify the ease of ion transport.



Figure 16: Printed samples with three different electrolytes and 5 % PMMA 50 µm beads

With these results in total six fully printed sample cells were assembled.

- + 2 cells with 40 %  $ZnCl_{_{2'}}$  11 % Agar-Agar + 4 % PMMA 50  $\mu m$  beads
- 2 cells with 25 %  $ZnCl_2$  + 15 %  $NH_4Cl$ , 8.7 % Agar-Agar + 4 % PMMA 50  $\mu$ m beads
- 2 cells with 25 % ZnCl<sub>2</sub> + 15 % NH<sub>4</sub>Cl, 8.7 % Agar-Agar + 5 % PMMA 50  $\mu m$  beads



Figure 17: Discharge curves of the six assembled fully printed cells (the raise of the curves after the discharge voltage of 0.9 V has been reached is an artefact of the potentiostat which erroneously tries to recharge)

The discharge curves of the cells at a constant load of 0.5 mA are shown in Figure 17. One of the cells failed quite early. The reason is unknown, but all the others performed very well with a duration of up to 70 hours. In Figure 18 the corresponding performance results are shown. They are given in energy values mWh or, as usual for batteries, in terms of capacity in mAh. The area of the cells is approximately 13 cm<sup>2</sup>. Thus, some cells show an – for printed zinc manganese dioxide cells – outstanding capacity of 2.7 mAh/cm<sup>2</sup>.



Figure 18: Energy and capacity of the cells (without the failed cell)

# 4. Conclusion

It could be shown that a printable electrolyte is possible by using a widely available and affordable ingredient Agar-Agar used as a thickener agent. By adding PMMA beads around 50  $\mu$ m the separator function can be realized in printing techniques only by avoiding the disruptive process of inserting a fleece soaked with liquid electrolyte.

By using a mixture of ammonium chloride and zinc chloride the overall capacity of the battery could not be increased but the batteries perform much better if a pulsed load is applied.

Further optimizations need to be done regarding the size of the beads and shelf life stability.

### Acknowledgements

Parts of the research was funded by the German Ministry of research and education in the scope of the project OxiFlexIT.



Some contributions stem from the project BEWELL that was funded under the EU program Horizon 2020. Thanks to all the partners in these projects, especially VARTA Microbattery GmbH.



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