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HIGREEW – Deliverable Report

<< D3.3 – Stack Delivery >>



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Publishable summary

The HIGREEW project set out to design, build, and demonstrate a prototype of a new high energy density generation of Aqueous Organic Redox Flow Battery (AORFB), based on a water-soluble low-cost organic electrolyte, and featuring low-cost components and long service life.

This deliverable report (D3.3) provides a summary of the stacks produced in work package 3 for optimizing and characterizing the divided HIGREEW chemistry in an energy storage system. The stack design was based on the chemistry and materials defined in work package 2 along with the verification and modelling work in work package 3.

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Abbreviations

Symbol / Short name	
BoP	Balance of Plant
BoD	Basis of Design
CEM	Cation exchange membrane
DoW	Description of Work
EU	European Union
EPDM	Ethylene propylene diene monomer
IET	Institution of Engineering and Technology
PAN	Polyacrylonitrile
PP	Polypropyleney
PVC	Polyvinyl chloride
TBC	To Be Confirmed
TPE	Thermoplastic Elastomer
TPV	Thermoplastic vulcanisates
WP	Work Package

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1 Introduction

The aim of work package 3 in HIGREEW was to produce prototype stacks for testing the performance of the HIGREEW electrolyte at scale, increase understanding of the performance of an energy storage system based on the HIGREEW electrolyte (stack plus balance of plant systems) and to increase understanding in how to design flow battery stacks for similar organic chemistries.

Work package 3 uses the divided form of the HIGREEW chemistry with separate positive and negative electrolytes which offers a higher cell voltage and energy density than with the mixed electrolyte used in Work packages 4 and 5.

The resulting stack design and manufacture activity in T3.4 was based on:

- Preliminary cell design (T3.1) based on results from WP2.
- Optimisation of critical cell and design parameters (electrode compression, flow rate, shunt currents, etc.) from T3.2
- Models for the cell and stack dealing with hydraulics and shunt currents behaviour from T3.3.

Two stacks were produced and commissioned, which will be used for further characterisation / testing of the divided HIGREEW chemistry in Task 3.6 Stack energy system development and stack integration.

This deliverable report documents the two stacks that have been produced and contains characterisation data that demonstrates that they are suitable for use with the HIGREEW chemistry by obtaining comparable performance to the laboratory cells used in work package 2 to define the chemistry and materials. This deliverable report is also evidence that Milestone 3 – “Optimised stack finalised for integration into the balance of plant” has been achieved.

2 HIGREEW Requirements

The requirements for the HIGREEW work package 3 stack were:

- Size up to 1 kW and 20-unit cells.
- Suitable for the divided HIGREEW chemistry and materials:
 - Negative electrolyte: 1.1M (SPr)₂V, 1 M KCl, 1 M NaCl,
 - Positive electrolyte: 1.1M mixture of potassium ferrocyanide and sodium ferrocyanide with total ferrocyanide concentration of 1.12M).
 - Membrane: Fumatech FS950
 - Carbon felt electrodes: SGL GFD 4.65 EA with a 11 to 30% compression
 - Bipolar plates: Eisenhuth PPG86 or Shin-Etsu carbon polymer composite.
- Pressure drop ideally less than 0.8 bar.
- Single cell prototype and stack prototype ideally with an electrical resistance < 0,6 Ω cm².
- Manufactured for easy disassembly and recycling.
- Shunt current losses reducing the coulombic efficiency by about less than 1%.
- Flexibility in design with a machined approach due to the still developing chemistry and to recognize that changes may need to be made to the stack during the trials in T3.6 in order to best operate such an organic RFB.
- Demonstrate scale up of application of the HIGREEW chemistry.
- Provide a test bed for novel seal designs in redox flow battery stacks which could be advantageous in stack prototyping and small to medium scale manufacture before injection moulding can be justified.

Given that two partners were involved in the stack design and testing were located some considerable distance apart (United Kingdom and Czechia), it was advantageous to split the stack build between the two main stack engineering partners to make it easier for them to make changes during the T3.6 trials with the additional advantage that initial results could be obtained faster at PINFLOW using a stack based on an existing design without having to wait for the delayed delivery of seal printing equipment at C-Tech and required development program to obtain good quality seals.

The functional specifications of the C-Tech and PINFLOW stacks were almost identical and should offer very similar flexibility and performance. The commonality includes:

- PVC construction material for rigidity, good machining characteristics and lower thermal expansion than PP.
- Fluid distributor which was developed in Task 3.3.
- Identical electrode felt.
- Both flexible in terms of the range of felt electrode compressions that are possible by changing the channel depth with both initially around 15%.
- Electrode height very similar so pressure drop comparable.
- Bipolar plates – same material and thickness.

The principal differences between the PINFLOW and C-Tech stacks were:

- Sealing methods. PINFLOW used their proprietary sealing methods from their commercial stacks. C-Tech used a new method of printed seals in shallow and wider o’ring which required new equipment and development time.
- Orientation – The PINFLOW stack is a landscape format, while the C-Tech stack is currently portrait. However, it has the same electrode flow channel height to offer the same stack pressure drop for the same flow fluid linear velocity. A future modification could be to widen the C-Tech stack to landscape format now that the sealing system has been developed.
- Manifold position. PINFLOW’s design which was based on their commercial VRFB stack locates the manifolds on the side walls of the stack to give the most efficient utilization of the stack area and depending upon the length (electrical resistance defined) of the feed channels to the flow distributor may not always allow the cell to fully drain. The C-Tech design located the manifolds

top and bottom of the electrode flow channel to allow complete drainage, as it was anticipated that during seal development / testing that the system would need to be repeatedly assembled and disassembled, so the ease and safety offered by full drainage was worth the extra materials cost for a development stack.

However, given that electrical and hydraulic performance of the stack is principally determined by the design factors which are essentially the same then similar performance is expected for both stacks.

3 PINFLOW Stack

3.1 Introduction and Design

The stack developed for WP3 was based on the commercially offered PINFLOW stack for VRFB, but the inner components were optimised for the needs of the chosen organic electrolytes (negolyte: 1.1M (SPr)₂V, 1 M KCl, 1 M NaCl, 1:1 posolyte mixture of potassium ferrocyanide and sodium ferrocyanide with total ferrocyanide concentration of 1.12M). The materials used (membrane Fumatech FS950, electrodes SGL GFD 4.65 EA, bipolar plates Shin-Etsu) were chosen according to the results of WP2 and the design of the flow frames (Fig .1) was based on CFD and the shunt current models developed within D3.3 with respect to optimisation of pressure losses, shunt current losses and electrolyte distribution within the electrode.

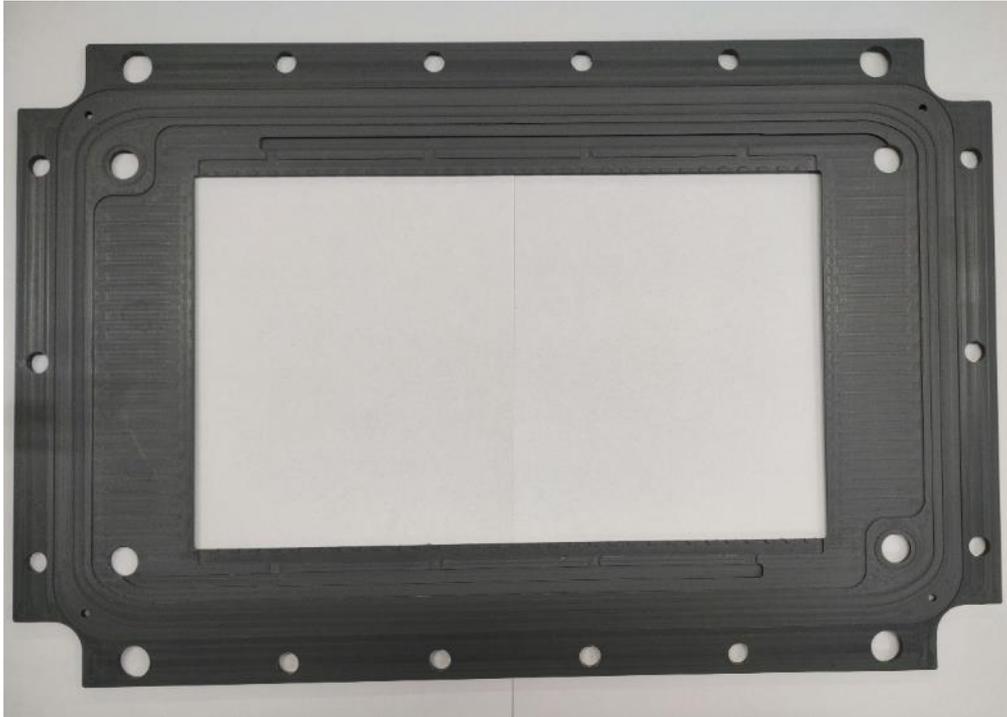


Figure 1 Design of flow frame optimized for HIGREEW chemistry at PINFLOW.

The large cell tests were conducted in three different sizes. Single cell with active area of 608 cm², 5-cell stack with active area 608 cm² (19 x 32 cm) and 20-cell stack also with active area of 608 cm². For battery stack testing two different systems were developed (one for single cell and 5-cell stack (Fig. 2) and one for 20-cell stack (Fig. 3)). These systems enabled measurement of cell potential, OCV, OCP, measurement of multiple temperatures and measurement of pressure losses within the battery stack. The stacks were characterised by measurement of EIS, load curves, galvanostatic charging and discharging cycles, and in terms of pressure losses in different states of charge, the obtained results were compared with measurements with laboratory 20 cm² systems.

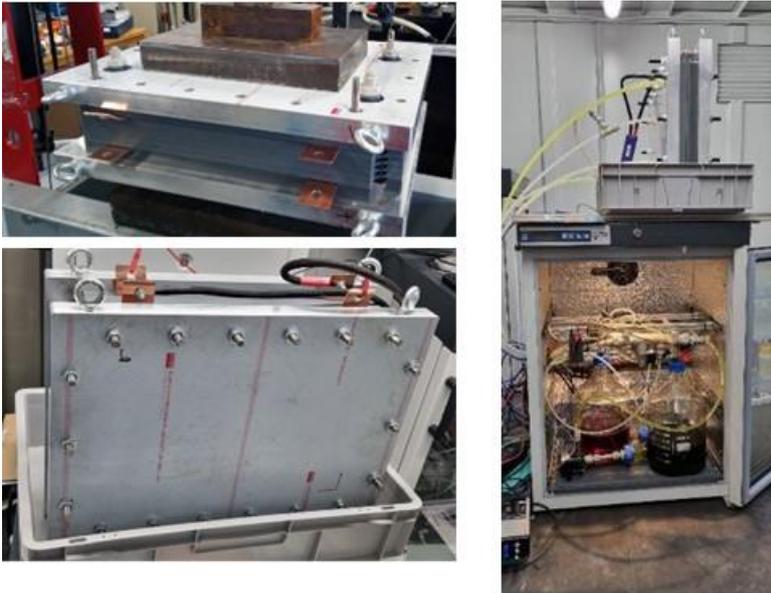


Figure 2 Small system for testing of single cell and 5-cell stack: a) assembling of 5-cell stack, b) connected 5-cell stack, c) electrolyte tanks and other measurement devices placed in temperate box.



Figure 3 Large system for testing of 20-cell stack.

3.2 Flow / Pressure Characterisation

Pressure losses were not compared with laboratory 20 cm² cell as laboratory experiments were performed with the usage of peristaltic pumps, which allows more flexibility and easier control for laboratory experiments. Results of pressure losses obtained from measurements with a single cell are presented in Figure 5. With increasing SoC pressure drop in the positive half-cell is slightly decreasing because of decrease of electrolyte viscosity while pressure drop within the negative half-cell is increasing, mainly above 50 % SoC, again because of increased viscosity of the electrolyte. The comparison of pressure losses within the single cell, 5-cell stack, and 20-cell stack is presented in Figure 4. There is a good agreement of measurement with different cells. A small discrepancy for the positive electrolyte for the 20-cell stack could be observed for higher flow rates. This discrepancy is probably caused by the fact that 20-cell stack was measured at different system, and for high flow rate (in fact 20 times higher than in case of single cell, as flow rates are related to the number of cells), there is an effect of piping system between the cell and the pressure sensors.

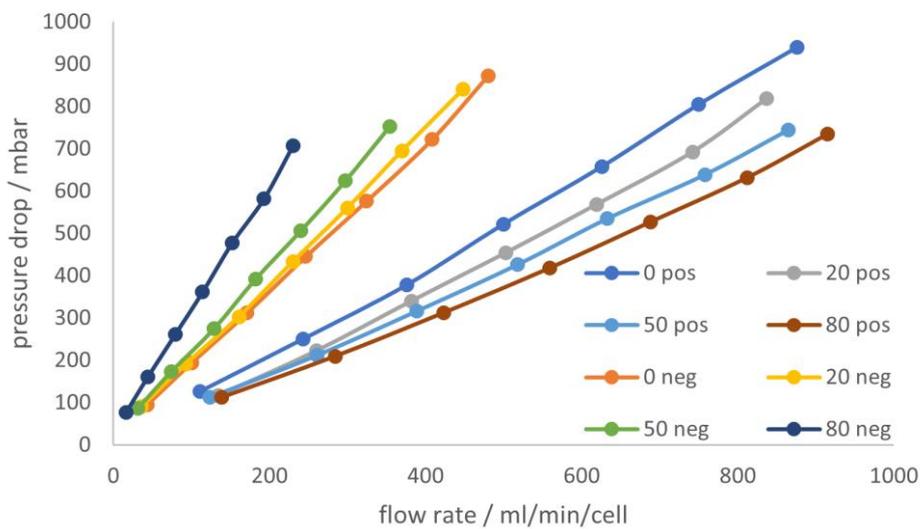


Figure 5 Pressure losses measured with single cell for different state of charge for 20°C

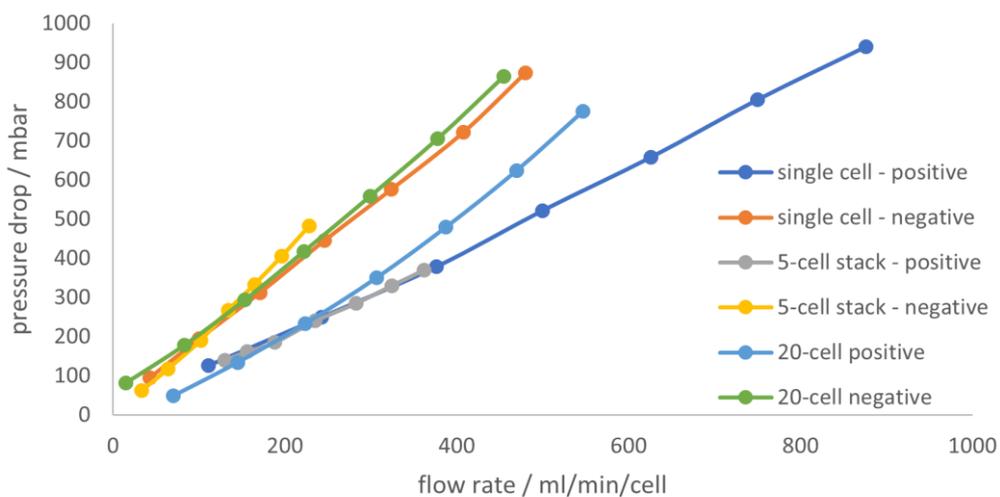


Figure 4 Pressure losses measured with single cell, 5-cell stack and 20-cell stack for 0 SoC and 20°C

3.3 Electrochemical Characterisation (EIS and Cycling)

The results of the EIS measurement with the 5-cell stack, and 20-cell stack show that the difference in the ohmic resistance between the individual cells were very similar and the maximal difference was less than 5 % and there is also good agreement on the ohmic resistance between the single cell, the 5-cell stack and the 20-cell stack, as presented in Table 1. Ohmic resistance obtained by EIS measurement. However, there is a relatively significant difference in ohmic resistance measured with laboratory cell and larger cells. This is probably due to a wrong calibration of the potentiostat used for the measurement of large cells as all those experiments were performed with the same electrolyte. Difference between WP2 and WP3 are related to the electrolyte batch employed. The values of ohmic resistance are higher than they should be as the resistance measured with the load curves (Table 2) is lower than the ohmic resistance.

Experiment	Ohmic resistance / Ohm cm ²
laboratory cell	2.9
single cell	4.25
5-cell stack	4.38
20-cell stack	3.95

Table 1 Ohmic resistance obtained by EIS measurement

Load curves

Results of load curves were measured at 50 % SOC, negative electrolyte flow rate of 0.25 ml/min/cm² and positive flow rate of 0.65 ml/min/cm². Measurements with laboratory cell, single cell, 5-cell stack, and 20-cell stack show relatively good agreement between individual experiments. The 20-cell stack was not possible to be measured at 20 °C, as the large system for the 20-cell stack was not placed in a temperate box as in the case of smaller systems. Heating of the system will be discussed in the part about the cycling experiments.

Experiment	Resistance from load curves - charging / Ohm cm ²	Resistance from load curves - discharging / Ohm cm ²	Temperature / °C
laboratory cell	3.05	3.15	20
single cell	3.65	3.81	20
5-cell stack	3.41	3.62	20
20-cell stack	2.95	3.09	25

Table 2 Resistance obtained from load curves for different batteries

Cycling

The results of the cycling experiments with laboratory cells, single cells and 5-cell stacks for 20 °C, negative electrolyte flow rate of 0.25 ml/min/cm² and positive flow rate of 0.65 ml/min/cm² are presented in Table 3. Efficiencies are well aligned for different battery sizes.

laboratory cell			
Cycle n.	CE	VE	EE
2	97.7%	60.5%	59.2%
10	98.6%	59.7%	58.9%
20	98.4%	59.3%	58.4%
single cell			
Cycle n.	CE	VE	EE
2	103.2%	57.2%	59.0%
10	98.8%	57.9%	57.2%
20	98.9%	57.4%	56.7%
5-cell stack			
Cycle n.	CE	VE	EE
2	101.5%	62.2%	63.2%
10	99.2%	61.2%	60.7%
20	99.7%	60.7%	60.5%

Table 3 Efficiencies for cycling experiments with different batteries

The results of the cycling experiments with the 20-cell stack are not directly comparable as the system was not placed in a tempered box and relatively significant heating of the system was observed, as presented in Figure 6 and the corresponding efficiencies per cycle are presented in Figure 7. During cycling, the voltage efficiency increases with increasing temperature of the system from 58 to 72 %. The voltage efficiency of the second cycle with an approximate temperature of 20 °C is around 61 % and corresponds to the efficiencies measured with smaller systems. The measured Coulombic efficiency above 100 % is given by heating the system during cycling and by the experimental procedure, where the battery did not start from

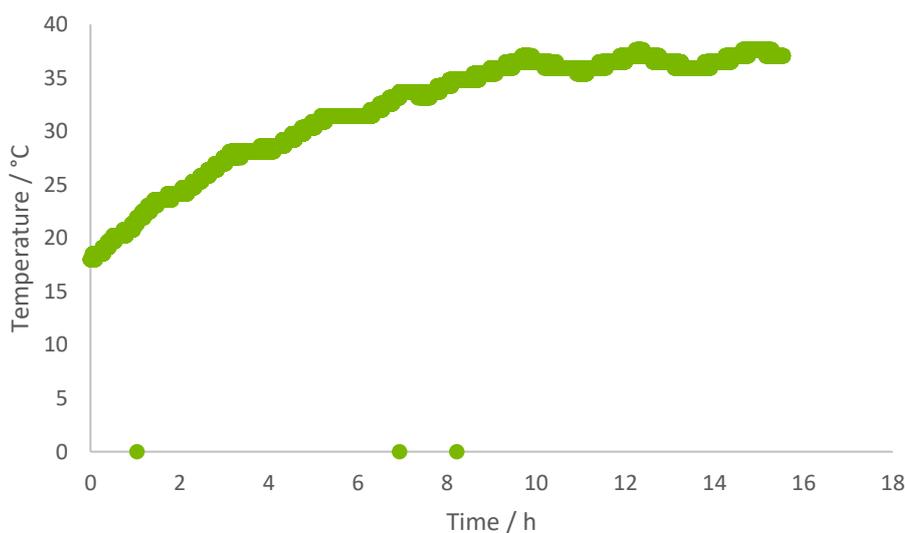


Figure 6 Temperature profile during cycling of 20-cell stack

discharged state but charged state due to the previous characterisation of the system.

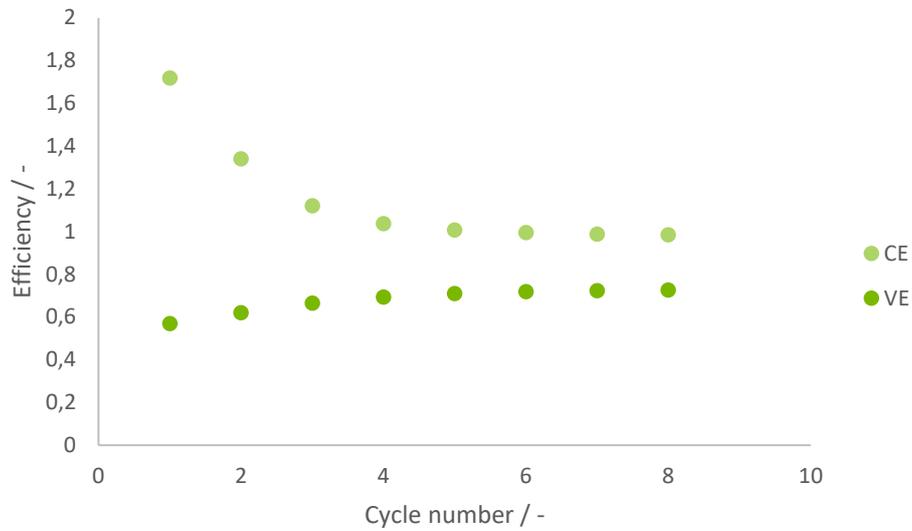


Figure 7 Efficiencies during cycling of 20-cell stack

3.4 Conclusions

- The standard Pinflow redox flow battery stack used for the vanadium chemistry could be easily modified to suit the HIGREEW chemistry.
- The HIGREEW stack was integrated with suitable balance of plant to form a fully instrumented energy storage system.
- An initial optimisation was performed for the HIGREEW electrolytes in terms of components and flow frame design and was tested in three different sizes: single cell, 5-cell stack, and 20-cell stack.
- The scale-up of the small laboratory cell was successful as the performance with all three sizes of the stack was comparable with that of the laboratory cells used in Work package 2 for the divided HIGREEW chemistry.

4 C-Tech Stack

4.1 Stack Design and Manufacture

The design of the stack can be seen in the computer aided design images in Figure 8. The main features were:

- Layup sequence:
 - Clamp plate with compression provided by tie bars, nuts and constant force washers.
 - End Housing containing a current collector and fluid fittings to supply the internal manifolds.
 - First unit cell frame assembly (carbon bipolar plate and a single PVC unit cell frame containing fluid manifolds, fluid internal distribution, cover plates and seals). Some of the seals are also provided by the seals on the end housing.
 - Membrane assembly – membrane glued into an outer PET two-layer picture frame for easier handling and to minimize membrane usage.
 - Bipolar unit cell assemblies (carbon bipolar plate inside two PVC unit cell frames containing fluid manifolds, fluid internal distribution, cover plates and seals) in a single easily handled unit separated by membrane assemblies.
 - Final unit cell frame assembly
 - End Housing containing a current collector.
 - Clamp plate
- Seals.
 - Manifolds and flow field outer seal on the unit cell frames – printed adherent o’ring type seals in shallow grooves.
 - Bipolar plates – printed adherent seals around the internal corners of the bipolar plate cavity of each half of the unit cell frames. The bipolar plates have 45° chamfers on the edges and seal when the two halves of the unit cell are compressed together. The two-unit cell frames are permanently held together by solvent welding of the PVC.
 - End housing seals around the current collector - printed adherent o’ring type seals in shallow grooves.
- Cover plates over the flow distributors to prevent them being blocked by membrane.
- Unit Cell frames. A two-part frame made up of identical half frames reversed around the bipolar plate. Internal seals around the bipolar plates and manifolds are provided by printed seals which are compressed by solvent welding of the PVC frames.
- Flow distributor design from the modelling work done in T3.3 and almost identical to that used in the PINFLOW stack.
- Electrode felt contained between the inlet and outlet flow distributors. On assembly this is compressed by the difference in height between the felt (nominally 4.6 mm) and the depth of the cavity in the unit cell frame up to the bipolar plate (around 3.8 mm). The felt compression can be changed by machining unit cell frames with a different cavity depth.
- Fluid manifold positions for both electrolytes. These were situated above and below the electrode flow channel with the aim of making the stack fully drain.
- Membrane assembly comprising an area of membrane which overlaps the electrode felt which glued between two layers of PET covering the full stack area. These minimize the membrane usage, provide easier handling during assembly and provide a much more robust area to seal against than the membrane.
- Removable guide pins which pass through the whole stack assembly including the clamp plates to provide alignment during assembly.

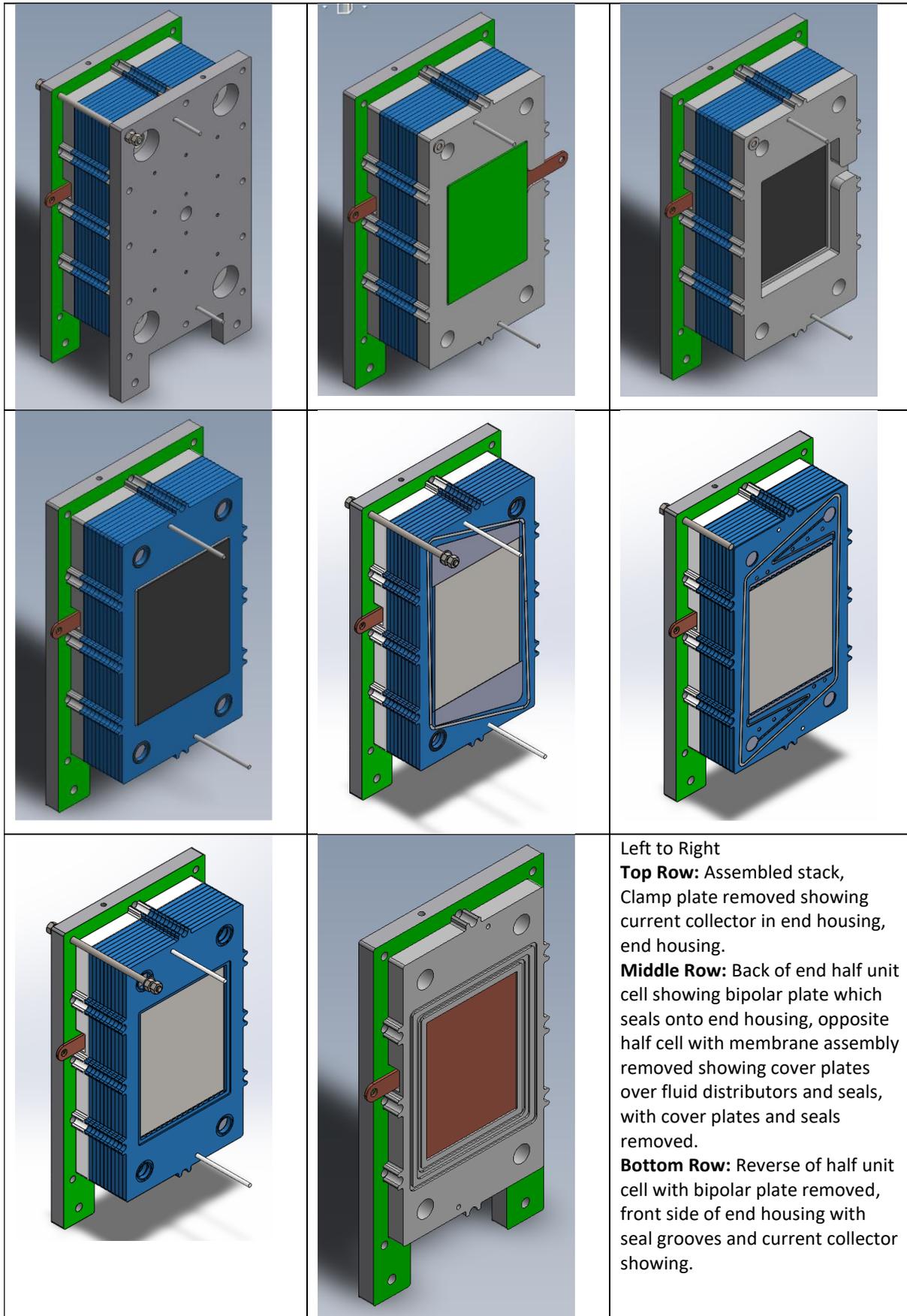


Figure 8 CAD of the C-Tech stack design showing disassembly in layers of the stack.

Machined stack designs normally use o’rings or gasket seals. O’rings can be difficult to use during assembly of larger stacks due to difficulties in retaining the gaskets and the depth of the grooves required to get a reliable seal can restrict the position of seals. Customization of gasket seals away from flat gaskets generally requires moulding with corresponding long development timescales, cost and reduced flexibility.

An alternative method which offers flexibility is “Form and Cure in Place Gaskets”. These are liquid resins applied via pneumatic nozzle using CNC machine or robot to give precise and programmable application of sealant. They offer precision control of thickness, width and position with a bead thickness down to 0.2 mm. Cure in Place (CIP) seals which are cured before assembly, only adhere to one seal face and can be disassembled. Whereas Form in Place (FIP) seals are cured after assembly and cannot be disassembled. Cure in place allows the local production of in-situ o’ring seals which are retained in place during assembly which offers significant advantages in assembly and for development of a flexible stack design. Figure 9 compares illustrates the differences.



Figure 9 Comparison of conventional preformed gaskets with cure in place and form in place gaskets.

The method was developed for electronics industry, particularly for enclosures but is now being applied wider including for automotive batteries and fuel cells. The requirements for fuel cells have many similarities with redox flow batteries having bipolar plates, fluid manifolds, membranes and external seals. After application the sealant is cured thermally, chemically or by UV. A range of resins with elastomeric properties are available, some with good chemical resistance.

The techniques offer flexibility due to the local application and programmability, potentially giving the ability to form complex / custom seals without tooling which would be ideal for development of a machined stack and may be useful up to medium volume production.

Trials with a local supplier (Intertronics Ltd) led to selection of cure in place using Wacker Electrosil N2189, a neutral-chemical curing system (alcoxy) silicone which cures at RT under influence of atmospheric moisture which gave good cured properties, good adhesion to PVC and required a 12 hour cure time before it usable as a seal.

The sealant was applied using a CNC robot with a controllable X-Y-Z table (Fisnar F4403N advance) and was dispensed with 14 gauge tips. Figure 10 shows the front and back faces of the unit cell frames, the robot dispensing sealant around the edge of the bipolar plate cavity on the back of the frame and examples of the deposited seals (bipolar plate and around the flow channel on the front of frame).

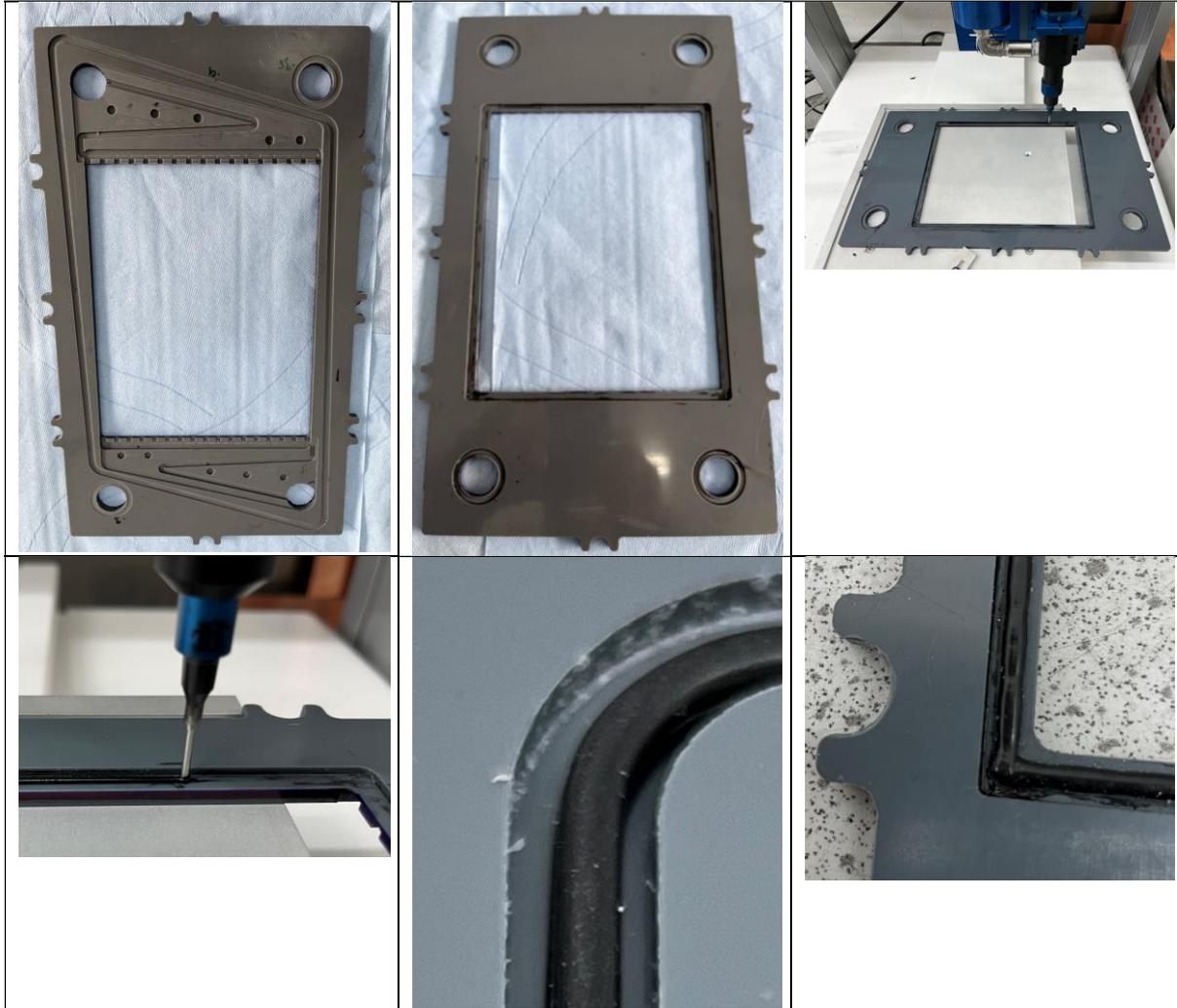


Figure 10 Deposition of cure in place seals for the HIGREEW stack and examples of the seals produced.

Assembly of the stack and it's positioning on the balance of plant test system is shown in Figure 11.

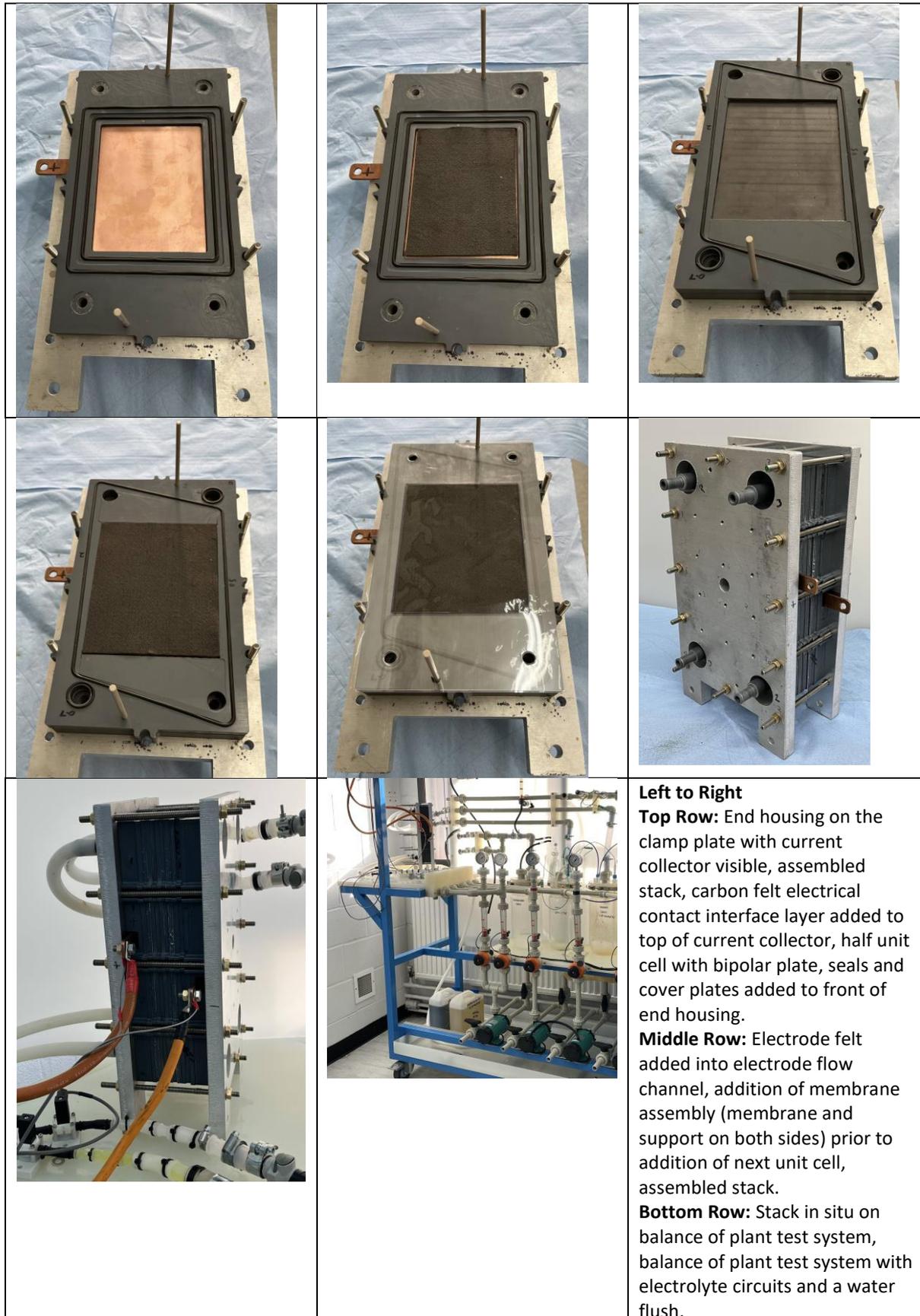


Figure 11 Assembly of the HIGREEW stack and installation in situ on a balance of plant test system.

4.2 Flow / Pressure Characterisation

As expected, the pressure losses across the stack as a function of flow rate as shown in Figure 12, were different for the two electrolytes which reflects the higher viscosity of the (SPr)₂V based posilyte. Slightly higher relative pressure drops were expected for (SPr)₂V based on the PINFLOW stack data but is probably due to the position of the pressure transducers which do not fully remove the contributions from the pipework and fittings. Pressure drops agree quite well with the PINFLOW stack data for the ferro/ferricyanide electrolyte when adjustment is made for the wider PINFLOW stack by doubling the C-Tech flow rates.

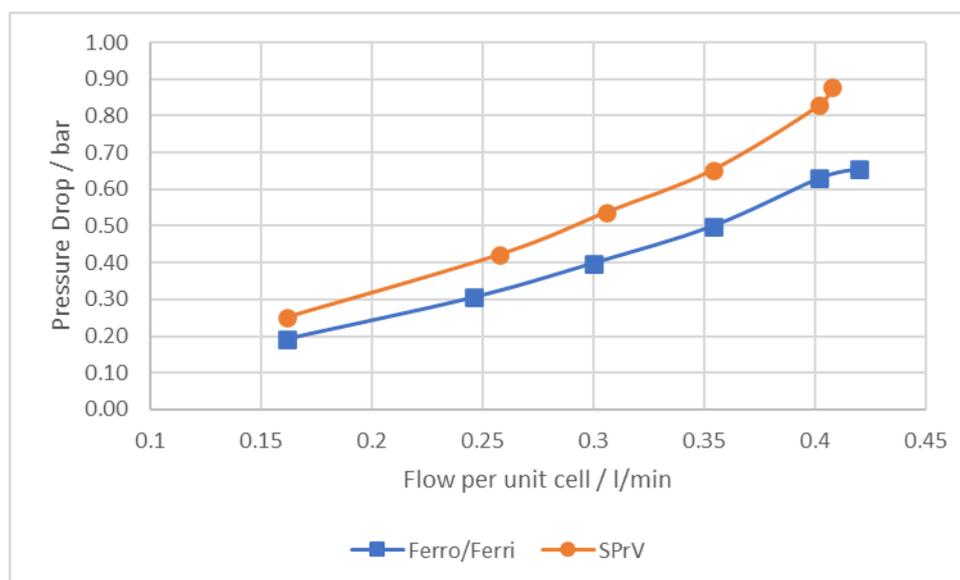


Figure 12 Pressure losses across the stack at 0% SOC and 19°C.

4.3 Cycling Data

Electrical performance of the stack was determined by conducting charge / discharge cycling using similar conditions to those used by PINFLOW in order to replicate the laboratory data from WP2:

- Charge Limit: 1.15V
- Discharge Limit: 0.6V
- Current Density: 50 mA/cm²
- Unit cell pressure drops matched at about 0.5 Bar.
- Posilyte flow Range: 0.75 to 1.1 ml/cm²/min
- Negolyte flow rate: 1.1 ml/cm²/min

Results from the first five cycles are shown in Table 14. The current, voltage and energy efficiencies are very similar to those obtained by PINFLOW at both laboratory and stack scale for the middle cycles (2 and 3) which were operated at a near constant temperature of 28-29 °C. This was expected due to the same materials and key stack design parameters being used.

Cycle No.	Charge to discharge efficiency values			Comments
	Current efficiency (%)	Voltage efficiency (%)	Energy efficiency (%)	
1	52.8	69.4	36.6	First cycle with initially cold electrolyte and charging from a lower state of charge than would be achieved with a 0.6V discharge

				cutoff. Consequently, the current efficiency was low.
2	96.2	67.6	65.1	Electrolyte at 28°C
3	89.5	64.0	57.3	Electrolyte at 19°C on start of charge.
4	101.4	64.3	65.2	Electrolyte at 28°C
5	95.0	66.2	62.9	Electrolyte at 28°C
6	65.8	63.7	41.9	Electrolyte was charged at 29°C but temperature dropped to 15°C overnight before the start of discharge.

Table 4 Cycling data from C-Tech stack. Current density 50 mA/cm², Charge limit 1.15V per unit cell, discharge limit 0.6V per unit cell, Posilyte flow 0.75 to 1.1 ml/min/cm² and Negalyte flow 1.1 ml/min/cm²

Similarly to PINFLOW, temperature effects on the stack performance were observed as increased temperature will decrease both the electrolytes resistivity and viscosity. Figure 13133 compares the discharge at a constant temperature around 28-29°C (cycle 5) with a discharge started at 15°C after being fully charged at 29 °C. The discharge voltage under load is appreciably lower at the lower temperature. The discharge voltage increases as the electrolyte temperature rises due to ohmic losses but never quite reaches the value of the 28°C electrolyte, consequently the discharge terminates earlier leading to lower voltage and current efficiency values. This indicates a strong effect of temperature on the performance of the HIGREEW electrolyte which should be studied further in later work.

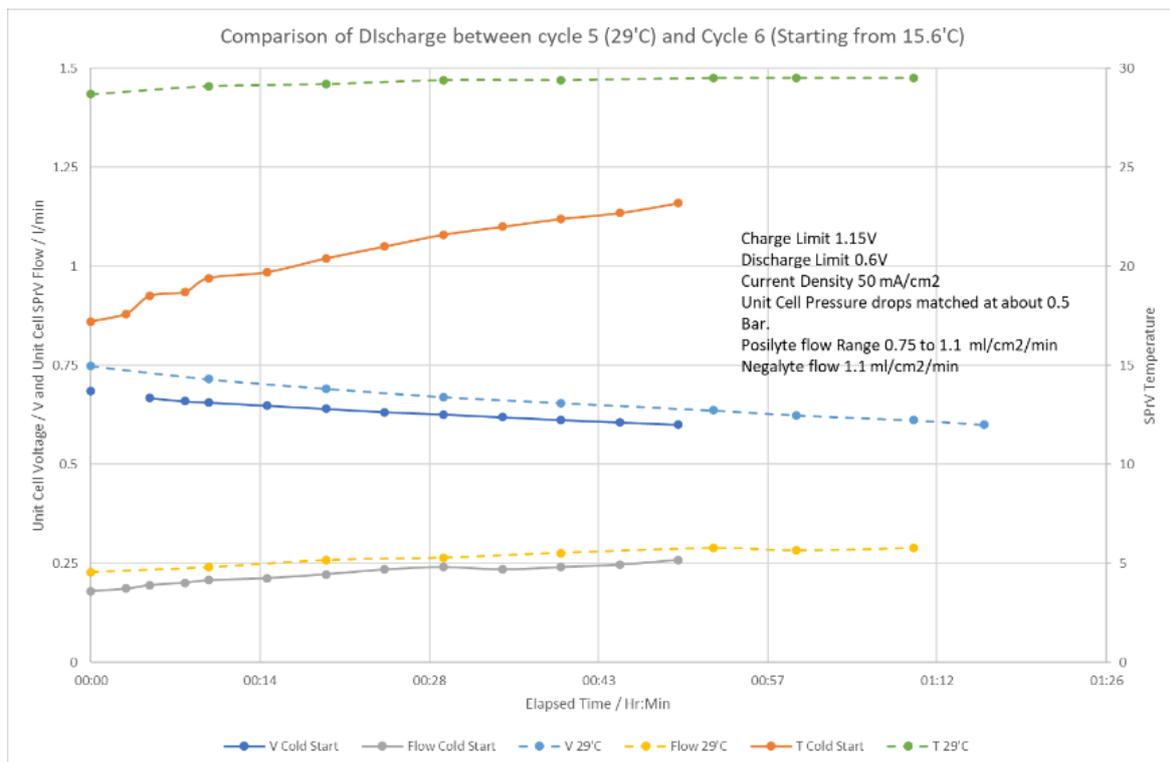


Figure 1313 Comparison of discharge between cycle 5 (29°C Temperature) and Cycle 6 (starting from 15.6°C after overnight shutdown).

4.4 Conclusions

- A flexible stack was commissioned which would be suitable for testing a range of flow battery chemistries particularly the HIGREEW chemistry.
- A novel printed seal system was demonstrated which could offer benefits prototyping flow battery systems by producing seals direct from CAD files.
- The stack was integrated into and commissioned on a balance of plant test system.
- The successful scale up was verified by the very similar electrical and hydraulic performance of the C-Tech stack to both the PINFLOW stack based on a commercial stack and the laboratory results from WP2.

5 Overall Conclusions

- Two flexible machined stacks have been produced for the HIGREEW chemistry which are part of the activity to learn about stack design for organic RFB.
- Both stacks have been commissioned and characterized.
- The performance of the HIGREEW chemistry and stack materials has been maintained during the stack engineering scale up. Excellent reproducibility of results has been demonstrated:
 - Going from a laboratory single cell (20 cm²) to commercial stack scale (20-unit cells of 600 cm²).
 - Between the PINFLOW and C-Tech stacks.
- Both stacks are installed on balance of plant test systems and will be used for further testing and optimization in Task 3.6.

6 Risk Register

The description of the Work Risk Register has been reviewed and no new risks have been identified.

7 Acknowledgements

The author would like to thank the partners in the project for their valuable comments on previous drafts and for performing the review.

Project partners:

#	Partner	Partner Full Name
1	CICe	CENTRO DE INVESTIGACION COOPERATIVA DE ENERGIAS ALTERNATIVAS FUNDACION, CIC ENERGIGUNE FUNDAZIOA
2	GAMESA	GAMESA ELECTRIC SOCIEDAD ANONIMA
3	UAM	UNIVERSIDAD AUTONOMA DE MADRID
4	CNRS	CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE CNRS
5	C-TECH	C-TECH INNOVATION LIMITED
7	UWB	ZAPADOESKA UNIVERZITA V PLZNI
8	PFES	PINFLOW ENERGY STORAGE, S.R.O.
9	UNR	UNIRESEARCH BV
10	SGRE	SIEMENS GAMESA RENEWABLE ENERGY
11	FRAUNHOFER	FRAUNHOFER-GESELLSCHAFT ZUR FÖRDERUNG DER ANGEWANDTEN FORSCHUNG E. V.

Appendix A – Quality Assurance

The following questions should be answered by all reviewers (WP Leader, peer reviewer 1, peer reviewer 2 and the technical coordinator) as part of the Quality Assurance Procedure. Questions answered with NO should be motivated. The author will then make an updated version of the Deliverable. When all reviewers have answered all questions with YES, only then the Deliverable can be submitted to the EC.

NOTE: For public documents this Quality Assurance part will be removed before publication.

Question	WP Leader	Peer reviewer 1	Peer reviewer 2	Technical Coordinator
	CTECH	PFES	FRAUNHOFER	CICe
1. Do you accept this deliverable as it is?	Yes	Yes	Yes	Yes
2. Is the deliverable completely ready (or are any changes required)?	Yes	Yes	Yes	Yes
3. Does this deliverable correspond to the DoW?	Yes	Yes	Yes	Yes
4. Is the Deliverable in line with the HIGREEW objectives?	Yes	Yes	Yes	Yes
a. WP Objectives?	Yes	Yes	Yes	Yes
b. Task Objectives?	Yes	Yes	Yes	Yes
5. Is the technical quality sufficient?	Yes	Yes	Yes	Yes



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