

NEXT GENERATION PEM ELECTROLYSERS UNDER NEW EXTREMES

Grant agreement no.: 779540 Start date: 01.02.2018 – Duration: 36 months Project Coordinator: Catherine McDougall - ITM

DELIVERABLE REPORT

		ON REINFORCED RECAST AND EXTRUDED AQUIVION® MEMBRANE	AND
Due Da	ite	January 31st, 2019 (M12)	
Author (s)		C. Oldani, M. Infantino, S. Tonella (SLV)	
Workpackage		3	
Workpackage Leader		SLV	
Lead Beneficiary		SLV	
Date released by WP leader		15 th February 2019	
Date released by Coordinator		18 th February 2019	
DISSEM	IINATION LEVEL		
PU	Public		X
РР	Restricted to other programme participants (including the Commission Services)		
RE	Restricted to a group specified by the consortium (including the Commission Services)		
со	Confidential, only for members of the consortium (including the Commission Services)		
NATUR	E OF THE DELIVERABLE		
R	Report		X
Р	Prototype X		X
D	Demonstrator		
0	Other		

SUMMARY			
Keywords	Aquivion® PFSA, Extruded, Recast and Reinforced Membranes, Torlon PAI		
Abstract	Deliverable D3.1 is aimed at the definition of the first generation of Aquivion-based membrane and ionomer for electrolysis operation under Neptune conditions. In this regard extruded E98-05S membrane (prepared using newly introduced quality inspection system), recast and reinforced membranes were prepared and characterized. Reinforced membranes were produced using novel Torlon PAI support produced via forcespinning after selection of the most promising commercial grade for this application. The three different membrane grades were evaluated in terms of proton conductivity by varying RH, mechanical properties, water uptake and dimensional stability upon soaking in hot water. Although promising, Torlon-based Aquivion-reinforced membranes are considered as not mature enough to be used in the Neptune final stack and the downselection was restricted to recast and extruded membranes.		
Public abstract for confidential deliverables	/		

REVISIONS			
Version	Date	Changed by	Comments
0.1	11 February 2019	C. Oldani, SLV	First draft

DELIVERABLE TITLE

CONTENTS

1.	Introduction	4
	Scope	
	Discussion	
3.1 9	Selection of Torlon PAI for support preparation via Force Spinning	5
3.2	Forlon Impregnation and Reinforced Membrane Characterization	9
3.3 I	Preparation of Aquivion Recast Membranes and Their Characterization	13
3.4	Preparation of Aquivion Extruded Membranes and Their Characterization	14
3.5 I	Membrane Comparison	17
4.	Conclusions and future work	19
5.	References	20
6.	Appendix	21

1. INTRODUCTION

Ionomer dispersion in electrodes and, above all, membrane are key component in Polymer Electrolyte Membrane (PEM) water electrolysis strongly affecting performance and durability of the system. Perfluorosulfonic acid (PFSA) materials offer great advantage over hydrogenated congeners in terms of proton conductivity and thermal, mechanical and chemical stability in harsh operating environment but, in order to meet the even more challenging requirements of water electrolysis conditions (i.e. temperature up to 140°C, pressure of 100 bar and current density up to 4-8 A/cm²), a step forward must be done.

In particular, the search for innovative supports to produce reinforced membranes attracts high interest because an highly performing support would allow the preparation of thinner (low resistance) membranes with high enough mechanical toughness. This need is clear not only for electrolysis but also for fuel cell applications. State-of-the-art expanded PTFE (e-PTFE) shows good performance but aromatic polymers may have advantages and are worth to be investigated. Also the demand of advanced techniques for support manufacturing is increasing and the need for less energy-intensive and more versatile systems is clear. Forcespinning is gaining momentum over more traditional techniques such as thermal processing/annealing and electrospinning and thus needs to be more deeply understood.

In order to support the increasing demand of electrolysis devices, scale up of membrane manufacturing is required and thus an even more higher productivity (fast and reliable quality control, reduction of scraps) of roll-to-roll manufacturing lines is mandatory for the more mature grades.

This Deliverable describes the achievements in preparation and characterization of advanced Aquivion-based membranes produced through melt extrusion, dispersion casting and support impregnation. Evaluation and ranking was done considering in-plane proton conductivity, dimensional changes in liquid hot water and mechanical properties as key performance index (KPI). The selection of the most suitable grades of Torlon PAI for production of advanced supports *via* forcespinning will be also discussed together the physical characterization of selected supports.

2. SCOPE

Deliverable 3.1 has the following objectives:

- Selection of the most suitable Torlon PAI grades to produce supports via forcespinning.
- Definition of optimized forcespinning conditions aimed at producing rolls of support.
- Impregnation of such supports with Aquivion dispersions (D98-25BS; EW:980 g/mol) and characterization of thus obtained membranes.
- Preparation and characterization of recast and extruded membranes (EW: 980 g/mol, 50 micron in thickness).
- Preparation and characterization of Aquivion extruded E98-05S membranes through an improved large-volume process.
- Comparison of the three membranes grades accordingly to the defined KPIs and definition of the most suitable membrane for the project.

3. DISCUSSION

3.1 SELECTION OF TORLON PAI FOR SUPPORT PREPARATION VIA FORCE SPINNING

Torlon[®] PAI (PolyAmide-Imide)¹ is an aromatic polymer commercialized by Solvay Specialty Polymers that combines the exceptional performance of thermosets polyimides (*i.e.* excellent dimensional stability and broad chemical resistance including strong acids and most organics) with the processing advantage of thermoplastics (Fig.1).

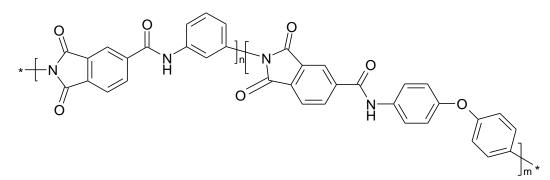


Fig.1: Structure of Torlon[®] 4000 T (n: 0.3, m: 0.7)

Torlon PAI family comprises more than 15 grades having slightly different structure, processing properties and enduses (production of high-temperature adhesives, non-stick coating primers, composites matrices, coatings, films, stock shapes for machining) but so far it was never used as support for reinforced membranes for electrolysis MEAs and few reports are available about Torlon processing to prepare spun fibers (mainly hollow fibers for gas separation and water filtration).²

Selection of the most suitable grade(s) for support preparation via forcespinning was carried out in two steps (Fig.2): the first screening was done in collaboration with Solvay experts and took into consideration the physico-chemical properties of the different commercially available grades; this allowed the reduction of the number of candidates from 15 to 5. The second step consisted in dissolution in solvents and forcespinning of the remaining grades in collaboration with a Subcontractor. The two most suitable grades, namely Torlon AI-10 and AI-10 LM, were selected considering the quality (number of defects) of the final mats. Supports with low surface defects are considered the entry level for further evaluation.

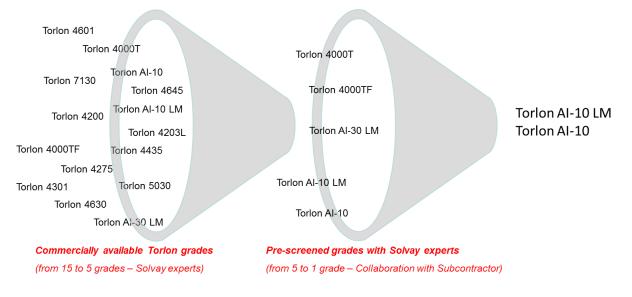


Fig.2: Selection process

The most common technique aimed at producing nanofibers is electrospinning. The search for a method that would eliminate and/or minimize many of the limitations encountered on such method focused on: increase material choice, improve production rate, and lower fiber costs through an environmentally friendly process. In this new method called forcespinning the electric field, used in the electrospinning process, is replaced by centrifugal forces. Nanofibers are produced by rotation of a spinneret and collected on a textile support. The combination of centrifugal forces with multiple configurations of easily interchangeable spinnerets makes the forcespinning a versatile method that overcomes many of the limitations of existing processes, namely, high electric fields and a solution that is typically dielectric. These changes significantly increase the selection of materials by allowing both non-conductive and conductive solutions to be spun into nanofibers. If necessary, high temperature solvent can also be used by heating the spinneret holding the material of interest. Additionally, a number of solid materials can be melted and spun without chemical preparation there is no need for solvent recovery since no solvent is involved³. A schematic of the forcespinning system is shown in Fig.3

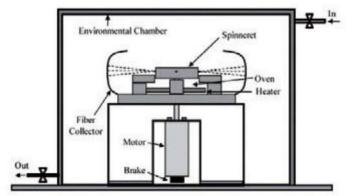


Fig.3: Forcespinning system

Fig.4 shows the surface of force-spun Torlon AI-10 LM and Torlon AI-10 in comparison with Torlon 4000 T (rejected grade). It is evident that the surface of selected grades is much more smooth and homogeneous than the rejected showing lower amount of defects (hairiness and beads from spinneret spinoffs).



Fig.4: Optical microscope images (same magnification) of selected Torlon AI-10 (left) and Torlon AI-10 LM (centre) and rejected Torlon 4000 T (right)

Microstructure of Torlon supports is characterized by a non-woven web constituted of fibers having diameter ranging from 0.3 to 1 micron. SEM (Scanning Electron Microscope) images at different magnification are reported in Fig. 5.

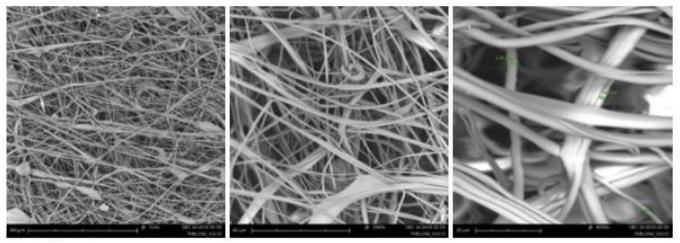


Fig.5: SEM images (different magnification) of Torlon PAI support microstructure

Considering the brittleness of such force-spun supports, stress-strain tests (ASTM D633 type V) have been carried out using a 10 N cell.

Being produced with the same polymerization system and using the same monomers, the toughness (stress and strain at break) is the same for the two samples whereas the modulus (6 *vs.* 22 MPa) is significantly different (Table 1).

This difference could be ascribed to the different manufacturing process of Torlon Al10 LM which was optimized to reduce the amount of residual methyleneaniline monomer to below 1000 ppm. This amine is expected to act as plasticizer and when in higher amount, as in Torlon Al-10, it leads to a decrease of Young's modulus and a more elastic behaviour of the material. When the plasticizer is reduced, as in Torlon Al-10 LM, Young's modulus is higher and the material is more brittle and fragile (Fig. 6).

Table 1: Mechanical properties of Torlon AI-10 and AI-10 LM force spun supports

Force spun support	Modulus (MPa)	Stress @ Break (MPa)	Strain @ Break (%)
Torlon AI-10	6	< 1	3
Torlon AI-10 LM	22	< 1	3

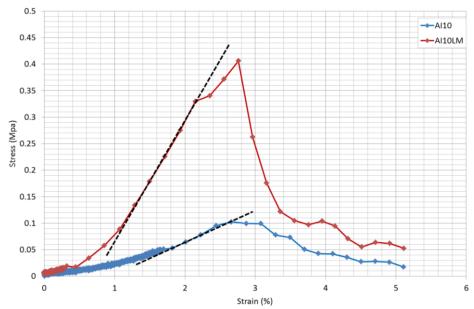


Fig.6: Calculation of the elastic modulus of the two samples as the slope of the curve at low strain (black dashed line)

After preliminary evaluation, Torlon AI-10 and AI-10 LM were then used to prepare rolls of support. It was found that in order to obtain self-supported Torlon films, it was necessary to have a grammage of at least 11-12 GSM; lower grammage led to a brittle material difficult to manipulate because of important crack formation. Such grammage allowed the manufacturing of self-standing support that can be wound as a self-standing roll (Fig.7, left). Forcespinning process produces highly electrostatic Torlon films that strongly adhere to the support textile causing a difficult peeling off and then cracks and film damages. Electrostatic attraction was minimized by discharging it with a grounding rod; this expedient allowed an easy and crack-free peeling off (Fig.7, right).



Fig.7: Manufacturing of Torlon PAI force-spun roll (left) and peeling off of Torlon film (pale yellow) from textile support (black) using the discharge rod (right).

Once prepared the novel Torlon-based support we moved to produce reinforced Aquivion membranes and compare their properties with cast and extruded congeners. High level flow chart for extruded, cast and reinforced manufacturing steps is presented in Fig.8.

Aquivion is obtained through an emulsion (oil in water) radical polymerization of tetrafluoroethylene (TFE) and a Solvay proprietary short-side chain sulfonylvinyl ether (SFVE). The polymer latex (polymer + surfactant + water) is coagulated by freeze-thawing and the resulting powder is extensively washed in order to remove surfactant and residuals. Melt processable powder in $-SO_2F$ form is shaped in pellet form (cylindric shape of 2 x 2 cm), extruded to thin membrane and finally hydrolyzed with a mineral base (KOH, NaOH) and a mineral acid (HCl, HNO₃, H₂SO₄) transforming $-SO_2F$ in $-SO_3H$.

Alternatively, key intermediate powder in $-SO_2F$ form is treated as above converting it in $-SO_3H$ and then dissolved in water at high temperature (above 200°C) and high pressure obtaining Aquivion dispersions that, once conveniently formulated, are used to prepare reinforced and cast membranes.

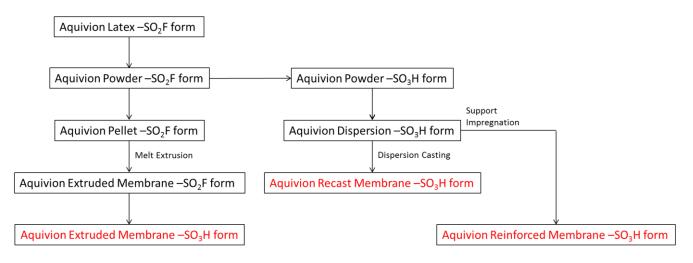


Fig.8: Flow chart briefly describing process to prepare extruded, recast and reinforced membranes

3.2 TORLON IMPREGNATION AND REINFORCED MEMBRANE CHARACTERIZATION

Taking advantage from previous funded projects (*i.e.* Stayers, Pemican and Impact), Solvay used Torlon force-spun films with Aquivion[®] PFSA⁴ dispersions to produce reinforced membranes through support impregnation. Torlon support was mounted on a PTFE frame and both the surface were impregnated with an Aquivion-based dispersion. This dispersion was prepared starting from a commercial aqueous Aquivion D98-25BS (EW: 980 g/mol, solid content: 25 wt%, stabilized) and formulated in order to have the following composition: Aquivion polymer: 20 wt%, *n*-propanol: 25 wt%, water: 45 wt% and *N*-methylpyrrolidinone (NMP): 10 wt%. Once impregnated membrane underwent an heat treatment in a vent oven at 65°C (1 hour), 90°C (1 hour) and then annealed at 190°C (1 hour). Membrane, having thickness of about 50 micron, so obtained is depicted in Fig.9.

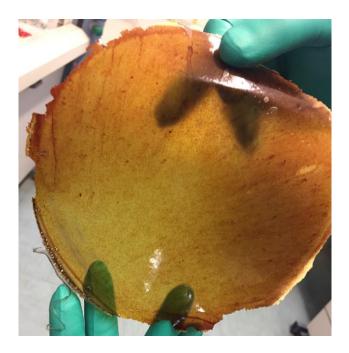


Fig.9: Aquivion membrane containing Torlon as reinforcement

Reinforced membrane was characterized *via* in-plane proton conductivity, dimensional stability in hot water and mechanical properties (tensile test).

The electrochemical measurements were performed at 80°C with a four-point-probe BekkTeck BT-112 conductivity cell. Humidified hydrogen (1000 sccm, supplied at the anode side of the cell) and heating was provided by a Greenlight Power Technologies FCATS-E 1 kW fuel cell test bench. The electrical connection was made to an Autolab PGSTAT-30 potentiostat/galvanostat (Metrohm). In-plane conductivity was measured in a relative humidity window from 20% to 120% RH. Cell resistance was determined as slope of cell voltage vs. current; conductivity was calculated considering the resistance value and geometrical parameters of sample. System was conditioned at the operating temperature for 1 h prior data acquisition.

Proton conductivity, as expected, increases when relative humidity increases from very low values (0.014 mS/cm) in dry conditions to much higher conductivity (31.6 mS/cm) in wet conditions. Worth of noticing is the steep increase of conductivity moving from 40% RH to full humidification (Fig.10). The very low conductivity under extreme low RH, closely resembles the behaviour of fully hydrogenated conductive membranes in the same conditions.

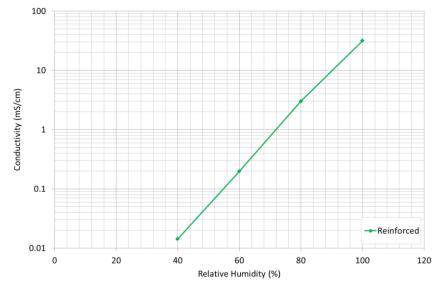


Fig.10: In-plane conductivity vs. relative humidity of Aquivion-based Torlon reinforced membrane

Dimensional stability and swelling were measured on die-cut (7x7 cm²) samples previously dried in a vacuum oven at 105°C for 1 h. Thereafter these were carefully weighed and their dimensions (length, width and thickness) measured (M_{dry}). Membranes were then soaked in demineralized water at 80°C for 4 h, cooled down at room temperature and, after wiping water droplets from the surface, weighed and the dimensional changes measured (M_{wet}). Water uptake and dimensional swelling were calculated accordingly to the following equation:

Water Uptake & Dimensional Swelling =
$$\frac{M_{wet} - M_{dry}}{M_{dry}} * 100$$

As expected dimensional changes in the three directions and swelling upon soaking in hot water is comparable in the two grades even if dimensional changes of Torlon AI-10 supported membranes are higher than the corresponding containing Torlon AI-10 LM. Thickness variation of membrane containing AI-10 is significantly higher than that of AI-10 LM-reinforced membranes (Fig.11). Worth to be noticed is that these membranes show an isotropic behaviour, swelling homogeneously in the three directions.

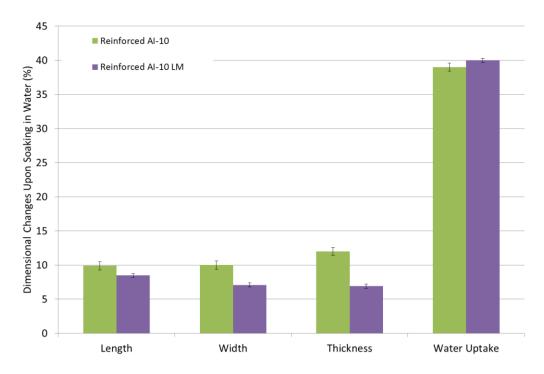


Fig.11: Swelling and dimensional changes of Torlon-supported Aquivion (EW980) membranes in 80°C water

Stress-strain curves were recorded with an Instron 5500R dynamometer equipped with a Bluehill 2 software at 23°C, 50% RH, traction speed from 1 to 50 mm/min and measuring specimens having dog bone shape (initial length: 21.5 mm and grip distance: 25.4 mm) according to ASTM D633 type V protocol. Reported curves represent mean values of measurements repeated at least 3 times.

Although mechanical properties of membranes are clearly increased upon impregnation with Aquivion D98-25BS dispersion, the influence of the support is still evident. Indeed, membranes produced from Torlon AI-10 (more elastic support) have lower modulus whereas membranes having the more brittle Torlon AI-10 LM support maintain the higher modulus (Fig.12).

The introduction of a soft, low crystallinity polymer as Aquivion is beneficial to increase both the stress and strain at break of membrane. Soft, semicrystalline Aquivion is thus expected to be able to dissipate more energy than the rigid polyaromatic structure of Torlon during traction test increasing the stress membrane can sustain and enhance the strain before break (Table 3).

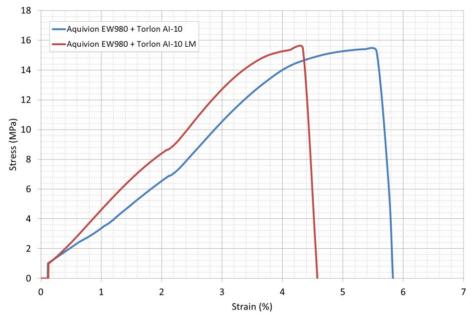


Fig.12: Stress-strain curves of Aquivion EW980 membranes reinforced with force-spun Torlon AI-10 and AI-10 LM

Table 3: Mechanical properties of Aquivion EW 980 membranes supported onto Torlon AI-10 and AI-10 LM
--

Reinforced Membrane	Modulus (MPa)	Stress @ Break (MPa)	Strain @ Break (%)
Aquivion EW980 + Torlon Al-10	426	16	6
Aquivion EW980 + Torlon Al-10 LM	540	15.1	4.5

SEM image of cross-section (obtained through fragile rupture in liquid nitrogen) of Torlon-reinforced Aquivion membranes show a different morphology when compared with Aquivion membranes containing e-PTFE as support. The latter (Fig.13, right) shows a three-layered structure with clear interphases between the two outer layers of dense Aquivion and the central layer where the pores are filled with Aquivion. Instead the former (Fig.13, left) displays an homogeneous structure where Torlon fibers are completely embedded in Aquivion matrix showing any discontinuity between the support and conductive phase.

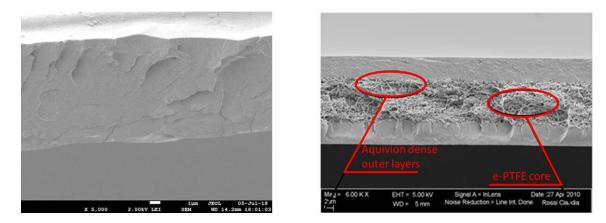


Fig.13: SEM images of Torlon-supported Aquivion membrane (left) and e-PTFE supported Aquivion membrane (right).

3.3 PREPARATION OF AQUIVION RECAST MEMBRANES AND THEIR CHARACTERIZATION

The recast short-side chain Solvay Aquivion[®] membrane with an equivalent weight of 980 g/mol and a thickness of 50 µm was prepared starting from commercial water-based Aquivion[®] D98-25BS dispersion (EW: 980 g/mol, 25 wt% solid content). This dispersion was formulated till obtaining the following composition: Aquivion[®] polymer (22 wt%), deionized water (36 wt%), n-propanol (32 wt%) and N-methylpyrrolidinone (10 wt%). The dispersion thus obtained was cast on tempered glass using a doctor blade (Zehntner ZUA2000) and an automatic applicator (Zehntner ZAA2300). After deposition, the film underwent a three-steps heating cycle in a vent oven: 1 h at 65°C, 1 h at 90°C and 1 h at 190°C. Membrane was then peeled off from glass using demineralized water. It was dried in a vent oven at 80°C.

As reinforced, cast membranes were characterized by measuring in-plane conductivity, swelling in hot water and stress-strain test using the same protocols described above.

Proton conductivity (Fig.14) is much higher than that of reinforced membrane at low as well as at high relative humidity and also its shape (i.e. the variation of conductivity by varying the relative humidity) is much more flat than Torlon-reinforced membrane.

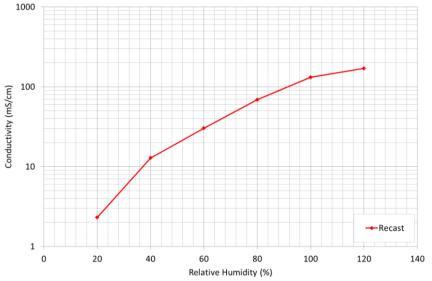


Fig.14: In-plane conductivity vs. relative humidity of recast Aquivion membrane

Cast membrane dimensional changes and swelling are reported in Fig.15. As noticed before, checking water absorption of reinforced membranes, also cast membranes show isotropic swelling in xyz direction. Water uptake is lower than that of Torlon reinforced membranes (26 wt% *vs.* about 40 wt%).

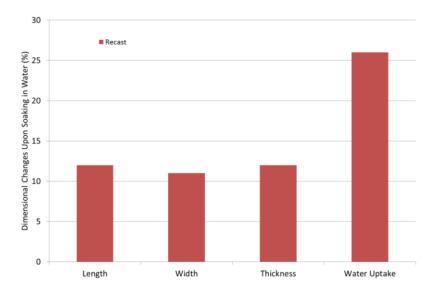
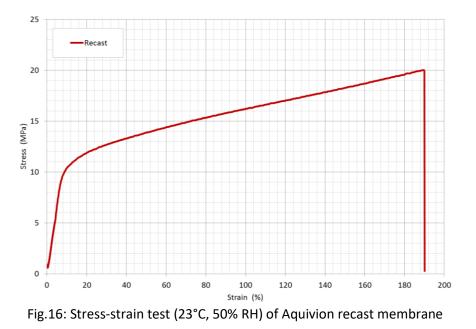


Fig.15: Swelling and water uptake of of recast Aquivion membrane

Mechanical properties of cast membranes (Fig.16) are clearly different from Torlon reinforced membranes reflecting the different nature of the two materials. Elastic modulus is significantly lower (190 MPa *vs.* about 500 MPa) due to the less crystallinity and lower rigidity of pure Aquivion than Torlon support.

On the other side, the higher elasticity given by Aquivion is beneficial in increasing stress (22 MPa vs. 15 MPa) and, above all, strain (185 % vs. 6 %) at break. Again, this behaviour can be explained considering the better capability of soft Aquivion to dissipate mechanical energy before failure.



3.4 PREPARATION OF AQUIVION EXTRUDED MEMBRANES AND THEIR CHARACTERIZATION

As discussed above, extruded membranes are currently produced starting from material in $-SO_2F$ form (the melt extrudable form) and then transformed in the active $-SO_3H$ form by treating them with a strong base and then with a strong acid. In order to increase the productivity moving from batch to roll-to-roll production, film hydrolysis is now carried out using a semi-automated line working in a clean room environment (Fig.17)

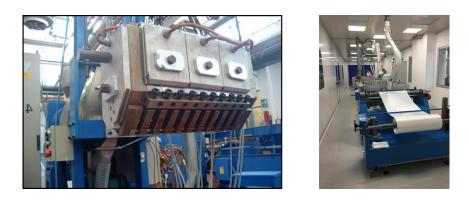


Fig.17: Extruder die (left) and hydrolysis machine (right). The white film is a PET support used to co-wound Aquivion membrane

In the frame of Neptune project an automated Optical Inspection System (OIS) has been implemented and now is currently working as real time quality control system for all commercial Aquivion membrane grades.

Defects and their acceptance limits are listed in Table 3 whereas Fig.18 shows an example of recently produced E98-05S membranes with medium quality and, for sake of comparison, the appearance of OIS output when checking a very poor quality membrane. Defects are also physically labelled on membranes. Worth to be underlined, lenses and gels, even if declared and labelled, are considered unlikely to be harmful for MEA performance and durability. Off-line inspection confirmed that membranes are pinhole-free even if sometimes OIS mix up very bright lenses with pinholes.

Table 3: Aquivion extruded membranes defects and acceptance limits

Туре	Description	Acceptance Limits
Gels	Organic material (polymer) distinct from bulk membrane.	Below 1 mm
	Gels can be generated by uneven melting of resin during	
	extrusion or partial fusion due to different crystallinity.	
Inclusions	Any foreign material.	Below 1 mm
Lenses	Elliptical and local membrane thinnings.	0
Pinhole	Any break on membrane	0

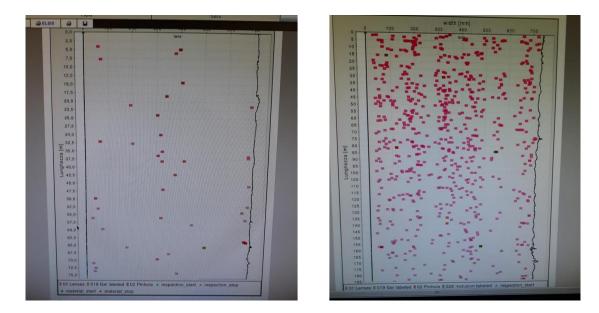


Fig.18: OIS output of a medium (left) and very poor (right) quality E98-09S membrane roll. Carpets show position of defects vs. length (y-axis) and colours identify them.

As for reinforced and recast, proton conductivity, dimensional stability and mechanical properties, have been checked also for extruded membranes.

Fig.19 shows proton conductivity upon changing of relative humidity; Fig.20 shows membrane swelling and water uptake whereas tensile test is reported in Fig.21. Melt extrusion process triggers a preferential orientation of polymer chains in xy-plane; for this reason stress-strain test has been measured in two perpendicular directions: machine (MD) and transversal (TD) direction showing, as expected, different behaviour. Anisotropic character of extruded membrane is also evident in Fig.18 where different dimensional changes are clear in the three directions. For sake of comparison, swelling of isotropic cast membrane (Fig.15) in much more uniform.

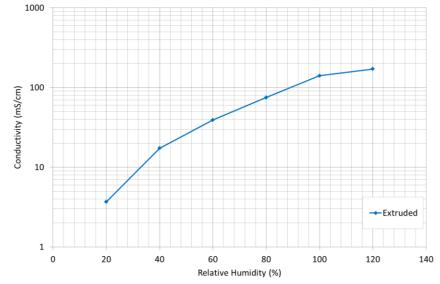


Fig.19: In-plane conductivity vs. relative humidity of extruded Aquivion membrane

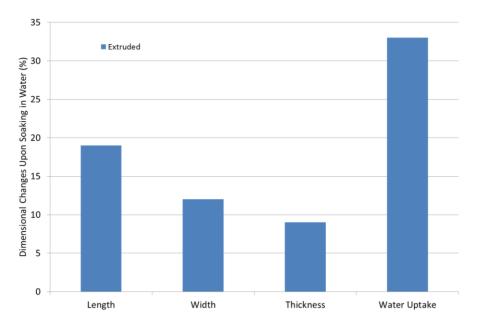


Fig.20: Swelling and water uptake of of recast Aquivion membrane

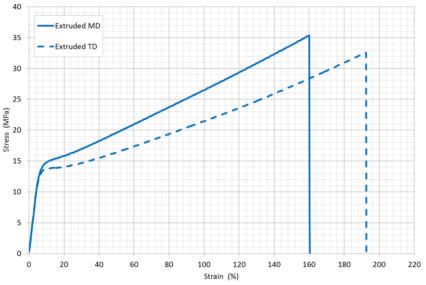


Fig.21: Stress-strain test (23°C, 50% RH) of Aquivion extruded membrane

3.5 MEMBRANE COMPARISON

Fig.22, 23 and 24 resume the comparison of conductivity, dimensional changes/swelling and mechanical properties of reinforced, recast and extruded membranes.

Extruded and recast membranes have conductivity higher than reinforced membranes in a wider range of RH. Besides a more pronounced conductivity this also indicates a better water management in extreme conditions; different water management among the membranes is clear also observing the shape of the curve.

Extruded and recast membranes have conductivity comparable as indicated by the overlapping of the two curves in Fig.21; the slight difference in conductivity at 20% RH could be ascribed to some hardware difficulties of the stand station in carefully control relative humidity in such extreme conditions. This explanation is supported considering that the two curves reach the same value at higher and less difficult to control humidity.

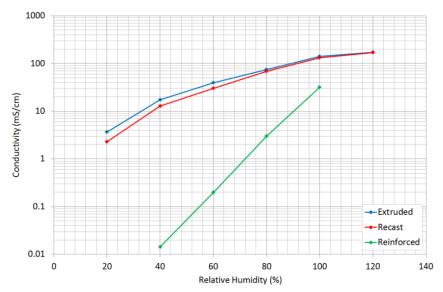


Fig.22: Proton conductivity of reinforced, recast and extruded membranes

Dimensional stability of the three membrane grades taken in consideration is not so different albeit the very different morphology of the membranes. As expected, dispersion-borne membranes are isotropic, showing the same swelling extent in the three directions whereas extruded membranes are anisotropic showing preferential swelling in one direction.

Water uptake is quite scattered and in some way reflects the structural order of membranes (Fig.23).

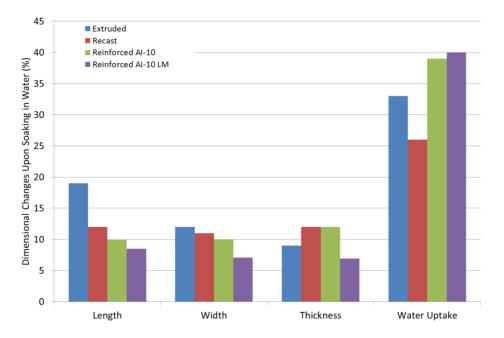


Fig.23: Dimensional stability and water uptake of reinforced (Torlon AI-10 and Torlon AI-10 LM), recast and extruded membranes

Mechanical properties of the three membrane grades tested are reported in Fig.24. As discussed above, reinforced membranes containing the rigid support of Torlon display an higher elastic modulus (slope of the curve at low strain) but, being unable to efficiently dissipate mechanical stress, they undergo a very rapid failure (low strain at break). Also the stress they can allow is low when compared to the other membranes.

Recast membrane, comprising pure Aquivion, has lower elastic modulus (reflecting the lower crystallinity and lower rigidity than Torlon-supported membranes) and shows an increased capability to manage mechanical stress leading to important elongation before failure (about 200%).

Extruded membrane has two different behaviours in the two processing directions (machine and transversal) leading to different stress and strain at break values. The more rigid and brittle direction (MD) has lower elongation but sustain higher traction stress than recast membrane whereas the softer and more elastic direction (TD) has elongation at break comparable with recast membrane but outperforms it in terms of stress at break (Fig.24).

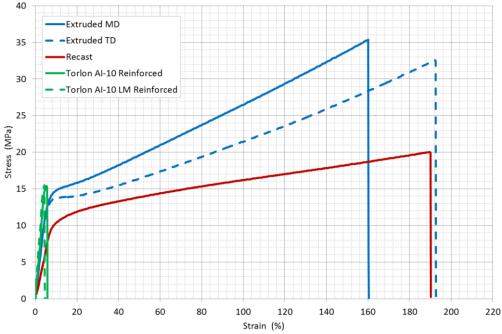


Fig.24: Stress-strain curves of reinforced (Torlon AI-10 and Torlon AI-10 LM), recast and extruded membranes

4. CONCLUSIONS AND FUTURE WORK

In this study three different Aquivion-based membranes (namely extruded, recast and Torlon reinforced) have been prepared and characterized on the basis of the KPI (proton conductivity, mechanical toughness, dimensional stability in hot water and water uptake)⁵ we consider as pivotal to design a membrane able to meet the challenging requests of next-generation PEM electrolysis.

Torlon-based reinforced membranes were developed starting from the selection of the most suitable grade, among the many commercial, for this application then force-spun supports were produced and used to prepare reinforced membranes.

Although promising these membranes seem not mature enough to be used as material in the Neptune final stack because there is still some way to go in terms of conductivity and support manufacturing to reach similar levels of more consolidated grades such as extruded and recast (Fig.25).

In the next future, efforts will be targeted on the further development and optimization of extruded and recast grades with a special focus on quality insurance of roll-to-roll manufacturing at large volume.

Final decision about the most suitable membrane grade (between extruded and recast) for Neptune final stack will be taken, accordingly to the description of activity, at M18 (July, 2019).

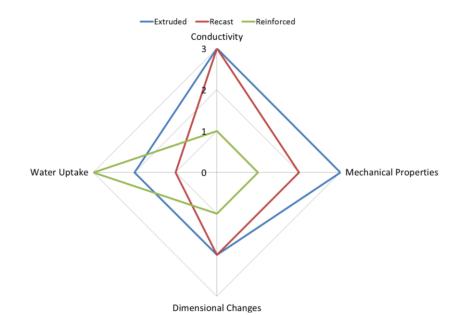


Fig.25: Comparison of the three Aquivion-based membranes considered in this study according to the KPI taken in consideration (proton conductivity, mechanical properties, dimensional stability, water uptake). 1: Low; 2: Medium; 3: High.

5. References

- 1. https://www.solvay.com/en/products/brands/torlon-pai
- 2. (a) Peng, N.; Chung, T.S. *J. Membr. Sci* **2008**, *310*, 455-465. (b) Han, G.; Chung, T.-S.; Weber, M.; Maletzko, C. *Environ. Sci. Technol.* **2018**, *52*, 3676-3684.
- 3. Sarkar, K.; Gomez, C.; Zambrano, S.; Ramirez, M.; de Hoyos, E.; Vasquez, H.; Lozano, K. *Materials Today* **2010**, *13*, 12-14.
- 4. https://www.solvay.com/en/products/brands/aquivion-pfsa.
- 5. Siracusano, S.; Oldani, C.; Navarra, M.A.; Tonella, S.; Mazzapioda, L.; Briguglio, N.; Aricò, A.S. *J. Memb. Sci.* (accepted for publication)

6. APPENDIX

LIST OF ACRONYMS

ABBREVIATION	UNIT OF MEASUREMENT	EXPLANATION
ASTM		AMERICAN SOCIETY FORTESTING AND MATERIALS
E-PTFE		EXPANDED POLYTETRAFLUOROETHYLENE
EW	g/mol	EQUIVALENT WEIGHT
GSM	g/m ²	GRAM PER SQUARE METER
КРІ		Key Performance Index
MD		MACHINE DIRECTION
MEA		MEMBRANE ELECTRODE ASSEMBLY
NMP		N-METHYL PYRROLIDINONE
OIS		OPTICAL INSPECTION SYSTEM
PAI		POLYAMIDEIMIDE
PEM		POLYMER ELECTROLYTE MEMBRANE
PET		POLYETHYLENETEREPHTALATE
PFSA		PerFluoroSulfonic Acid
PTFE		POLYTETRAFLUOROETHYLENE
RH	%	RELATIVE HUMIDITY
SCCM	cm³/min	STANDARD CUBIC CENTIMETERS PER MINUTE
SEM		SCANNING ELECTRON MICROSCOPY
SFVE		SULFONYLVINYL ETHER
TD		TRANSVERSAL DIRECTION