Characterisation of polyurethane, epoxy and silicone-based sealants and adhesives for their potential use in vanadium redox flow batteries

Andreas, Arlt', Max, Poxleitner, Ralf, Weishaupt
Business Development, Wevo-Chemie GmbH, Ostfildern, Germany
Email: andreas.arlt@wevo-chemie.de

1 Introduction

Different 2-component polyurethane, epoxy and silicone sealants and adhesives (Fig.1) have been characterised for the potential use in vanadium redox flow batteries by testing their chemical resistance in vanadium containing sulfuric acid-based electrolyte in different oxidation levels of vanadium (V2+, V3,5+ and V5+) at different temperatures from 22 to 40 °C for a period of 135 days.

Circular test specimens of cured polymers with diameters of 55 mm and 70 mm and a thickness of 10 mm have been immersed in the V2+ and V3,5+ electrolyte at 40 °C and the V5+ electrolyte at room-temperature (22 °C), representing the different charging levels of the electrolyte present during the operation of the flow battery. After immersion, the test specimens have been optically inspected by means of a light-microscope at 50x and 175x magnification for surface changes and possible dimensional changes (Fig. 4).

Additionally, the change of the shore hardness and the weight change of the test specimen (Fig. 2) as well as the colour change and the transparency of the electrolyte before and after the immersion (Fig. 5) have been determined. A significant change of weight and dimension is either a result of swelling and/or undesired oxidation processes and therefore a weak chemical resistance. An optical change of the polymer and the electrolyte is an indication of an undesired interaction with the electrolyte and oxidative or reductive stresses.

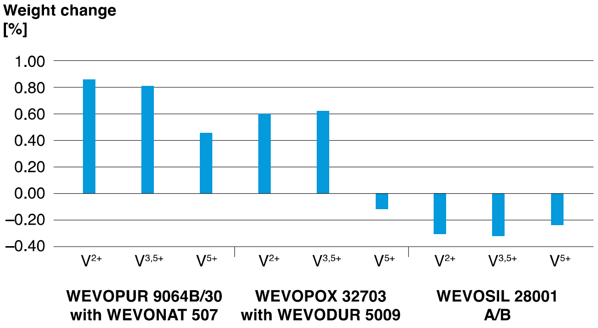
2 Tested material properties

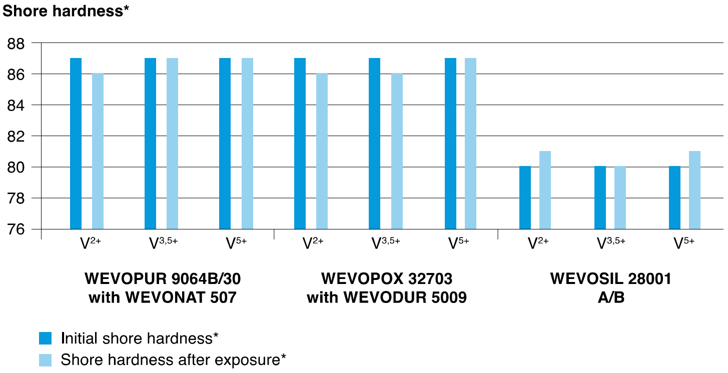
	Test standard	**WEVOPUR 9064B/30 with WEVONAT 507**	**WEVOPOX 32703 with WEVODUR 5009**	**WEVOSIL 28001 A/B**
Type		2-component room temperature curing polyurethane resin	2-component hot curing epoxy resin	2-component room temperature curing silicone sealant
Mixing ratio (part per weight)		100 : 35	100 : 12	100 : 100
Mixed viscosity [mPa·s]	Rotational viscosimeter	1,200–1,600	5,000–8,000	30,000–60,000
Pot life at 22 °C [min.]		app. 30	app. 30 min@120 °C	60–90
Density A component [g/cm^3]	DIN EN ISO 2811-1:2016-08	2.02–2.08	1.65–1.70	1.28–1.32
Density B component [g/cm^3]	DIN EN ISO 2811-1:2016-08	1.20–1.24	1.00–1.04	1.28–1.33
Shore hardness	DIN EN ISO 7619-1:2012-02	85–92 D	80–90 D	60–70 A
Operating temperature [°C]		–30 up to +140	–40 up to +155	–60 up to +200
E modulus [N/mm^2]	DIN EN ISO 527-2:2012-06		5,000	4.5
Elongation [%]	DIN EN ISO 527-2:2012-06		1	100
Glass transition temperature [°C]	TMA ISO 11359-2:1999-10	88	117	–55
Coefficient of thermal expansion [ppm/K]	TMA ISO 11359-2:1999-10	63 (< 85 °C) 195 (> 90 °C)	53 (< 110 °C) 157 (> 120 °C)	145 (< –55 °C) 210 (> –55 °C)
Water absorption [%]	30 days at 22 °C	0.3	0.2	< 0.2
Dielectric strength [kV/mm]	DIN EN 60243-1:2014-01	31		> 30
Dielectric constant at 50 Hz, 22 °C	DIN EN IEC 62631-2-1:2018-12	4.9	5.1	3.1
Dissipation factor at 50 Hz, 22 °C	DIN EN IEC 62631-2-1:2018-12	0.05	0.02	0.013
Volume resistance at 23 °C/50 % r. h. [Ω · cm]	DIN EN 62631-3-1:2017-01	10^{15}	10^{15}	10^{14}
Surface resistance at 23 °C/50 % r. h. [Ω]	DIN EN 62631-3-2:2017-01	10^{15}	10^{14}	10^{14}
Target application		anti-corrosive coating, encapsulation, potting	anti-corrosive coating, encapsulation, potting	sealing, encapsulation, potting

Figure 1: Process parameters, mechanical and electrical properties of the tested polymers

3 Chemical stability test

The weight and the shore hardness of the test specimens have been tested before and after the electrolyte exposure (Fig. 2 and Fig. 3).





* Values for WEVOPUR and WEVOPOX in shore D and WEVOSIL in shore A.

Figure 2: Weight change of polymer test specimens after exposure to the V electrolyte

Figure 3: Change of shore hardness of polymer test specimens after exposure to the V electrolyte

4 Microscopic pictures after chemical exposure

Figure 4 shows optical analysis of test specimens with a light microscope at a magnification of 50x and 175x before and after exposure to the different electrolytes. The test specimens have been scratched by means of a cutter knife with a cross-shaped pattern. The more visible the cross pattern is and the sharper the lines after electrolyte exposure are, the more stable the polymer is against the electrolyte.

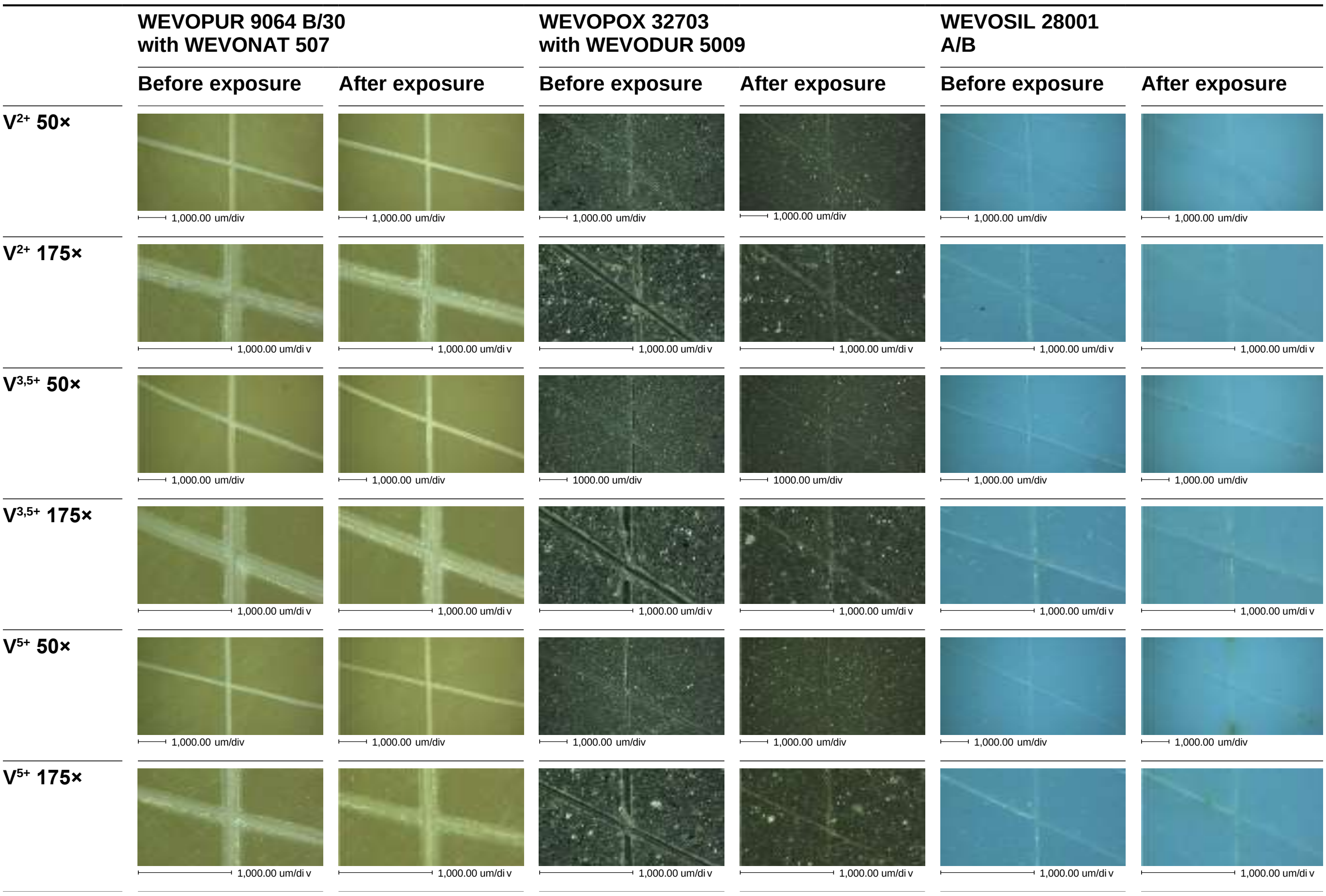
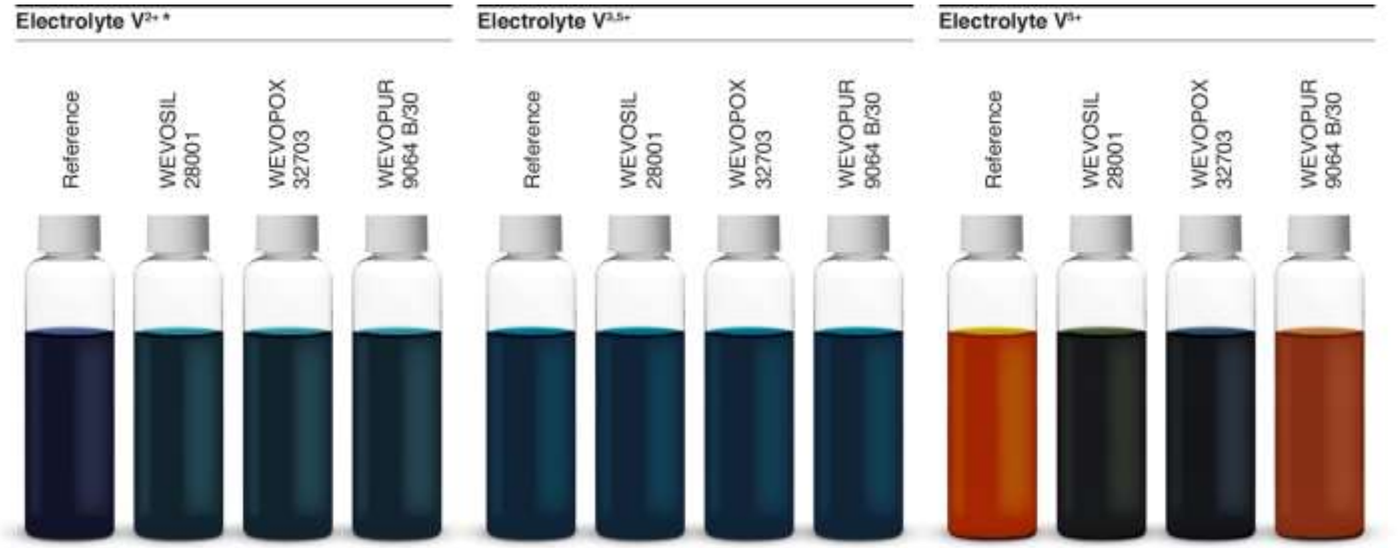


Figure 4: Optical-Microscope pictures at a magnification of 50 and 175

5 Electrolyte condition after chemical exposure

The electrolytes at different oxidation levels have been optically inspected concerning changes of their color and transparency (Fig. 5).



* The slight green change in the V^{2+} electrolyte might be due to oxidation via air oxygen.

Figure 5: Appearance of electrolyte at different oxidation levels before and after chemical exposure of test specimen

6 Conclusions

Due to the minor influence on the colour and the transparency of the different electrolytes and the very small changes of the shore hardness, dimension, weight and its minor surface alteration, the silicone sealant WEVOSIL 28001 and the polyurethane resin WEVOPUR 9064 B/30 can be regarded as very resistant against the aggressive V electrolyte at all oxidation level. This makes making them suitable as adhesives, encapsulation materials and sealants for the assembly of flow battery stacks. The polyurethane resin could also be used as a compound for full stack encapsulation and as an anti-corrosive coating for the protection of the bus bars of the stack. The epoxy resin WEVOPOX 32703 showed no influence on the colour of the V^{2+} and $V^{3,5+}$ electrolytes, and a slight influence on the colour of the V^{5+} electrolyte and the test specimen didn’t show any significant optical and dimensional changes after exposure. Due to its low weight change and stable shore hardness, it can be regarded as chemically stable and electrolyte-resistant, making it suitable as an anti-corrosive coating for bipolar plates and bus bars and as a potting compound for the full encapsulation of the battery stack.