Chemical Cleaning of Water-Cooled Generators: Effect on System Materials

Thomas Bauer, Matthias Svoboda, Sindy Dockheer, and Robert Svoboda

ABSTRACT

As an option to return efficiency to and avoid damage in water-cooled generators, plugged hollow conductors are cleaned using various methods. Chemical cleaning employs either acids or chelating agents. For this to be efficient, the cleaning solution should contain an oxidizer. However, these solutions attack the system materials more than those without an oxidizer. A study was conducted in which stator cooling water system materials were exposed to commonly used chemical cleaning agents and their corrosion resistance was analyzed. Of the investigated solutions, ammonium persulfate and a mixed acid solution with sulfuric acid, phosphoric acid and hydrogen peroxide were by far the most aggressive, followed by acids without an oxidizer. The least aggressive chemicals with regard to the generator materials are based on chelant cleaning, provided it is carried out in a carefully controlled and coordinated manner.

INTRODUCTION

Power plant operators face severe problems when dealing with reduced cooling efficiency in large water-cooled generators. Often this is caused by plugging of copper hollow conductors. The copper oxides plugging the generator windings can be removed by chemical cleaning. For this to be efficient, the cleaning solution should contain an oxidizer.

The subject of the present paper is an analysis of the corrosion resistance of generator water cooling system materials when exposed to different chemical cleaning agents. The most aggressive solutions were ammonium persulfate and a mixed acid solution with sulfuric acid, phosphoric acid and hydrogen peroxide, followed by acids without an oxidizer. The least aggressive chemicals are based on chelant cleaning, provided it is carried out in a carefully controlled and coordinated manner.

BACKGROUND

Since the early 1960s the stator coils of large generators are usually water cooled. To guide the water, the stator bars are equipped with hollow conductors of small cross section, typically in the order of 1.5 x 5 mm. With copper hollow conductors, copper oxides are formed. These are always a mix between CuO and Cu₂O, while CuO is predominant in high-oxygen chemistry systems and Cu₂O

with low-oxygen chemistry. Depending on the operating history and design, these oxides may eventually plug up the hollow conductors, which in turn leads to reduced cooling efficiency, load limitations, forced outages and/or damage to the generator [1–4]. This is especially an issue when the generators become older.

As an option to postpone or even avoid a rewind due to plugged hollow conductors, various cleaning methods are applied [2,5,6]. Chemical cleaning employs either acids or chelating agents (Figure 1).

CuO is readily dissolved in acids and with suitable chelating agents (i.e. based on EDTA (ethylenediaminetetraacetic acid)).

Cu₂O however is more difficult to dissolve chemically. Dilute solutions of hydrofluoric, sulfuric and phosphoric acids, and also the principal organic acids, dissolve Cu₂O with disproportionation into Cu⁺⁺ ions and metallic Cu [7,8]. Our own investigations indicate that this can also be expected from chelating agents. In consequence, efficient chemical cleaning of copper hollow conductors also employs oxidizing agents.

The question arises as to the possible side effects of these chemical cleaning agents on the stator bars and other materials in the stator cooling water system (SCWS).

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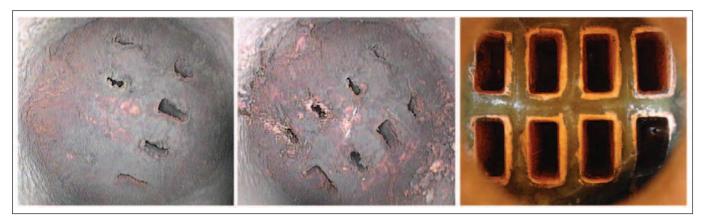


Figure 1: Water box before (left) and after (middle) mechanical cleaning; right: after subsequent chelant cleaning. Note that all pictures were taken of the same water box (T57).

Previous Investigations

In 2003, Ontario Power analyzed the Cuproplex® on-line process [6] regarding the stability of materials found within the SCWS when exposed to the chemicals in use, employing a highly diluted EDTA solution and H₂O₂. A previously used citric acid cleaning method (without oxidizer) was analyzed as a comparison [9]. It is outlined that citric acid attacks both the copper and the solder material; however, the attack remains within the corrosion allowance specification by the OEM, allowing for 2–3 cleanings during the lifetime of the machine. The Cuproplex® on-line process barely attacked the tested system materials and, following the OEM's specifications, could be applied as often as 80 times while staying within the corrosion allowance.

These different corrosion effects are also reflected in the amount of copper removed during the two different chemical cleaning procedures. The Cuproplex® on-line process removed 2.8 kg of copper from the entire stator cooling water system (SCWS), including 72 stator bars (and also lowered the stator bar temperatures by 5 °C), whereas the citric acid cleaning removed 4.4 kg of copper from only 24 stator bars with more than 50 % deriving from metallic copper. With extrapolation to 72 bars, this would result in 13.2 kg of copper, meaning that the citric acid cleaning removed at least 4 times more copper from the bars than the on-line process (assuming that all copper removed was from the bars).

Scope of the Present Investigation

The scope is to extend the findings of the Ontario Power research to other common cleaning agents as well as to higher concentrations of EDTA.

EXPERIMENTAL SETUP

Sample Preparation

All samples were prepared and analyzed as follows: samples were cleaned with H_2O (conductivity below 0.1 μ S·cm⁻¹), an organic solvent (either ethanol or acetone) and again with H_2O . The sample was then placed into the sample holder (Figure 2, right) and rotated at 200 rpm in the corresponding solution with given parameters (Figure 2, left; Table 1). Afterwards, the sample was removed, washed again as before testing, and analyzed accordingly.

Cleaning Solutions

The samples were exposed to 5 different acid-based and 3 different chelating-based cleaning solutions.

To have comparable results, all acid-based solutions were treated similarly with a duration of 120 min, a temperature of 40 °C and a chemical concentration (weight) of 5 %. The only exception was the mixed acid solution (Mixed Acid) with 5 % phosphoric acid, 10 % sulfuric acid and 2.5 % hydrogen peroxide as oxidizing agent. The citric acid exposure duration is far below the previously mentioned tests by Ontario Power and thus the results might differ, but they still deliver the benefit of reasonably comparable values with the other acid cleaning methods.

The chelant concentrations were chosen to exceed common concentrations and durations by a safe margin. The concentration has to be chosen on a case-to-case basis and depends on several process parameters (e.g. SCWS flow, mixed-bed flow, operating history, clogging severity, etc.).

To prevent artifacts in the measurement, the samples were also exposed to aqueous solutions without chemicals (BLANK).





Figure 2: Left: Test setup – the sample is rotated in a sample holder. N_2 was bubbled through the solution via the central connection of the lid. Right: Sample holder.

Chemical	Concentration [g · kg ⁻¹]	Duration [min]	Temperature [°C]	Identification			
-	_	2 880	40	BLANK			
EDTA	2	2 880	40	EDTA-1			
H ₃ PO ₄	50	120	40	Phosp. Acid	Without oxidizing agent		
H ₂ SO ₄	50	120	40	Sulf. Acid			
Citric acid	50	120	40	Citric Acid			
(NH ₄) ₂ S ₂ O ₈	50	120	40	APS Acid			
H ₃ PO ₄	50						
H ₂ SO ₄	100	120	40	Mixed Acid			
H ₂ O ₂	25				With oxidizing agent		
EDTA/H ₂ O ₂	2	1 500	40	EDTA-2			
EDTA/H ₂ O ₂	50	50	25	EDTA-3			
EDTA	1	29 000	40	EDTA-4			

Table 1:

Chemical solutions used for the exposure of the samples.

MATERIALS TESTED

Copper and Cooler Material

As representative for the bare copper, Cu-OF-CW008A (<u>Table 2</u>) was chosen. It is oxygen and phosphorous free with a purity of \geq 99.95 % and is widely used within generator stator and rotor bars. As a standard representative of cooler material, CuNi 90/10 was used.

In addition to visual and gravimetric analysis, the solutions with the copper samples were also analyzed for their copper concentration after exposure.

Braze and Solder Materials and Samples

A major concern when chemically cleaning the stator cooling water system is dealing with leakages at the braze/solder connections (water box leakages). Four representative materials were analyzed, ranging from copper (Copperflow 3) over Copper/Silver (Silfos 5, Silfos 15) to Silver/Cadmium/Copper/Zinc (Easyflow 2) based materials. The brazes Copperflow 3 and Silfos 5 came as brazing foils, while the solders Silfos 15 and Easyflow 2 were tested as rods.

Trade Name	Chemical Composition	Sample Name							
Stator winding									
Cu-OF-CW008A	Cu ≥ 99.95 %	Cu							
	Cooler materials								
CuNi 90/10	Cu 90 %, Ni 10 %	CuNi 90/10							
	Brazes and solders								
Copperflow 3	Cu 93.8 %, P 6.2 %	Copperflow 3							
Silfos 5	Ag 5 %, Cu 89 %, P 6 %	Silfos 5							
Silfos 15	Ag 15 %, Cu 80 %, P 5 %	Silfos 15							
Easyflow 2	Ag 42 %, Cu 17 %, Zn 16 %, Cd 25 %	Easyflow							
Ві	razed and soldered samples								
Cu/Copperflow 3	Cu/Copperflow 3 Cu/Cu 93.8 %, P 6.2 %								
Cu/Silfos 5	Cu/Ag 5 %, Cu 89 %, P 6 %	Cu/S5							
Cu/Silfos 15	Cu/Ag 15 %, Cu 80 %, P 5 %								
Cu/Easyflow 2	Cu/Ag 42 %, Cu 17 %, Zn 16 %, Cd 25 %	Cu/Easyflow							
	Rotating pump seal								
Impregnated graphite seal ring	DIN EN 12 756	Graphite SR							
Steel									
Stainless steel 316	Cr 16–18.5%, Ni 10–14%, Mo 2–3%, Fe	Stainless steel							
	Polymers								
Nitrile-butadiene-rubber	Nitrile-butadiene-rubber	NBR							
Teflon	Polytetrafluoroethylene	Teflon							
Viton	Viton	Viton							
Ethylene propylene diene monomer rubber	Ethylene propylene diene monomer rubber	EPDM							
C	Cu plates glued with Loctite								
Cu/Loctite 7400	Cu/Loctite	Loctite							
	Cu-Epoxy alloy								
Cu/Epoxy-1	Cu/Epoxy	Epoxy-1							
Cu/Epoxy-2	Cu/Epoxy	Epoxy-2							

Table 2: Sample materials, their trade names and chemical compositions.

For the brazed samples, two copper plates were brazed together, using induction brazing, while the soldered samples were torch brazed.

Leakages in the winding are sometimes sealed with epoxy resin or Loctite. Their chemical stability was also in the scope of the investigation and is discussed in detail below.

Rotating Pump Seals, Stainless Steel and Polymer Samples

During chemical cleaning, leakages were occasionally observed at the stator cooling water pump seals and thus an antimony impregnated graphite seal ring (material code AQ1VGG (DIN EN 12 756)) was added to the test program. Due to the chemical similarity of the metal counter ring to stainless steel, only the latter was analyzed.

A standard 316 stainless steel was analyzed, representing the majority of SCWS materials found. Additionally, one OEM has stainless steel hollow conductors to successfully prevent copper oxide plugging [10].

Polymer materials can be found in the SCWS, ranging from Teflon hoses to gaskets and O-rings. Four different polymers were chosen as representative examples: Viton, nitrile-butadiene-rubber (NBR), polytetrafluoroethylene (Teflon) and ethylene propylene diene monomer rubber (EPDM).

Loctite and Epoxy Resin Samples

The surface of a copper plate was cleaned with H_2O , acetone and H_2O again. Loctite 7400 was placed on one copper plate and then sandwiched with a second plate, letting it dry for 48 hours. The Cu samples for epoxy application were cleaned and subsequently an epoxy layer was placed on top of the copper plate. For comprehensive results, two different commonly used epoxy-based resins were analyzed.

Analysis Methods

Gravimetric analysis before and after exposure to the different solutions of all samples was done with an accuracy of \pm 0.5 mg. The corresponding corrosion in mm was calculated from the gravimetric loss and the sample surface. Analysis of the dissolved copper concentration was performed with inductively coupled plasma optical emission spectrometry (ICP-OES); additional visual observation of the samples and their corresponding solutions was also recorded.

RESULTS

Copper and CuNi 90/10 Cooler Material

Significant corrosion and weight losses were observed for the copper and CuNi 90/10 samples in the two acid solutions containing an oxidizing agent, APS Acid (ammonium

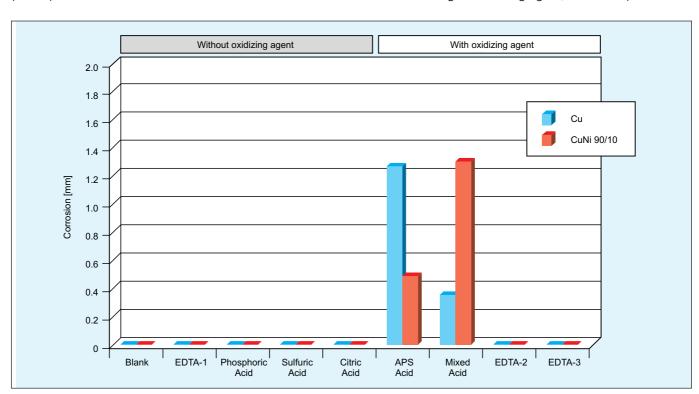


Figure 3: Calculated corrosion in mm of copper and cooler material in different chemical cleaning agents without/with oxidizer.

persulfate) and Mixed Acid (Figure 3). While in the latter solution, the corrosion measured for the copper sample was 0.48 mm and for CuNi 90/10 1.26 mm (6 and 20 % weight loss, respectively), an opposite corrosion behavior was observed in the APS solution: 1.30 mm for the copper (20 % weight loss) and 0.48 mm (8 % weight loss) for CuNi 90/10.

Visual inspections (<u>Figure 4</u>) of the copper plates confirmed the weight loss and color change of the solution and indicated harsh attack by Mixed Acid as well as APS

Acid. Additionally, also the surfaces of the Phosphorus and Sulfuric Acid samples showed significant visual changes compared to the blank solution.

Although no significant gravimetric change was observed for Sulfuric and Phosphoric acid, the copper concentration was > 10 times higher within the solution compared to the blank sample. In agreement with the weight analysis, the Mixed Acid and APS Acid solutions produced much higher copper concentrations (<u>Table 3</u>), indicating attack on the metallic sample.

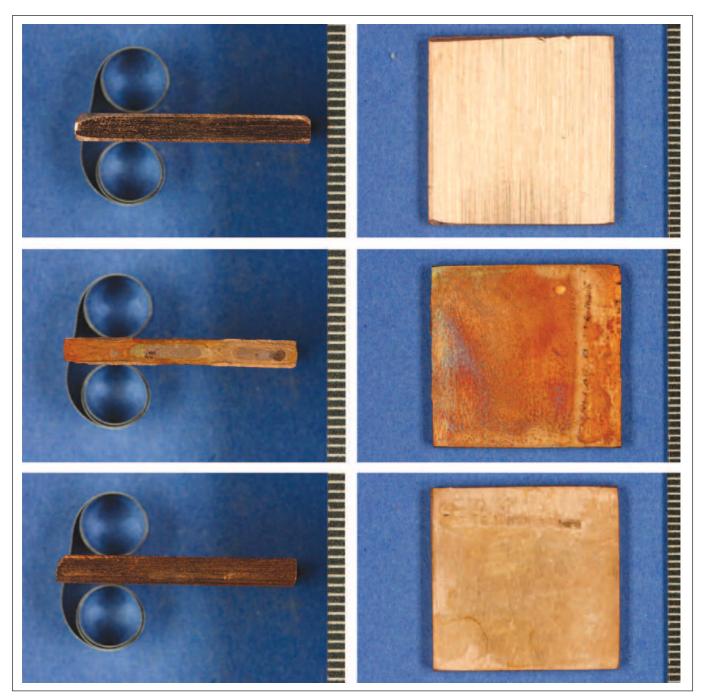


Figure 4: Visual inspection of copper plates. Representative samples from top: EDTA-2, Mixed Acid and APS Acid.

Without Oxidizer						With Oxidizer			
	BLANK	EDTA-1	Phosp. Acid	Sulf. Acid	Citric Acid	APS Acid	Mixed Acid	EDTA-2	EDTA-3
Cu [mg·kg ⁻¹]	0.34	0.7	4.6	3.7	0.5	4 739	105	1.4	0.01

Table 3:

ICP-OES data of analyzed samples.

Braze and Solder Materials

All analyzed brazes and solders were heavily attacked by both APS and Mixed Acid, the latter dissolving more than 90 % of Silfos 15. If an oxidizing agent (H_2O_2) is added to an EDTA solution (EDTA-2), the "raw" braze can also be slightly attacked (<u>Figure 5</u>, EDTA-2, Silfos 15). This is in disagreement with the analysis of the brazed samples (see <u>Figure 6</u>), where no degradation was observed. The Ontario Power investigation did not observe any attack on braze or solder material. They investigated EDTA/ H_2O_2 and an inhibitor, which delays the reaction kinetics of H_2O_2 [9].

Graphite Seal Ring, Stainless Steel and Polymer Samples

All of the investigated materials (graphite seal ring, stainless steel, NBR, Viton, EPDM and Teflon) stayed within the detection limit of ± 5 mg.

Brazed and Soldered Samples

The weight loss of the brazed and soldered samples was comparable with the analysis of the copper and bare brazes and solders. APS and Mixed Acid heavily attacked all samples (> 5 % weight loss). The corrosion for APS was 0.92–1.35 mm for the brazed and soldered samples and about three times higher for Mixed Acid samples (2.89–3.97 mm). This is also in agreement with visual inspection.

No changes were observed for the chelating agent based methods EDTA-2 and EDTA-3 with oxidizer as well as for the acid solutions without oxidizer.

Visual inspection (Figure 7) confirmed the results from the corrosion analysis. Both copper and braze were heavily attacked by the acid with oxidizing agent.

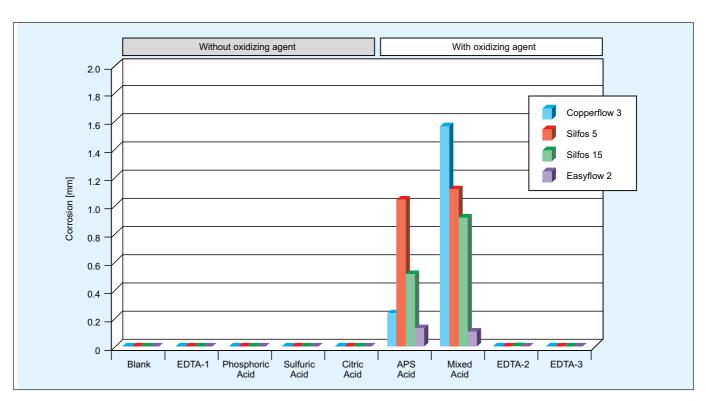


Figure 5: Calculated corrosion in mm of brazes (Copperflow 3 and Silfos 5) and solders (Silfos 15 and Easyflow 2) in 9 different chemical solutions without/with oxidizer.

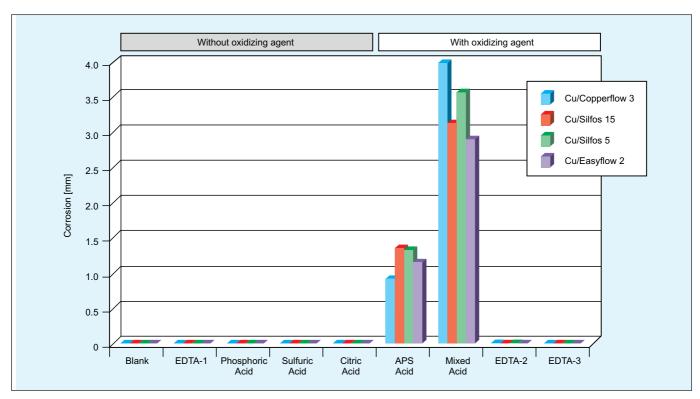


Figure 6: Calculated corrosion in mm of brazed and soldered, sandwich-type samples Cu/Copperflow 3, Cu/Silfos 5, Cu/Silfos 15 and Cu/Easyflow 2 in different chemical solutions without/with oxidizer.

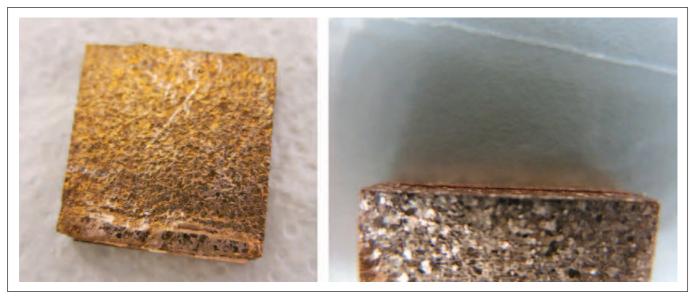


Figure 7:

Representative visual inspection of brazed and soldered copper samples (Cu/Copperflow 3 – left: Mixed Acid; right: APS) including an oxidizing agent in the solution.

Loctite and Epoxy Resin Samples

The results obtained from the Loctite and epoxy samples are similar to those for the brazed and soldered samples. Again, the most aggressive chemical solution to the mate-

rial was the Mixed Acid followed by APS Acid. Acid-based cleanings with an oxidizer partly separated the epoxy from the copper surface (Figure 8). For the Cu-Loctite samples,

both copper and Loctite were attacked and dissolved by acid-based cleanings with oxidizing agent. This was not observed for the EDTA-based cleanings, even if an oxidizing agent was present (EDTA-2, EDTA-3).

A long-term, EDTA-based process (EDTA-4) was already validated by the Ontario Power investigation [9]. To extend their observations, epoxy- and Loctite-based samples were analyzed regarding their corrosion parameters. No weight loss or any visual changes were observed for the epoxy and the Loctite samples (see Figure 9).



Figure 8: Visual inspection of Cu-Epoxy 1 (left, right top) and Cu-Loctite (right bottom) samples in Mixed Acid solutions.

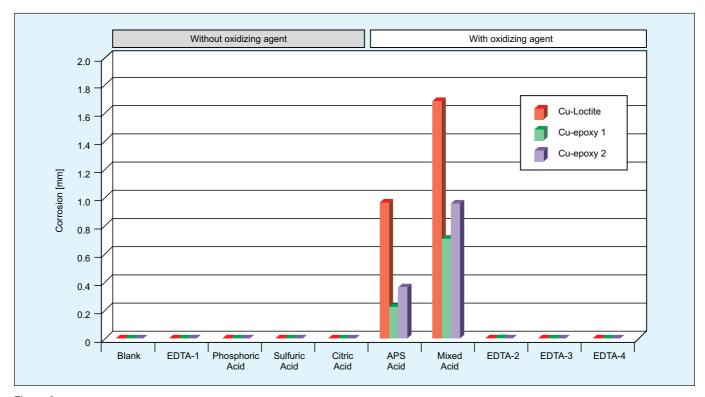


Figure 9:

Calculated corrosion in mm of Cu-Loctite, and two different Cu-epoxy glued samples in different chemical solutions. An additional EDTA-based process (EDTA-4) was also analyzed and validated for the epoxy resins and the Loctite samples without/with oxidizer.

SUMMARY AND CONCLUSION

For efficient cleaning, the chemical solutions should contain an oxidizer. However, these solutions attack the system materials more strongly than those without an oxidizer.

APS Acid (ammonium persulfate) and the Mixed Acid (sulfuric acid, phosphoric acid and hydrogen peroxide) solutions were by far the most aggressive, followed by acids without an oxidizer. The least aggressive chemicals with regard to the generator materials are based on chelant cleaning, provided it is carried out in a carefully controlled and coordinated manner.

Polymers, stainless steel and graphite seal rings are attacked neither by acid nor by chelating agents.

The corrosion of all samples is summarized in <u>Table 4</u> and classified into solutions with and without the presence of

an oxidizing agent. Besides the obvious heavy attack of APS Acid and Mixed Acid on different materials, ICP-OES analysis of the solution showed that Phosphorus and Sulfuric Acids also attacked the Cu samples.

Regarding corrosion limits, similar results were published by Ontario Power. The corrosion allowance specified by the OEM was already exceeded after 3 citric acid cleanings whereas and EDTA-based process barely attacked the tested materials and thus could be applied more than 80 times before reaching the corrosion limits.

Summing up, amongst the solutions containing an oxidizing agent necessary for an efficient cleaning, only the EDTA-based solutions EDTA-2 and EDTA-3 exhibited a very low or even negligible corrosion rate.

Without Oxidizing Agent					With Oxidizing Agent				
	BLANK	EDTA-1	Phosp. Acid	Sulf. Acid	Citric Acid	APS Acid	Mixed Acid	EDTA-2	EDTA-3
Cu	0.00	0.00	0.00	0.00	0.00	1.26	0.35	0.00	0.00
Cu/Copperflow 3	0.00	0.00	0.00	0.00	0.00	0.92	3.97	0.02	0.00
Cu/Silfos 15	0.00	0.00	0.00	0.00	0.00	1.35	3.12	0.01	0.00
Cu/Silfos 5	0.00	0.00	0.00	0.00	0.00	1.32	3.55	0.02	0.00
Cu/Easyflow 2	0.00	0.00	0.01	0.00	0.00	1.15	2.89	0.01	0.00
Copperflow 3	0.00	0.01	0.01	0.01	0.00	0.23	1.56	0.01	0.01
Silfos 5	0.01	0.01	0.00	0.01	0.00	1.04	1.11	0.01	0.00
Silfos 15	0.00	0.01	0.00	0.01	0.00	0.51	0.91	0.02	0.00
Easyflow 2	0.00	0.00	0.00	0.01	0.00	0.14	0.13	0.00	0.00
CuNi 90/10	0.00	0.00	0.00	0.00	0.00	0.48	1.30	0.00	0.00
Graphite SR	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00
Steel	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00
Teflon	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
EPDM	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Viton	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
NBR	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Cu-Loctite	0.00	0.00	0.00	0.00	0.00	0.96	1.68	0.01	0.00
Cu-Epoxy 1	0.00	0.00	0.00	0.00	0.00	0.22	0.70	0.00	0.00
Cu-Epoxy 2	0.00	0.00	0.00	0.00	0.00	0.36	0.95	0.00	0.00

Table 4:

Calculated corrosion of samples in mm after exposure to different chemical solutions. White: no corrosion observed; dark grey: heavy corrosion. For details about test conditions and samples, see Tables 1 and 2, respectively. The detection limit for polymer samples is 0.05 mm due to the low sample weight and the detection limit of the weight analysis.

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