

Review

Corrosion in supercapacitors- An Overview

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Abstract: Corrosion of metals and carbon in its numerous forms and of further functional materials like metal oxides used as active masses and auxiliary materials in supercapacitors, contributes to both the ageing and degradation of supercapacitor components and complete devices of the various established types. Examples, symptoms and mechanisms of the ageing and degradation on the material/electrode and the device level are presented and discussed. Remedies and suggestions for improvements to avoid these unwanted contributions to device ageing and failure are indicated.

Keywords: Corrosion; carbon corrosion; metal corrosion; materials degradation; ageing

Nomenclature

Term	Description
AC	activated carbon
EDLC	electrochemical double layer capacitor
EES	electric energy storage
ESR	electrical series resistance
ICP	intrinsically conducting polymers
SC	supercapacitor

1. Introduction

Corrosion, commonly assumed to be the corrosion of metals (rust of steel as in [1]) with huge economic losses, is also a major challenge in devices and systems for electrochemical energy conversion and storage. Different from this popular understanding, it is not at all limited to the degradation of metals and their alloys, corrosion may affect basically all materials. Because corrosion of metals and other electronically conducting materials is an electrochemical process with the metal dissolution as the anodic reactions and various cathodic reactions (most prominent reduction of dioxygen in the presence of at least a minimum of moisture providing an electrolyte “solution” or of protons in acidic environments), electrochemists have studied intensely corrosion, its causes and mechanisms and approaches to limit corrosion [2 - 14]. This report provides an overview of the corrosion of active masses (i.e. materials participating in charge storage) and auxiliary materials and possible reme-

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dies in supercapacitors.

Supercapacitors are electric energy storage EES devices storing electrical energy either without any conversion by electrostatic storage or by fast superficial redox reactions at electrode surfaces. They provide high current capabilities and associated high power densities; unfortunately, their energy densities are still inferior when compared with available secondary batteries. Depending on the utilized charge storage mode in an electrode of a supercapacitor classifications of available systems, i.e. combinations of two electrodes, have been proposed. Basic modes of operation are:

1. Charge storage by charge separation in the electrochemical double layer;
2. Charge storage by redox reactions preferably close to the materials surface.

Both operating principles are shown schematically in Figure 1.

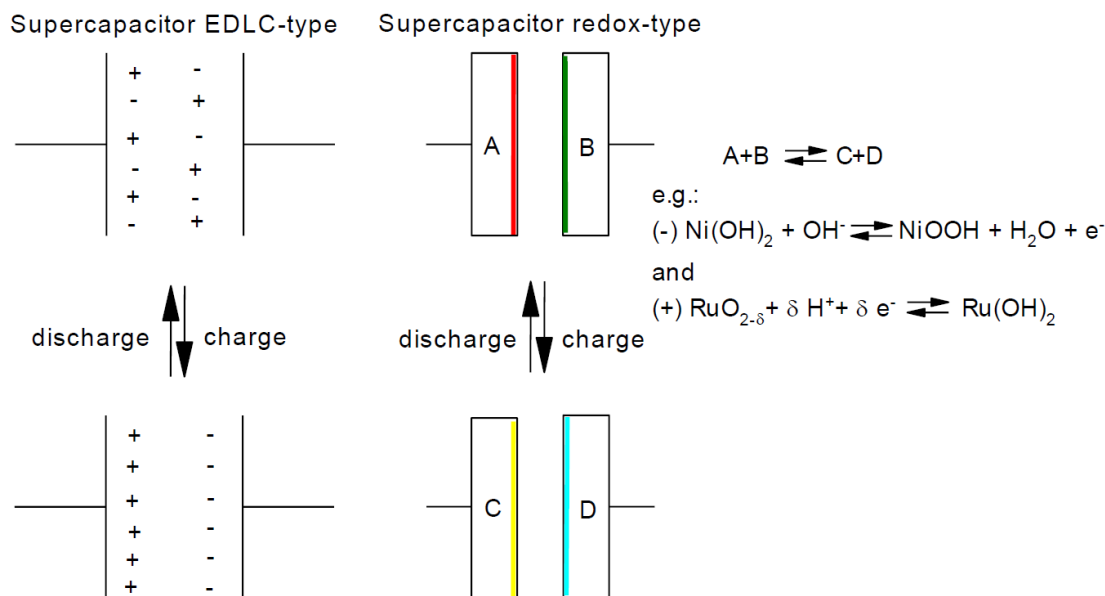


Figure 1. Charge and discharge of a supercapacitor of the electrochemical double layer capacitor (EDLC-type (left) and of the dox capacitor type (right).

The accumulation of ionic charges in solution in front of the electrode during charging of an electrochemical double layer capacitor (EDLC-supercapacitor) is indicated; the corresponding charge accumulation inside the electronically conducting electrode material is not shown explicitly. During discharge, dissipation of the ions proceeds as shown. In the case of the redox capacitor (names of these devices vary wildly; no commonly accepted name has been established [15]), redox transformations of the electrode materials near their surfaces are schematically indicated. The relationship with a secondary battery is obvious; but the transformations inside a battery electrode are not limited to a zone close to the surface as with the supercapacitor electrode. This ongoing merger of operating principles going beyond the materials has been discussed elsewhere [16 - 18].

The relationships between a supercapacitor (in the picture of the EDLC-type), a conventional plate capacitor, and an electrolytic capacitor are schematically illustrated in Figure 2. The scheme also includes simple electric equivalent circuits containing, in addition to the capacitance, the electrical series resistance (ESR), sometimes called the internal resistance or impedance.

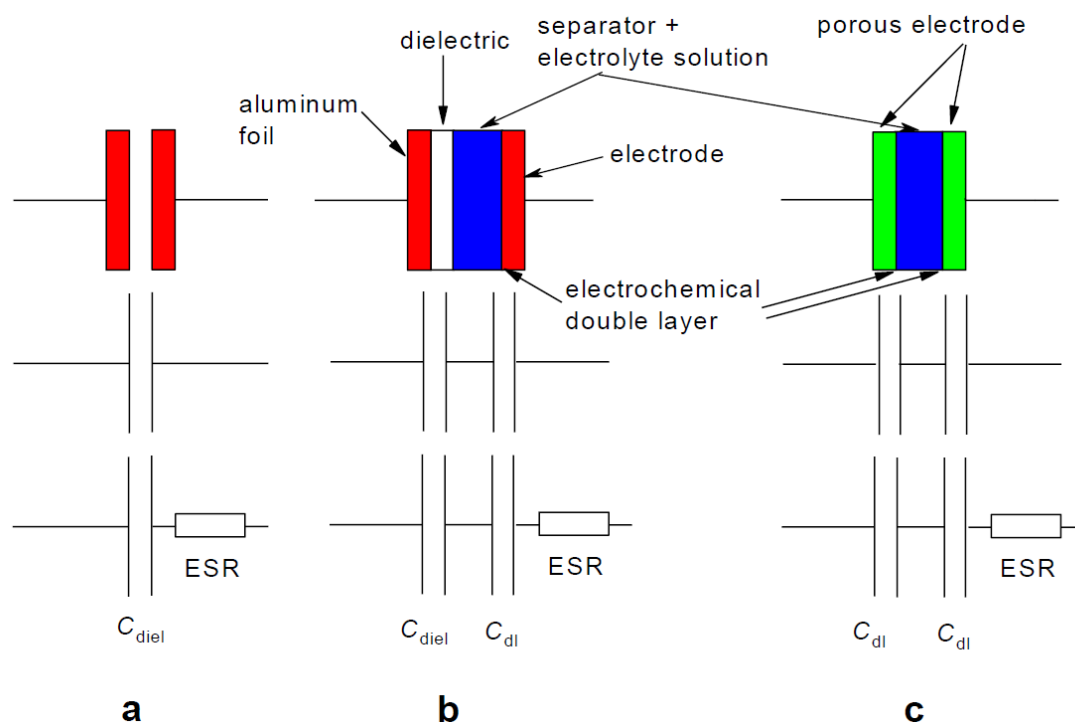


Figure 2. Schematic cross sections, simplified and extended equivalent circuits of (a) dielectric capacitor, (b) electrolytic capacitor; (c) electrochemical double layer capacitor. Inductive contributions/elements are not shown, they are of minor importance in most supercapacitor applications, C_{diel} : capacitance established between two metallic electrodes separated by a dielectric medium, C_{DL} : capacitance of the electrochemical double layer, ESR: electrical series resistance.

As shown in Figure 1, the combination of two electrodes of mode (or type) 1 with a separator between them and an electrolyte or electrolyte solution yields an electrochemical double layer capacitor (EDLC type). Combination of two electrodes of mode 2 (redox capacitor) yields a supercapacitor strongly resembling a battery. Because of the particular properties of the electrode materials, their electrochemical response to a changing electrode potential or cell voltage looks capacitor-like (pseudocapacitive), accordingly, the term pseudocapacitor (among many other terms) enjoys some popularity. Unfortunately, the term lacks logic. Because redox processes are essential, within this report the term redox capacitor is used as proposed elsewhere before [15]. The terminology and associated confusion are discussed elsewhere [19, 20]. When two electrodes operating with the same storage mode are combined into a cell, the device is called symmetric. Combination of electrodes of mode 1 and mode 2 yields an asymmetric device. Sometimes the term hybrid is applied; this has been put into perspective in previous discussions [21]. Typical examples of commercial supercapacitors of various shapes and capacitances are depicted in Figure 3.

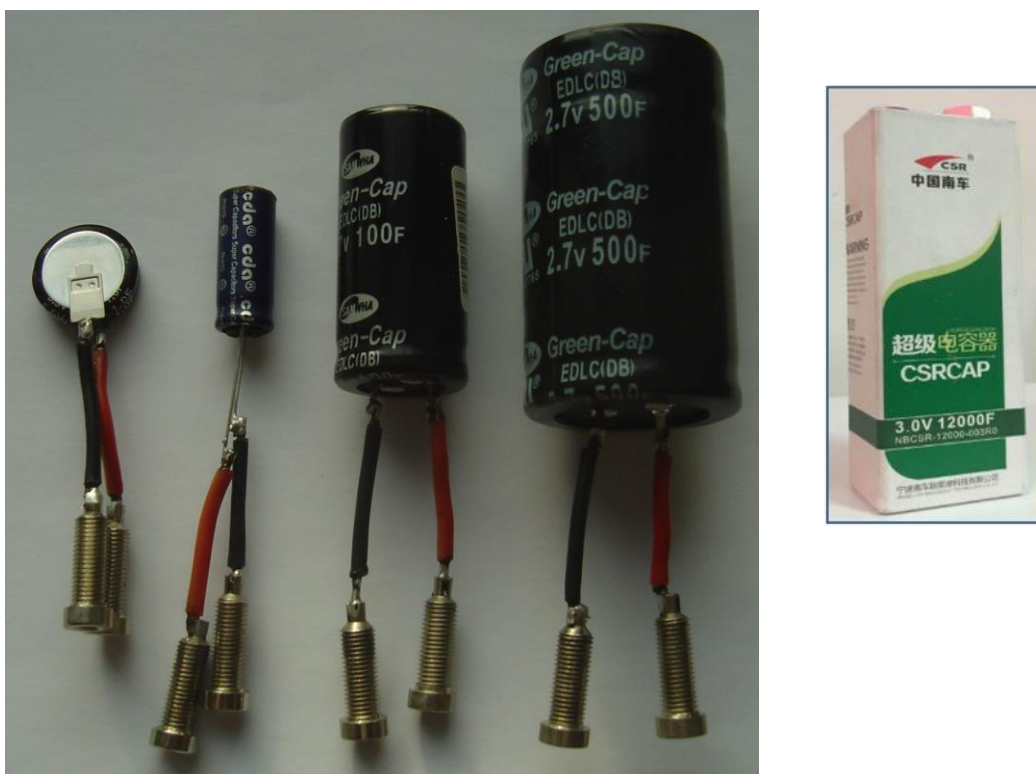


Figure 3. Typical examples of commercial supercapacitors of various shapes and capacitances.

Further development of materials, electrodes, devices and applications for supercapacitors has been reviewed repeatedly, see, e.g. [19, 22–45].

Despite the overwhelming importance of corrosion in many fields of science and technology, it has been recognized as a challenge and problem in supercapacitor research and development only rather late, although corrosion in other systems for EES has been taken into account already for many years. A literature search provided only a relatively small number of publications where the keyword corrosion appears in connection with supercapacitors or any of the infrequently used synonyms. Results are displayed in Figure 4. The actual total number decreases further when the role of corrosion in a particular study is inspected in more detail. Sometimes “corrosion” steps are involved in the preparation of a material (for examples, see [46 - 59]). The use of corrosive activators in the preparation of *N*-doped activated carbons (ACs) for EDLC-devices as an option to avoid later unwanted challenges has been examined [60]. Unwanted metal dissolution of the negative electrode in a metal-ion supercapacitor can also be called corrosion (as an example, see zinc corrosion in [61, 62]). Anyway, the awareness of the challenges posed by corrosion of active materials, current collectors and further auxiliary materials seems to be rather low, quite in contradiction to the frequently claimed longevity of supercapacitors, particularly of the EDLC-type. In very few cases, the author’s understanding of the term “corrosion” could not even be guessed [63].

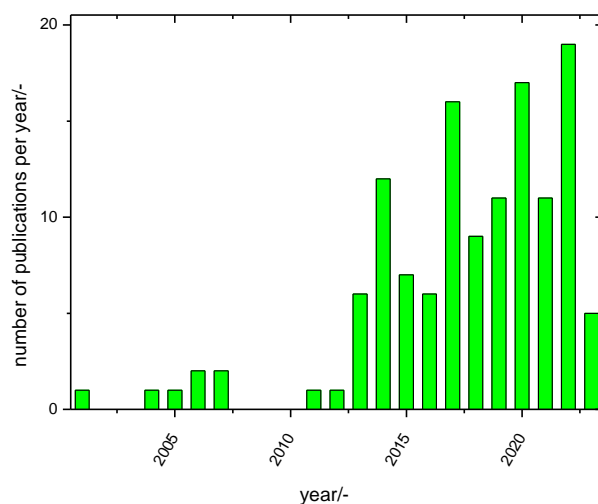


Figure 1. Annual publication numbers of reports with “corrosion” and “supercapacitor” anywhere in the title, keywords or abstract (Data from Web of Science[®] retrieved on April 30th, 2023). Further publications addressing this topic (mentioning the keyword(s)) somewhere in the text could not be counted; when noticed and considered relevant in the present context they were evaluated below nevertheless. The very few publications on “electrochemical capacitors” or “double layer capacitors” instead of “supercapacitors” were included; the associated confusion suggests once more systematic use of technical terms. More on this topic can be found in [19, 20].

Starting with the rather limited evidence provided in retrieved reports, in the following sections, observations, suggested causes and mechanisms, and proposed remedies are addressed, beginning with the material and component level and proceeding to the device level. Corrosion of packs and modules is not addressed because of the lack of reported observations.

Ageing of supercapacitors reviewed elsewhere [64] is significantly caused by corrosion processes (see e.g. [65, 66]). Self-discharge of supercapacitors is sometimes related to corrosion processes (see e.g. [67]); an overview also addressing this connection is available [15]. The compatibility of electrolytes with auxiliary materials in supercapacitors has been discussed in [68]. Some observations related to corrosion in supercapacitors have been addressed elsewhere [69, 70].

2. Corrosion of Carbon in Supercapacitors

2.1 Observations

Most supercapacitor performance and ageing studies addressed carbon corrosion; a review on the subject can be found elsewhere [64]. Indirect evidence is related to losses in performance, in particular losses of capacitance. More direct evidence in terms of gas evolution (CO₂) has been reported (e.g. see [71]). Direct carbon oxidation is rather unlikely, although it has been claimed earlier [72, 73]; instead, a chemical attack of carbon by reactive intermediates of electrochemical water oxidation like oxygen-containing radicals has been proposed. Related research results obtained in fuel cell research basically support this assumption [74 - 76]. Evidence of the formation of a possible protective “passivating layer” on carbon exposed to an aqueous deaerated solution of 0.5 M H₂SO₄ at electrode potentials positive to the one needed for oxygen evolution has been reported [77]. Specific sites and functional groups supporting corrosion have been examined with non-aqueous electrolyte solutions; the number of H-terminated edge sites and oxygen-containing functional groups correlated best with recorded electric charge due to corrosion [78].

2.2 Remedies

As has been concluded elsewhere [64], reducing the number of oxygen-containing functional groups may help improve the corrosion stability of carbons in EDLC devices [78]. A graphitic ordered mesoporous carbon has been identified as being remarkably stable against corrosion [79]. Considering the role of water in carbon corrosion, coating of carbon materials with electropolymerized polyacrylic acid has been suggested as an option to keep water away from the carbon, thus widening the cell voltage window of operation [80]. Attaching functional groups via diazonium chemistry to block corrosion-prone sites on carbon materials has been examined as a way to mitigate carbon corrosion and modify the wetting properties [81].

3. Corrosion of Other Active Masses in Supercapacitor Electrodes

3.1 Observations

The poor corrosion stability of a nanocomposite cobalt ferrite/graphene was attributed to its high electronic conductivity [82, 83]. Insufficient stability of many redox-active materials potentially of interest for application in supercapacitor electrodes in contact with acidic electrolyte solutions has been noticed [84].

A high (but unspecified) corrosion resistance has been attributed to some transition metal oxides recommending their use as active mass in redox supercapacitor electrodes [85]. Similar arguments have been provided for a composite of nitride/NiO/cobalt nitride [86] and for heterostructures of $\text{Co}_2\text{N-Ni}_3\text{N}$ on nickel foam [87]. The repeated claim for high corrosion stability of a core/shell polyaniline/graphene oxide/copper nanocomposite is not corroborated with experimental evidence [88]. The high corrosion stability of WO_3 as coating or part of active masses has been highlighted [89]. How the introduction of carbon powder into $\text{Ni}_{0.7}\text{Co}_{0.3}(\text{OH})_2$ could prevent corrosion could not be deduced from the report [90]. Some dependency of corrosion rate of RuO_2 on the shape and morphology of the oxide has been observed [91]. Related observations have been reported elsewhere [92]. How the corrosion potential of ternary composites PANI/rGO/ ZrO_2 prepared with different concentrations of the zirconium precursor in the preparation solution affected the rate capability of the material remained unexplained [93].

3.2 Remedies

Cobalt and its compounds are of interest because of high theoretical charge storage capabilities and because their corrosion susceptibility can be mitigated by coating with e.g. graphene [94]. A similar beneficial effect of a graphene coating has been observed with nickel phosphide, Ni_2P [95]. Coating of TiN in nanowire arrays for use as an electrode with a layer of carbon afforded corrosion protection [96]. Corrosion of the negative zinc electrode in a zinc-ion supercapacitor could be mitigated by using a bromide-containing hydrogel electrolyte [97]. The effect was attributed to the inhibition of dioxygen movement towards the negative electrode diminishing the cathodic partial reaction of zinc corrosion. Another mitigation effect was observed in a zinc-ion supercapacitor using a hydrogel electrolyte with adsorption of some hydrogel constituents on the zinc surface, inhibiting corrosion and supporting even metal deposition during charging [61]. Coating the negative zinc electrode with hydrophobic nano-silica reduced zinc corrosion and dendrite formation [62]. Co_3O_4 shows promising data for supercapacitors but suffers from poor electronic conductivity and inadequate stability; coating with ZnO ameliorated the latter drawback [98]. Trinickel disulphide (Ni_3S_2), having attractive storage capability, is prone to corrosion; this should be suppressed by coating with polyaniline [99].

The use of intrinsically conducting polymers (ICPs) in composites with redox active materials with attention to improved stability, i.e. suppressed corrosion, has been reviewed elsewhere [100, 101]. Polypyrrole (another ICP) electrodeposited on aluminium (actually into cracks of the Al_2O_3 immediately formed on the metal when brought into contact with the aqueous electrolyte solution) provided redox storage capacity and further corrosion protection [102, 103].

Coating of corrosion-prone silicon nanowires attractive for charge storage with silicon carbide afforded corrosion protection and enhanced stability [104]. Similar benefits were achieved with a coating with graphene [105]. High corrosion stability of silicon carbide, now used as support and current collector, has been stressed elsewhere [106, 107].

Corrosion of the negative zinc electrode in storage devices (zinc-ion batteries and capacitors) could be reduced by a porous coating with ZnO [108].

4. Corrosion of Auxiliary Materials in Supercapacitors

4.1 Observations

In an EDLC supercapacitor, corrosion products formed at the positive electrode of activated carbon with a stainless steel current collector accumulated between active mass and current collector possibly increasing the internal resistance [109]. With EDLC devices containing NaCl electrolyte solutions, corrosion of stainless steel and even gold current collectors has been noticed after a large current flow [110]. Formation of fluoro- and oxyfluoro-aluminium complexes as corrosion products of the aluminium foil current collector was observed with organic solvent-based electrolyte solutions [111]. In addition, pit formation has been reported for EDLC devices with such solutions [112].

Differences in corrosion activity for aluminium used as current collector between ionic liquids, *N*-butyl-*N*-methylpyrrolidinium bis(trifluoromethanesulfonyl)imide ($\text{PYR}_{14}\text{TFSI}$) and *N*-butyl-*N*-methylpyrrolidinium bis(fluorosulfonyl)imide ($\text{PYR}_{14}\text{FSI}$), the former one was less aggressive [113]. Possibly very small trace amounts of chloride ions in the latter ionic liquid were a cause for the difference. Further details can be found in [114].

With tetraethylammonium tetrafluoroborate electrolyte, formation of an electroconductive coating on the

aluminium foil current collector of the positive electrode was noticed [115]. Corrosion of the aluminium current collector at the positive electrode in lithium-ion batteries and supercapacitors has been discussed in the context of using bio-derived organic binders and aqueous processing of lithiated metal oxide electrode materials [116]. A comparative study of acetonitrile-based electrolyte solutions of alkali and alkaline earth metal salts with $\text{N}(\text{SO}_2\text{CF}_3)_2$ revealed excessive corrosion in the presence of magnesium cations [117].

The influence of current collector corrosion (stainless steel 316L was used) in contact with various electrolyte solutions on performance of an EDLC-type supercapacitor has been studied in detail [118]. Corrosion was most severe with alkaline electrolyte solutions because of the lowest protection by a passivation layer. A mixed water-dimethylsulfoxide (DMSO) solvent-based electrolyte solution showed low aluminium corrosion attributed to the DMSO [119]. Corrosion of the aluminium foil used as current collector for the negative electrode in an EDLC-device has been addressed; and protective coatings have been recommended as a remedy [120].

Corrosion of the NiCo foam used as current collector and electrochemical dissolution of the MnO_x used as active mass were identified as causes of cell performance degradation [121]. Metal nitrides, in particular TiN, have been claimed to show high corrosion resistance in supercapacitor applications [122]; see also [123]. Similar claims for metal oxynitrides [124, 125] and MXenes [126] have been reported. An electrode of polyaniline (PANI) on a support and current collector of hexagonal BN showed little corrosion [127]. A nanocomposite film of graphene/ Cu_2O on a copper foil afforded an increased corrosion resistance of the copper [128].

Water-in-salt electrolyte solutions enable a wider electrochemical window of stability for basically aqueous electrolyte solution systems keeping the advantages of these solutions. In a comparative study of steel, titanium and copper current collectors, copper was found not suitable, titanium offered an even wider potential window at the positive electrode whereas stainless steel enabled the same at the negative electrode [129].

4.2 Remedies

Corrosion of the stainless steel used in the positive electrode current collector and of the AC used in this electrode of a symmetric EDLC-type device with an aqueous 1 M Li_2SO_4 electrolyte was slowed down or even inhibited by addition of $0.1 \text{ M}\cdot\text{L}^{-1}$ sodium molybdate (Na_2MoO_4) [109]. This was attributed to a Faradaic reaction of the molybdate keeping the actual positive electrode potential below $E_{\text{SHE}} = 0.798 \text{ V}$. Protection by the added molybdate was also attributed to strengthening of the hydrated iron oxide layer, i.e. improved passivation. The beneficial effect of added molybdate was confirmed by these authors elsewhere again [130]. By comparison, a neutral aqueous Li_2SO_4 electrolyte solution was most suitable in terms of a wide window of stable cell voltage and low corrosion [131]. Elsewhere a neutral aqueous Na_2SO_4 electrolyte solution was found most beneficial regarding corrosion of current collector and cell case made from stainless steel in an EDLC-device [132]. A similar conclusion was reported for an aqueous 1 M Na_2SO_4 electrolyte [133]. A siloxane coating of the stainless steel current collector resulted in significantly lower corrosion when an aqueous 1 M Na_2SO_4 electrolyte was used in an EDLC-device [134]. Replacement of H_2SO_4 used as the electrolyte in an EDLC-device with heteropolytungstic acid $\text{H}_3\text{PW}_{12}\text{O}_{40}$ resulted in significantly reduced corrosion of the 316 stainless steel current collector [135].

Aluminium as a current collector in EDLC-devices is prone to corrosion in neutral aqueous electrolyte solutions; this drawback can be overcome by using a nickel-aluminium alloy instead [136]. Coating of aluminium foil with vanadium oxides or addition of phosphoric acid have been considered as options to prevent aluminium corrosion [116]. A coating with zirconium nitride has been as particularly corrosion-stable [137]. With organic solvent-based electrolyte solutions with LiTFSI as an electrolyte, corrosion of aluminium current collectors could be inhibited by adding 1 % of LiPF_6 [138]. Protection was attributed to formation of a protective layer of fluoroaluminates. In studies of an asymmetric supercapacitor with a negative AC electrode and MnO_2 as the positive electrode removal of dioxygen was found to decrease corrosion of the stainless steel current collector [139].

Single wall carbon nanotubes have been recommended as corrosion-resistant current collector replacement for metals in this function [140]. The meaning of anti-corrosion properties in this context remains unclear. In a slightly wider sense, carbon nanotubes (CNTs) have been recommended as corrosion-stable ingredients for (in particular for flexible and wearable) EDLC-devices [141]. Graphene has also been identified as a particularly corrosion-resistant component in supercapacitors [142]. Coating nickel foam first with carbon, then with PANI increased the corrosion resistance of nickel high enough to make its use in neutral or even slightly acidic electrolyte solutions (wherein bare nickel is not stable) possible [143]. For more related examples involving nickel foam see [144, 145]. SiC e.g. as nanowire array has been identified as corrosion-stable support material [146].

A spray-coated and corrosion-resistant current collector composed of silver nanowires and graphene oxide nanoflakes enabled a stretchable supercapacitor [147]. Coating of copper nanowire initially rather susceptible to corrosion can be inhibited by coating with an Ag-Au alloy shell [148]. Gold-coated polyethylene terephthalate polymer foil was (not quite unexpectedly) found to be very corrosion stable [149]. Aluminium coated with graphite has been proposed as a corrosion-resistant current collector [150]. Further coatings of aluminium foil for improved corrosion resistance and also improved electrode/device performance have been reported [68].

Coating of corrosion-susceptible metals with graphene has been suggested earlier [151]; further options of coating of current collectors for corrosion protection have been reviewed [152].

Replacement of aluminium with titanium as a current collector as suggested in [153] will be subject to economic considerations. General replacement of metallic current collectors by carbon-based materials (graphite foil, graphene-coated substrates) has been recommended to avoid corrosion problems [154]. Advantages of carbon fiber paper in addition to high corrosion resistance have been highlighted [155]. In zinc-ion capacitors, stainless steel foils are sufficiently stable and significantly cheaper than titanium foils [156].

5 Corrosion of Complete Cells and Devices

5.1 Observations

In a report on an asymmetric supercapacitor with a polyvinylchloride (PVC) packaging, high corrosion resistance of the device is claimed [157]. How the PVC used for the packaging should be environmentally friendly remains mysterious. Application-related aspects of corrosion of composite materials applied in structural capacitors have been reviewed [158].

5.2 Remedies

Carbon nanofibers directly grown on carbon cloth manufactured into an EDLC-device resulted in a highly stable device because of the absence of corrosion-prone electrode surfaces and metallic auxiliary components [159]. A neutral 1 M Na₂SO₄ electrolyte solution was found to cause lowest corrosion by comparison with other electrolytes [160]. Lower corrosion activity of neutral electrolytes has been reported elsewhere [66]. Unfortunately, the basically preferable use of neutral electrolytes comes with the drawback of lower ionic conductivity of the electrolyte solution. As a compromise, mixtures like 0.25 M H₂SO₄ + 1 M KNO₃ have been suggested [161]. General aspects of electrolyte solutions, including current trends and corrosion, have been reviewed [162]. Addition of ionic liquids to aqueous electrolyte solution has been proposed as an option to reduce corrosion of metallic current collectors [163]. High concentrations of electrolytes reduced corrosion of aluminium current collectors because the amount of free, i.e. non-coordinated, solvent molecules supporting corrosion was reduced [164]. Deep eutectic solvents cause less corrosion [165]; for further data see [166].

6. Conclusions and Outlook

Corrosion in its various forms of metals, carbon in its numerous forms, metal oxides and other materials employed as active masses or in auxiliary functions in supercapacitors contribute to their ageing resulting first in performance losses and finally in failure. Given the high expectations for a long lifetime this may be particularly surprising. The collected experimental evidence and sometimes tentative interpretation in terms of corrosion causes and mechanisms for the numerous modifications and combinations of materials still need to be expanded with more attention to the specific unwelcome processes addressed above, they allow nevertheless suggestions of remedies on the material, electrode and device level. Taking into account these suggestions, longer and more reliable lifetimes of supercapacitors of both the already well-established EDLC-type as well as the redox type may be feasible.

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Conflict of interests

There is no conflict of interest for this study.

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