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Abstract

Electro-separations (e.g., electro-filtration, electrosorption, electro-deposition, electro-precipitation, electro-coagulation) have received growing interest towards the selective recovery of value-added compounds from wastewater. The coupling of electro-separations with electro-conversion (e.g. advanced electro-oxidation, organic electro-synthesis) has given rise to new materials and systems that uniquely combine reactivity and selectivity. These new reactive separation platforms offer several synergistic advantages beyond each individual component, such as (i) mass transport enhancement, (ii) increased removal rates and yields towards biorecalcitrant pollutants, and the (iii) improvement of recovery and regeneration of porous electrosorptive materials. Significant efforts have recently been devoted to the functionalization of tunable conductive materials for enhanced selectivity (e.g., by leveraging redox-electrochemistry), and in parallel, hybrid systems designs are emerging that enhance efficiency and modularity. Going forward, a major challenge remains to evaluate the efficiency of reactive electroseparation schemes in real effluent contexts, and to test the lifetime and viability of these electrochemical systems at larger scales.

Keywords:

Capacitive deionization; Electrosorption; Membrane-based electrodes; Peroxicoagulation; Redox polymers; Process Intensification

1. Introduction

The world is facing critical water issues with the concomitant rise of water stress, partly due to climate change, and partly due to the increase of water demand from agriculture, industry and households [1]. As such, new strategies for water and wastewater management are urgently needed to implement water reuse, which is recognized as one of the most reliable solutions to the impending crisis. The recovery of value-added inorganic (e.g., metals, phosphorus) and organic (e.g., phenols, short chain carboxylic acids, biomolecules) compounds represents an emerging co-development strategy, that enables the circular economy loop through generating value-added streams from waste.

The implementation of new advanced physico-chemical treatments is required in wastewater plants to achieve the removal of biorecalcitrant pollutants, while extracting the valuable molecules [2]. As a design target, these systems should reduce the overall capital costs, while achieving a high removal rate and yield of recovery even in dilute water streams. Furthermore, the treatment system should also minimize the release of unwanted byproducts. Advanced electrochemical systems are gaining interest through their modularity and possible pathways for process intensification. Electrochemical systems can combine several unit operations in hybrid reactors, especially through the combination of electro-conversion (i.e. involvement of electrochemical reactions) [3–10] with electro-separation (i.e. absence of conversion) technologies, which we will refer to as reactive electro-separation [11]. For these integrated reactive separation systems, a critical component is the control of interfacial selectivity, through the development of new electrode materials to enhance the separation performance [12]. In conjunction, at a systems scale, there is increased attention needed for the selection and optimization of operating conditions, and electrochemical process design. Here, we will highlight recent developments in reactive electroseparations, and discuss future opportunities in this growing area of research.

2. Main reactive electro-separation systems

2.1. Reactive electro-filtration

Reactive methods for breaking electroneutrality in water to drive salt migration have grown in attention as energy-efficient methods for selective separations and desalination. Core to these technologies are filters and membranes that enable charge and species separation and reaction. The major electrochemical technology for desalination is electrodialysis, which relies on water splitting at each electrode and alternating anion- (AEM) and cation-exchange membranes (CEM) between two electrodes to drive ionic transport [13–15]. While this technology has historically been used for desalination, modifications to the membranes and integration of ion exchange resins have led to

emerging applications, including resource recovery [14]. Recently, Kim et al. combined electrodialysis with redox-flow concepts and nanofiltration membranes (NFs) to eliminate the need for AEMs, which carry a high cost and propensity for fouling [16]. A poly(ferrocenylproylmethacrylamide-co-[2-(methacryloyloxy)ethyl]trimethylammonium chloride] (P(FPMAm-co-METAC)) was used as a water-soluble redox-copolymer containing redox-active ferrocene groups, to prevent crossover over nanofiltration membranes, while reducing the energy consumption of a conventional sing-unit cell electrodialysis system by 88% [16]. Conventional electrodialysis relies on the water-splitting reaction to drive ionic transport through AEM and CEM while redox-mediated electrodialysis uses reversible redox reactions to drive deionization (**Fig. 1**). Since the standard reduction potential of the ferricyanide and ferrocyanide redox pair is lower than the water-splitting reaction, their redox-flow system lowered energy consumption by 52% compared to traditional electrodialysis [17]. Membrane tuning by layer-by-layer polyelectrolyte was also found to be able to impart ion selectivity to redox-electrodialysis – with the benefits of improving membrane stability by suppression of radicals or pH change [17]. In particular, these redox-mediated electrodialysis concepts have been expanded to different aspects of food and biomanufacturing, including the ion-selective recovery of carboxylic acids, and the sustainable valorization of whey proteins [17–19].

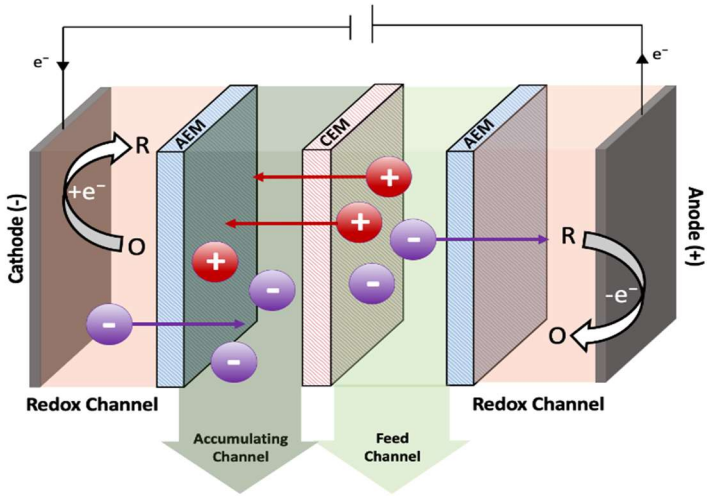

Figure 1. Schematic illustrating redox-mediated flow electrodialysis.

Reactive electrochemical membranes (REMs) can also be considered as a major technology class of reactive electrofiltration, in which the membrane is often the working electrode and promotes an electrocatalytic reaction (**Fig. 2**) [20,21]. For example, REMs can combine an electrochemical advanced oxidation process (EAOP) with physical separation and could mediate some drawbacks of even polymeric electrodialysis, including organic and mineral fouling and high production costs [22]. More recent work by Le et al. applied Magnéli phase Ti_4O_7 REM to the oxidation of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS). After a single pass through the REM, they reported

concentrations of < 86 ng L^{-1} and 35 ng L^{-1} PFOA and PFOS, respectively, reduced from starting concentrations of 4 mg L^{-1} and 5 mg L^{-1} [23]. Work by Guo et al. in 2016 began tests on a novel chemically resistant Magnéli phase REM for water treatment that could eliminate fouling and scaling [22].

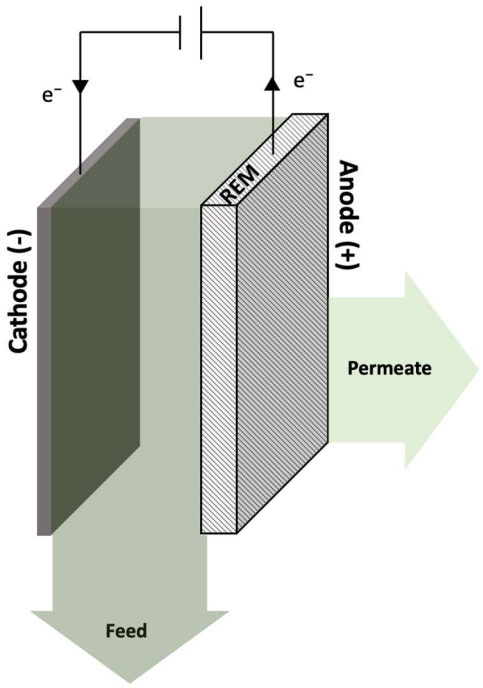

Figure 2. Schematic illustrating a reactive electrochemical membrane (REMs) system.

Overall, reactive electrofiltration methods that combine membranes with surface redox-reactions provide a modular platform beyond desalination. REMs provide a pathway for process intensification through combination of reaction and separations, while redox-flow electrodialysis concepts can provide a platform for both selective membrane separations as well as low energy, low-cost desalination. Going forward, we envision parallel efforts in process design and scaleup to de-risk many of these emerging technologies for practical applications, at the same time as growing studies in the materials design of redox-materials and membranes for more efficient separations and transformations.

Other electrokinetic-based water treatment methods have recently gained growing interest for desalination and selective ion removal include electrodeionization (EDI) and shock electrodialysis or shock ED. These technologies rely on electrokinetic processes, and the transport of charged contaminants in an electrolyte in response to an applied electric field and can remove organic and inorganic ions from water [24]. Among them, shock ED is an emerging electrochemical technique for water treatment in which deionization shock waves are generated in a charged porous material. A

relatively high over-limiting current is passed perpendicular to the direction of fluid flow between ion selective membrane [25]. This creates zones of ion depletion or enrichment within the fluid [26]. In 2015, Schlumpberger et al. published the first example of a continuous and scalable shock ED system [26], with over 99% of salt removal and a water recovery of 79% at high current. In 2019, Conforti and Bazant showed the first use of shock ED to continuously separate ions from multicomponent electrolytes [25]. They showed magnesium ion, Mg^{2+}, could be selectively removed from a mixture of sodium chloride, NaCl, and magnesium chloride, $MgCl_2$. For feed solutions with 9:1 Na:Mg, a peak magnesium removal of 99% is achieved at a total desalination of only 68% [25].

Finally, it must be noted that electrohydrodynamics (EHD) can be used for a range of applications of environmental remediation. EHD can play a key role in process intensification, which has led to a growing number of applications (e.g., water treatment, soil remediation, sensing, monitoring, synthesis, surface finishing of materials) [27–29]. Electrokinetic remediation in particular has seen extensive development over the years for a range of heavy metal decontamination, organic species removal, among others, with several reviews providing an extensive summary of the field [30,31].

2.2. Reactive electrosorption

Electrosorption, the mechanism in capacitive deionization (CDI), is a technique in which species in solution are attracted to a positively polarized electrode and subsequently released by applying a negative polarization [32–34]. While CDI has proven effective in certain contexts of desalination, recent studies have expanded beyond double layer effects to achieve selectivity through redox-reactions [35]. In order to transition from a purely capacitive method to more selective ion-binding, redox-active materials can be utilized [24,36,37]. Redox reactions can create a tunable surface charge on immobilized electrodes, attracting ions towards the respective electrodes – and the redox-site can be tuned for selectivity and also controlled voltage [38]. When the opposite bias is applied, the redox reaction is reversed, and the ions are expelled (**Fig. 3a**). Furthermore, this electrochemical ion binding process can be coupled with electrocatalytic moieties to promote reaction upon adsorption or desorption (**Fig. 3b-c**) [39,40].

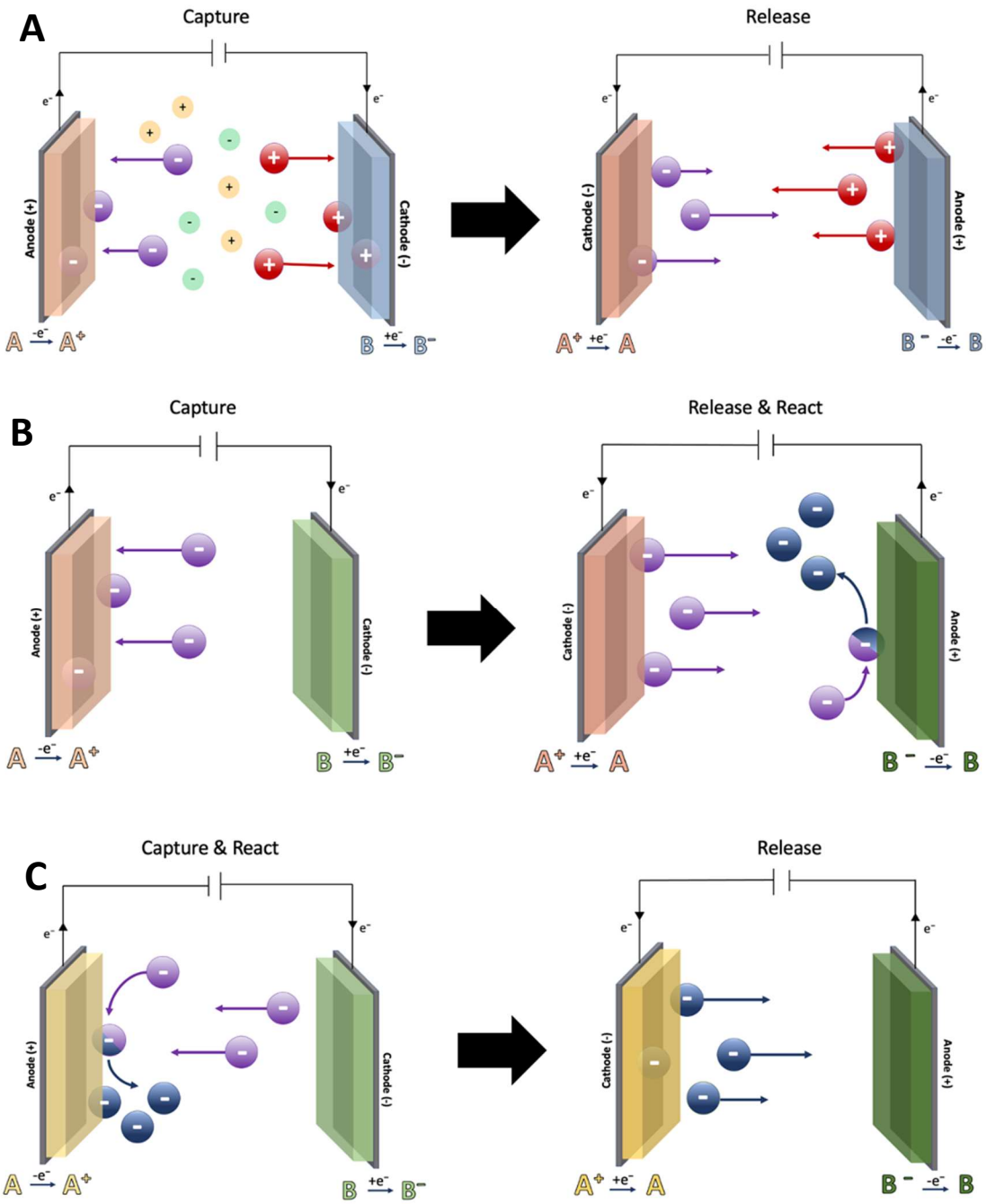

Figure 3. Schematic illustrations showing redox-mediated reactive separation mechanisms: A) Redox-reaction driven asymmetric electrosorption (left) and release (right). B) Asymmetric electrosorption by redox species (left) and reactive conversion upon desorption (right). C) Coupled reaction and reaction by redox-electrodes.

Redox-mediated electrosorption has been applied to a range of wastewater treatment and resource recovery contexts, with some of them highlighted as follow:

- **Arsenic**

In many cases, the removal of anionic As(V) over neutral As(III) species is favored by membrane separation and conventional adsorption techniques. As such, there is a growing focus on the development of electrochemical remediation techniques which transform As(III) into the less harmful As(V) [39,41]. Su et al. utilized redox-active polyvinylferrocene/carbon nanotubes (PVF/CNT) electrodes to selectively capture As(V) at concentrations as low as 100 ppb in the presence of competing excess ions [42]. Song et al. performed a similar study using PVF/CNT functionalized electrodes to adsorb arsenic at zero applied voltage leading to the selectivity of As over competing anions like Cl^-, SO_4^{2-}, and NO_3^-, which have low affinity at 0 V. A total As removal efficiency of 51% of the original 150 µg/L present in the feed solution was performed with a low electrical energy consumption only on the order of 0.12 kWh/m^3 [41].

Next, through an asymmetric electrosorption and electrocatalytic system, Kim et al. achieved over 90% removal efficiencies in wastewater where the concentration of arsenic was as low as 10 ppb [39]. As(III) was selectively captured by a redox-active PVF electrode, and catalytically converted into As(V) during desorption by the TEMPO group of poly-TEMPO-methacrylate (PTMA), another redox-electrode [39]. This combined system showed an order of magnitude decrease in energy consumption compared to the sequential system (in which reaction and separation are decoupled), thus highlighting the importance of integration reaction and separations. Recent work by Shi et al. combined redox-active ferrocene with an iron(Fe) based metal-organic framework (MOF), Fe-MIL-88B-NH_2. From an initial arsenic concentration of 150 ug/L, they showed a reduction to < 10 ug/L with the process requiring only 0.025 kWh/m^3 [43].

- **Nitrate**

The removal of hazardous NO_3^-, which accumulates in land and water [44–46], has been proposed by conventional desalination techniques like electrodialysis, reverse osmosis and ion exchange [32]. More recently, there has been an increase in the investigation of electro-adsporption based techniques which can be coupled with ion exchange membranes, to increase the selectivity of CDI-based technologies towards nitrate [32]. To expand upon conventional membrane capacitive deionization (MCDI), Kim et al. coated active carbon electrodes with AF20E, a nitrate-selective resin, in an ion exchange resin and tested their system using real municipal wastewater. The electrodes containing AF20E proved more selective for NO_3^-, over chloride (Cl^-) and sulfate (SO_4^{2-}), compared to an electrode coated with just an ion exchange polymer [35].

While the above techniques aim to remove the nitrate present in water, reactive electroseparation technologies can also offer a platform to convert nitrate into value-added ammonia [40]. Kim et al. recently combined polyaniline, a nitrate-selective redox-active polymer, with cobalt oxide, an electrocatalyst for nitrate-to-ammonium conversion. Using a wastewater source containing dilute nitrate of 0.27 mM, a wholly electrified system for capture, up-concentration, and conversion was demonstrated within a single electrochemical cell[40]. The impact of the electro-separation step on the efficiency of electro-conversion was highlighted, where concentration of the dilute nitrate into a more concentrated stream significantly enhanced current efficiency towards ammonia production.

- **Per- and polyfluoroalkyl substances (PFAS)**

Electrochemical methods have proven effective at removing highly persistent per- and polyfluoroalkyl substances (PFAS) [46,47] from water, with Kim et al. reporting an electrosorption system that effectively removed perfluorooctanoic acid (PFOA) with a high uptake capacity of > 1000 mg g^{-1} [48]. An asymmetric design of redox electrodes was accomplished by integrating P(TMAx-co-TMPMA1-x)-CNT with a boron-doped diamond (BDD) electrode to selectively adsorb/release and subsequently degrade PFOA. The developed redox-electrosorbent proved highly selective towards PFOA, with a separation factor of 500 PFOA versus Cl^- in the presence of 200-fold excess of chloride [48]. Beyond PFOA, other shorter chains have also been evaluated and treated using redox-electrosorption methods. Manufacturers have switched to short-chain PFAS compounds, like hexafluoropropylene oxide dimer (HFPO-DA, tradename GenX), which poses further challenges due to their higher mobility and recalcitrance to adsorptive treatments like granular activated carbon (GAC) and anion exchange [46,49]. The redox copolymer and BDD system described above was also evaluated for capturing and destroying GenX, with 100% defluorination being achieved after 24 hours of operations [50]. A recent study by Román Santiago et al. tuned redox-active copolymers with differing ratios of fluorinated and amine functional groups to study the effect of these interactions on short chain PFAS binding, including perfluorobutanoic acid (PFBA) and perfluorohexanoic acid (PFHxA) [51]. The key impact of fluorophilicity in promoting higher adsorption of the shorter PFBA was elucidated through both electrosorption tests and molecular dynamic simulations.

Overall, there has been a fast-growing interest in leveraging redox-electron transfer reactions for selective separations, as well as the integration of capture and conversion for different environmental remediation and resource recovery applications [52–55]. We envision that a growing combination of tools will be leveraged to increase predictive capabilities for tailoring electrodes for selective electrosorption, including (i) chemical design and synthesis, (ii) multiscale modeling from an atomistic

model all the way to macroscopic transport and process models, and (iii) operando methods for investigating interfacial binding.

2.3. Reactive electro-deposition

Electrodeposition is a widely and historically used technique to remove heavy metal ions from wastewater. In particular, three-dimensional porous electrodes have been extensively studied for the capture and removal of metals, especially from dilute solutions [56–58]. Electrodeposition relies on a redox process in which a metal ion in solution can be deposited onto the cathode upon reduction and subsequently oxidized upon acid leaching. Electrodeposition can be performed selectively by applying an optimal reduction potential to target deposition of specific ions [13,37,59,60].

More recently, electrodeposition has been applied for battery recycling, for the separation of cobalt and nickel from spent NMC cathodes [61,62]. A major challenge encountered is often the relatively close physicochemical properties of Co(II) and Ni(II), including close electrodeposition potentials under aqueous conditions. Armstrong et al. explored electrodeposition to separate Co and Ni, ultimately producing a 90% Co/10% Ni alloy, while concluding that additional steps, including predeposition and refining, were needed [61]. Yan et al. employed a unique technique combining electrodeposition with electrodialysis to treat spent electroless nickel plating bath, achieving 82% recovery of Ni(II) while simultaneously removing 52% of the HPO_3^{2-} present [63]. Kim et al. sought a more direct approach to control the selectivity of Co/Ni separation by tailoring an electrode with a polyelectrolyte, PDADMA [62]. The selectivity was tuned by polymer loading and electrolyte control. When concentrated chloride was used as a background electrolyte Co exists in the form of a stable anion $CoCl_4^{2-}$ while nickel exists as the cationic complex $[Ni(H_2O)_5Cl]^+$. This speciation led to a difference in onset potentials, creating a window in which nickel can be selectively electrodeposited [62].

Further redox reactions can be coupled to electrodeposition through electrodeposition-redox replacement methods (EDRR), a technique in which a target metallic element is deposited through the redox replacement reaction of a more reactive metal [64]. This method can be applied for noble-metal recovery, including platinum (Pt), silver (Ag), and gold (Au) [65]. Halli et al. applied EDRR to actual industrial hydrometallurgical process solutions, which contain primarily Ni and almost negligible concentrations of platinum (Pt) below one ppb. They achieved selective recovery of Pt by utilizing novel pyrolyzed carbon (PyC) electrodes, ultimately measuring an average of 90 weight percent Pt for the nanoparticles deposited on the electrode surface [64]. As seen, even though the field of electrodeposition has been well-established, the incorporation of new functional electrodes and modes of operation have expanded their applicability to a range of metal recovery and purification contexts. With the emerging supply-chain challenges and growing awareness towards recycling, we

envision the study of molecular selectivity in electrodeposition to play a key role for the efficient recycling of metals from complex, multicomponent feedstocks.

2.4. Reactive electro-precipitation

Electro-precipitation is based on the local alkalization at the vicinity of the cathode material, through the reduction of oxygen (O_2) and/or H_2O into hydroxyl ions (OH^-) (**Fig. 4**) [66,67]. This local OH^- accumulation is known to favor the cathodic precipitation of mainly calcium carbonate ($CaCO_3$) and magnesium hydroxide ($Mg(OH)_2$) in the presence of hard water [68–71]. This phenomenon can be implemented in water softening systems to remove the divalent cations. In practice, the reverse polarity technique is implemented in other electrolytic systems whose objective is to avoid scaling [72]. It consists of reversing the roles of cathode and anode at a given frequency, leading to the redissolution of precipitates in the bulk. This method could not be effective enough with effluents having high water hardness and/or with systems implementing cathode materials that do not stand for high anodic potentials. Therefore, efforts have been made to propose alternatives to minimize the scaling. The hybrid combination with advanced electro-oxidation has been investigated recently.

The generation of powerful oxidant such as hydroxyl radical ($^{\bullet}OH$) at a high O_2 evolution overvoltage anode (e.g., BDD), has shown to decrease the cathodic scaling (~5-10%) issue at higher current density (16 mA cm^{-2}) in a thin film reactor (500 µm interelectrode distance) [73]. This approach was concomitant with the local anodic acidification that converted (bi)-carbonates (HCO_3^-/CO_3^{2-}) into CO_2, which reduced carbonates content in the bulk. In the meantime, the use of porous carbon-based cathode material (e.g., carbon paper) was shown to increase the electro-precipitation phenomena [74]. Thus, the microfluidic conditions along with the types of cathode and anode materials are critical parameters to consider for increasing efficiency of the process [75,76]. These factors could be tuned to favor phosphate recovery, which is listed as critical raw material [77], especially because the interest of phosphate electro-precipitation was previously shown in simpler system (i.e., without involvement of microfluidic) [78]. However, the efficiency of the process to achieve high purity of phosphate needs to be improved in real matrices. It has been noted that organic matter can adsorb on amorphous calcium phosphate during physical cathodic co-precipitation, which slows down the electro-precipitation mechanism and could reduce the purity of crystallized calcium phosphate recovered [79].

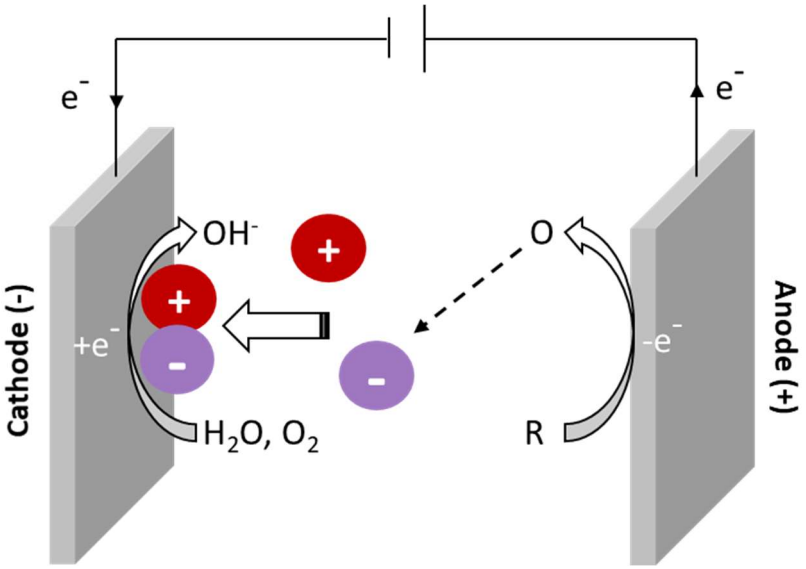

Figure 4. Schematic illustrations of cathodic electro-precipitation with possible interaction (dashed arrow) with oxidant generated at anode.

2.5. Reactive electro-coagulation

Electro-coagulation is known for a long time by promoting the *in situ* generation of coagulant from anodic dissolution of the materials (e.g. iron, aluminium) [80,81]. It provokes the subsequent coagulation with particulate matter, colloidal matter and part of dissolved pollution, that are removed by decantation of the solid phase (i.e., flocs). Additional mechanisms such as precipitation and adsorption on flocs can contribute to the removal of contaminant species. This technology is known to remove inorganic (e.g., phosphate, nitrate, heavy metals) and organic compounds (e.g., natural organic matter) [82]. Electro-coagulation has more recently shown to be particularly useful to remove micro-plastics from wastewater [83].

In combination with an electro-conversion system, namely the peroxi-coagulation [84], the dissolved organic pollution can be also removed with high yield (> 90%) [85]. When iron is polarized as anode, it can react after dissolution with the hydrogen peroxide (H_2O_2) electrogenerated at the carbon-based cathode and produce $^{\bullet}OH$ through Fenton reaction [86,87]. This leads to the concomitant degradation and mineralization of dissolved organic pollution (e.g., 1,4-dioxane [88], 2,4-dichlorophenoxiacetic acid [89], 7-hydroxycoumarin [90]).This process combination could be particularly useful for micro- and nano-plastics removal, while eliminating the persistent dissolved pollutants and recovering value-added compounds (e.g., metals). However, since the value-added compounds are trapped into the hydroxide sludges, there is the need for an additional selective post-treatment to recover the products from the sludge.

3. Conclusions and perspectives

Reactive electro-separations technologies are gaining increased attention for their role in achieving environmental remediation, water treatment and purification, and a circular economy for valuable resources. These advanced systems could benefit from the electrochemical control through the polarization of emerging conductive materials to increase the selectivity of recovery, by tailoring electrode materials characteristics (porosity and pore size distribution, electroactive surface, conductivity, surface functions, elemental composition, among others) and optimizing operating conditions (including current density/electrode potential, inter-electrode distances and more generally the reactor design, ionic membranes, among others). The physico-chemical properties of the target compounds (such as molecular size, electrophoretic mobility, molecular structure, point of zero charge, acid dissociation constant) along with the matrix composition (type and concentration of salts, etc) are also crucial parameters impacting the efficiency. There are several areas for continued study to improve existing reactive electroseparation systems, as well as opportunities to develop new technologies, including:

- Investigate the matrix effects (ions, organic matter, etc) on the reactive separation systems, particularly with real wastewater, and develop solutions to overcome potentially impacting side-effects such as fouling, electrode degradation, or competing ions that may impact selectivity.
- Develop sustainable and energy-efficient methods to recover the value-added compounds deposited on the electrodes after electro-separation step. A major challenge is the development of low energy/low waste technique for recovering the valuable electrodeposited species.
- Evaluate the long-term stability of newly proposed electrode materials, and associated cost-effectiveness, including environmental cost. The development of accurate process models and technoeconomic assessments can be a critical step in de-risking many technologies for application in the wastewater contexts [91,92].
- Make the full system sustainable, by recovering all valuable compounds, as well as co-electrogenerating green energy source (e.g., H_2, bioenergy [93–95]).
- Develop innovative, scalable, and flexible design to ensure synergies in reactive electro-separations systems (e.g., moving electrochemical cells that could combine advantages of micro-reactors with macro-reactors [96,97]). Innovative design should permit antagonist requirements (e.g., low/high overvoltage, low/high electrode gap, low/high current) for hybrid electro-processes combination, such as the proper integration of electrosorption (which is

usually operated lower current density) with advanced electro-oxidation (operated at high current density). Moreover, new designs should seek to intensify mass transport and promote homogeneous current and potential distributions, while avoiding the external addition of salts to increase the ionic conductivity.

- On the long-term, translating existing technologies from proof-of-concept and bench-scale demonstrations to pilot scale, ideally through industrial/academic partnerships that can provide a pathway for product development and real-market end use.

Acknowledgments

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