American Journal of
Biomedical Science & Research

www.biomedgrid.com
ISSN: 2642-1747

Mini Review

Mediated and Non-Mediated Parallel Paired Electrolysis

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To Cite This article: *CAC Sequeira*, Mediated and Non-Mediated Parallel Paired Electrolysis. Am J Biomed Sci & Res. 2026 31(6) AJBSR. MS.ID.004100,* **DOI:** 10.34297/AJBSR.2026.31.004100

Received: July 27, 2026 **Published:** August 11, 2026

Abstract

Paired electrolysis is highly valuable from the viewpoint of efficiency as well as atom and energy economies. In order to optimise the latter two for chemical reactions, the development of paired electrochemical processes is necessary. When both of the electrodes in an electrochemical cell (divided and undivided) are applied as working electrodes, and both sides of the processes (oxidation and reduction) yield valuable compounds, this ideal electrolysis phenomena are defined as paired electrosynthesis, that can be classified as convergent, parallel, divergent, and linear paired electrolysis. This paired electrolysis offers the opportunity to reduce the spent energy and time, when compared with a single electrolysis system that is only used to achieve a product of interest, while ignoring the other side of the electrolysis (anodic or cathodic). In an ideal case, 200% current efficiency could be achieved during paired electrosynthesis using cathodic and anodic processes to provide the same product. Paired electrosynthesis is a highly efficient green process and therefore, is beneficial for preserving resources and minimising waste. However, while a paired electrosynthesis is beneficial, both oxidation and reduction processes must be compatible to counter the yield losses, and equally ease separation and purification of both sides of the electrode products. Greater efforts are required to perform paired electrosynthesis with a more systematic and rational approach to achieve optimal products under paired conditions. Nevertheless, new computational tools could be applied for assistance in this matter. There is a considerable level of adventure in designing new paired electro synthetic processes and accompanying opportunities to design innovative and powerful synthetic strategies. Herein, an overview of several examples of parallel paired electrosynthesis and their advantages are summarised that will aid researchers to both develop a greater understanding of this subject and subsequently employ this type of paired electrolysis for green and sustainable synthesis of organic molecules.

Keywords: Parallel paired electro-organic synthesis, Green chemical engineering processes, Atom economy, Energy economy, Organic electrochemistry, Energy conversion

Introduction

Developments in the methodology of organic electrochemistry are still continuing to emerge [1] due to electrochemistry presenting itself as a green, modern, and safe technique to achieve a diverse range of chemical transformations [2,3] The primary reactant to perform such chemistry is the humble electron that can be used as an inherently clean alternative for an oxidant or chemical reducing agent. Electrosynthesis is an excellent way of minimising energy consumption and can also be used successfully for the *in-situ* production of hazardous and unstable reagents, in addition to maximising energy efficiency. Different electro synthetic methods have been used, such as direct oxidation [4] or reduction of substrates [5] indirect electrolysis (mediated or catalysed)[6-11] or by electrogenerated bases such as CH_3CN [12-17] DMSO, or DMF [18] The process of paired electrosynthesis has been known

for a long time as a convenient and practical methodology [19] the appellation of which has been to describe electrochemical processes in which both the reaction at the anode and the reaction at the cathode contribute usefully to the formation of the product(s). The literature, however, predominantly reports reaction at only one electrode [20-21] either anodic oxidation or cathodic reduction (Figure 1). Hence, the reaction products from one of these is usually wasted. In contrast, paired electrolysis could maximise energy efficiency by using both cathodic and anodic reactions without waste and undesirable products. As shown in (Figure 2), the total energy required to drive any electrolysis reaction is defined by the potential difference needed for accomplishing both the oxidation and the reduction half reactions [22].

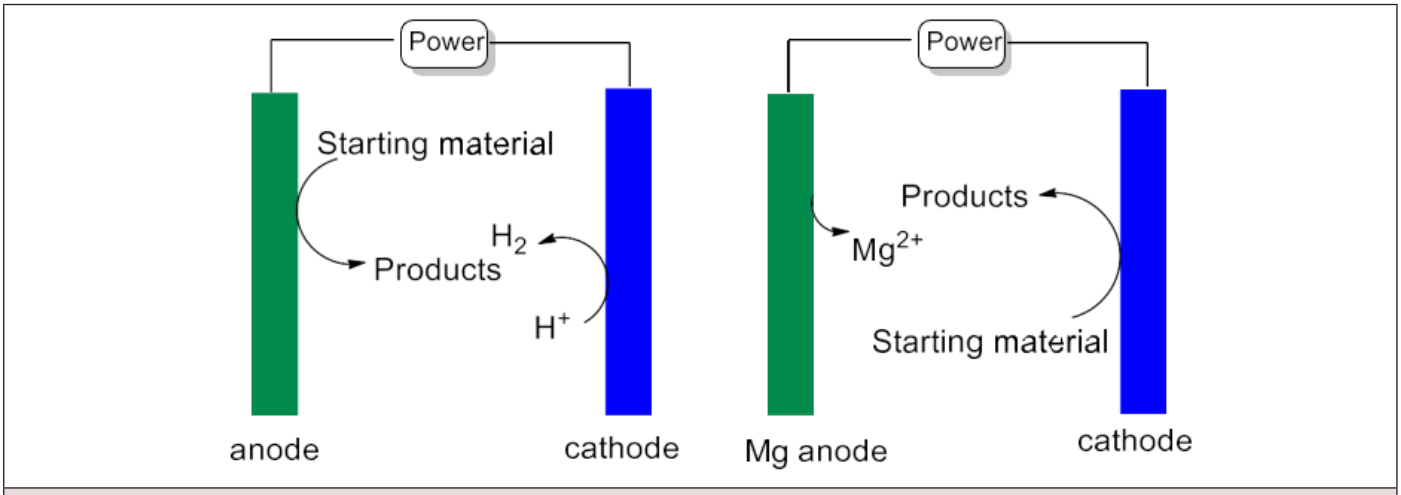


Figure 1: The formation of a desired product at only one electrode (by oxidation or by reduction).

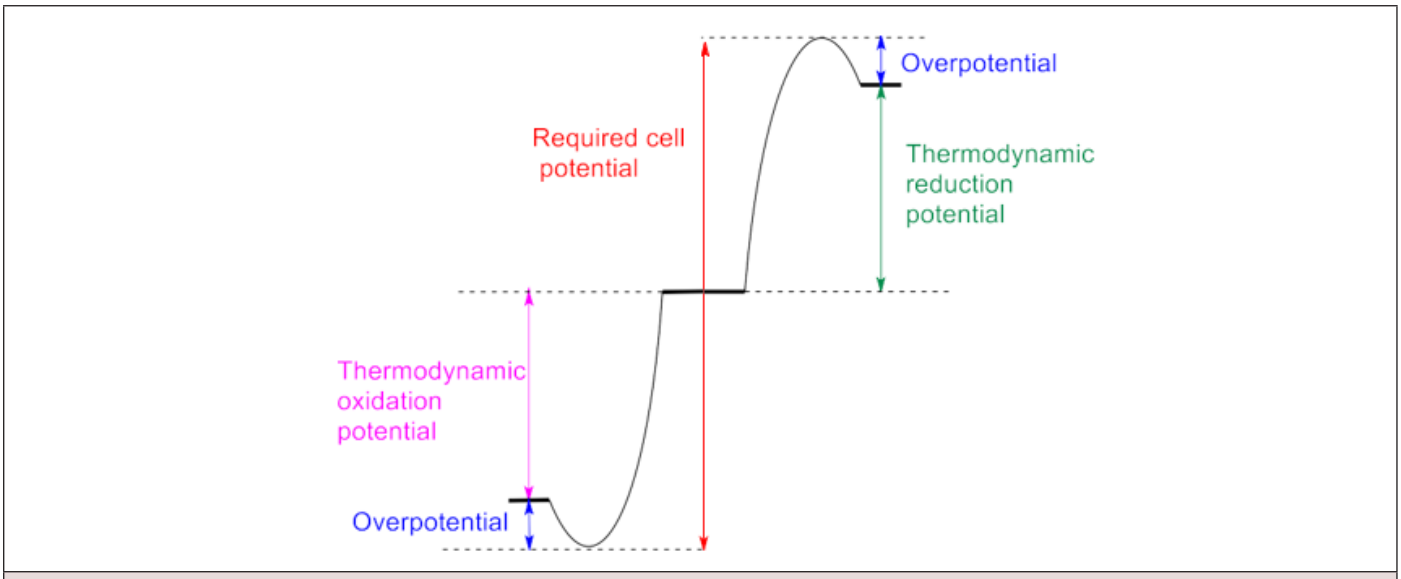


Figure 2: Energy requirements for electrolysis. Reproduced from ref 22, Copyright (2021) John Wiley & Sons.

One of the vital bottlenecks in paired reactions is the electrode material/processes' selectivity [23-25] which has to be chosen to allow the formation of a product without further undesired side reactions. Typically, paired reactions are classified into four types: convergent, parallel, divergent, and linear paired electrolysis (Figure 3). *For the convergent paired electrolysis*, the intermediates formed on the anode and cathode react together to provide the desired product (Figure 3a). *In parallel paired electrolysis*, the anode and cathode reactions are simultaneous, but non-interfering or reacting; two different products can be obtained from two different starting substrates (Figure 3b). *In divergent paired electrolysis*, one starting material is reduced as well as oxidised, providing two different products (Figure 3c). In *linear paired electrolysis*, one product will be obtained by the substrate's oxidation, followed by reduction, or vice versa (Figure 3d).

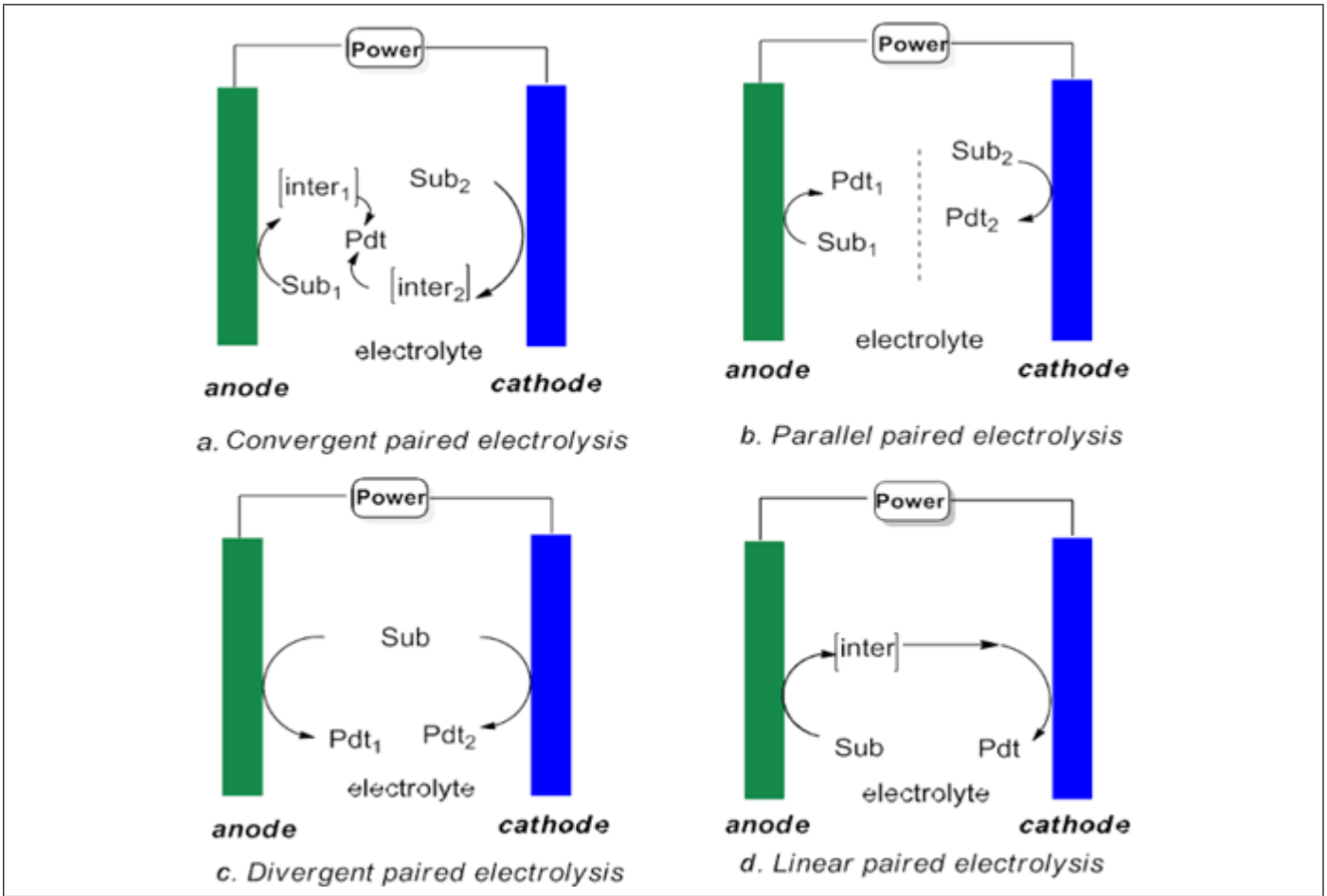


Figure 3: Types of paired electrolysis: a. Convergent paired electrolysis, b. Parallel paired electrolysis, c. Divergent paired electrolysis, d. Linear paired electrolysis; intermediate= [inter]; substrate= Sub; product= Pdt.

In electrochemical reactions, more consideration is given to optimize the efficiency of atom usage without paying similar attention to optimizing the use of energy. Equally, the part of the energy associated with the counter reaction is simply overlooked. However, to optimize both the atom and energy economy of a reaction, the development of paired electrolysis is important to maximize the outcome (oxidation and reduction products), thereby maximizing the energy efficiency of the reaction.

To make paired electrochemical process a sustainable, the number of things could be considered such as i) sustained electrolysis ii) maximum faradaic yield iii) optimized power conversion efficiency iv) current matching of the half-reactions in an electrolysis cell v) chemical compatibility between half-reactions vi) formation of products with high value than the chemical reactants, vii) renewably sourced chemical feedstocks viii) design of modified and selective electrodes to avoid unwanted reactions [26-27] These adaptations in electrolysis could improve the atom economy at the possible expense of energy economy. This increased sustainable phenomenon of paired electrolysis can be implemented within larger synthetic setup where the reagent produced from paired electrolysis further can be used for another possibly non-electrochemical reaction and so on more chemical transformations are achievable. By pairing electrode processes at a closely spaced anode and cathode such as in micro-flow reactors where the diffusion layer of anode and cathode overlap to give the desired product and paired electrochemical processes can now be managed without the need for a deliberately added supporting electrolyte. The product is obtained directly by solvent removal and without laborious separation phases, which is of considerable benefit in chemical syntheses [28].

Generally, paired electrolysis is economical for a number of reasons.

i) The energy consumption considerably reduces. The reaction takes place simultaneously at the

working-and counter-electrode and this fact offers the prospect to reduce energy and time by pairing the two processes so that the chemicals formed at each electrode are valuable. Thus, the yields may increase past 100%, thereby maximising the energy efficiency. Moreover, in case of microflow when inter-electrodes distance is very short, paired electrolysis can now be accompanied without supporting electrolyte and the diaphragm at low overpotential.

ii) Theoretically, in an ideal case to provide the product, current efficiencies can be achieved with

a value of up to 200%, since the current efficiency is equal to the mole of products per unit of charge used in the electrochemical cell (when a mole of electrons is passed through the electrochemical cell

and both electrodes accomplish valuable chemical transformations, the overall yield is simply the total of the respective yields of the reactions that occur at each electrode). In some cases, hydrogen is evolved at a cathode in protic solvents like water or methanol, along with desired anodic products. The evolving hydrogen can be economical and utilized as fuel material.

iii) The process costs and resources that can be saved once both sides of the electrochemical cell

are used instead of a separate arrangement or an additional cell/ reactor with extra electrodes, electrolytes and vessel space.

iv) Maximum atom economies are also possible because the formation of wastes/side products

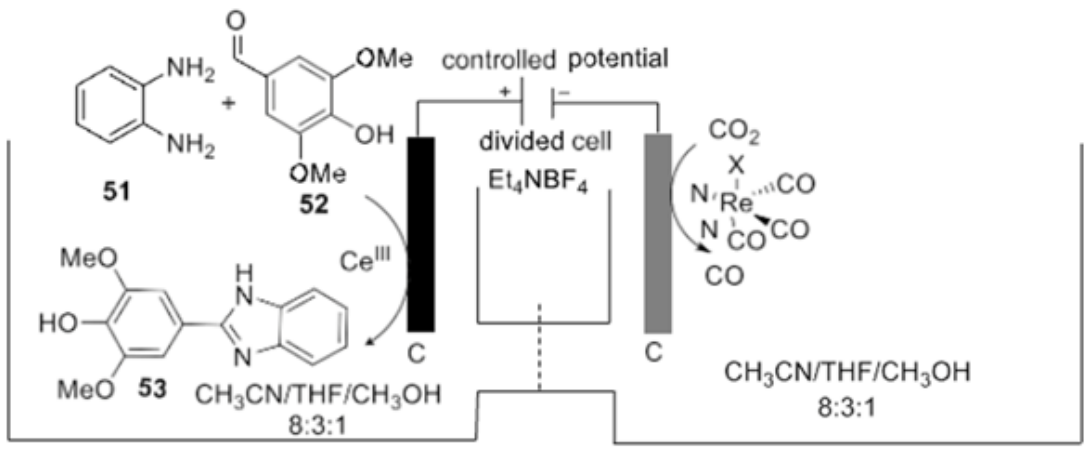
are minimised. Furthermore, fewer separation and synthetic steps are required. However, reaction control is critical in paired electrolysis, and therefore, the cell and the setup have to fit for each target reaction. Work-up, product isolation and purification are important for a successful synthesis and can be even more problematic in a paired synthesis. The paired reaction has been successfully utilised in industrial production, such as for the paired electrosynthesis of nylon [6,29] the parallel paired reaction for the formation of 1-butyl-4-(dimethoxymethyl) benzene and isobenzofuran-1(3*H*)-one [30] the electroformation of gluconic acid and sorbitol via divergent electrolysis of glucose [31] the formation of glyoxylic acid via convergent paired electrolysis [32] the electrocatalytic convergent paired degradation of *Para*-dinitrobenzene [33,34] the electrosynthesis of methyl ethyl ketone via linear paired electrolysis,34 and the use of electrogenerated azobenzeneasabasetoachieveethylhexanoate[35]Recently,wehave published review articles emphasising the importance of organic electrochemistry for the synthesis of heterocyclic compounds [36-37] annulation reactions [38] alkyne functionalisation [39] alkene difunctionalisations [40] and the electrochemical phosphorylation of organic compounds [41] In 1999, Chou and co-workers reviewed the types of paired electrolysis [42] in 2017 the Pletcher group [43] reviewed the advantage of flow electrolysis cells for paired synthesis, the basic strategies of paired reactions were reviewed by Hilt in 2019 [44] and in 2020, the Nanda group reviewed the key parameters of paired electrolysis [45] However, looking at these reviews, we found that they do not contain the new examples of paired electrolysis reactions. It is for this reason that we have been reviewing many recent developments of paired reactions. Herein we review an updated summary of recent developments in parallel paired electrolysis.

Parallel Paired Electrolysis for Organic Transformations

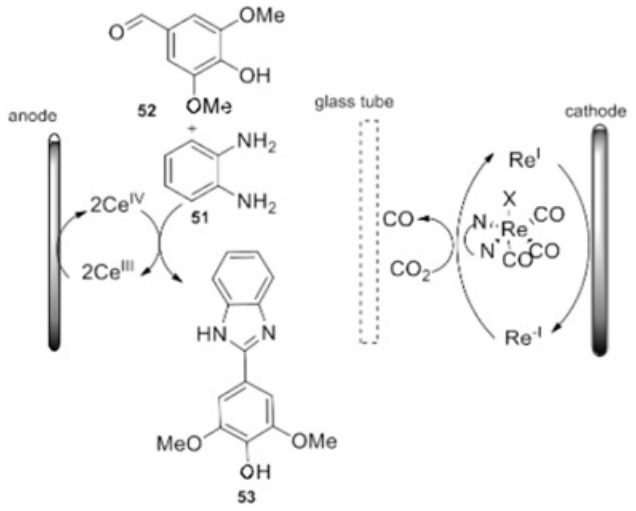
Parallel paired synthesis has been reported in two types; mediated and non-mediated synthesis. Both were successfully used to manage different reactions, such as reported below.

Mediated Electrolysis: Nowadays, parallel paired electrochemical synthesis has proved useful

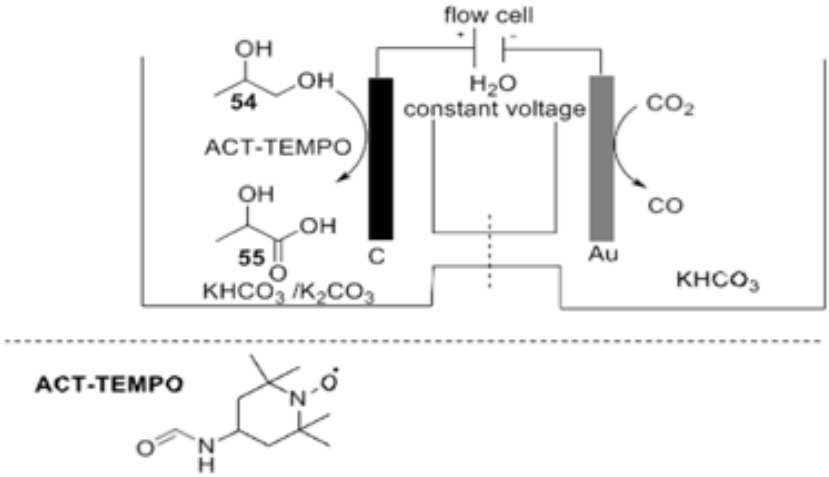
for industrial application, especially for interesting chemical reactions that convert CO_2 gas into a valuable chemicals and fuels, e.g. CO reagent [46] in 2016, Moeller and co-workers [26] reported a parallel paired electroreduction of CO_2 to CO catalysed by (Re(4,4'-di-tert-butyl-2,2'-bipyridine) $(CO)_3Cl$. The paired synthesis is combined with the electrosynthesis of 2-(3,5-dimethoxy-4-hydroxyphenyl)-benzimidazole 53 from the oxidation of syringaldehyde 52 and o-phenylenediamine 51 by employing Ceric ammonium nitrate as a catalyst (Scheme 1). The reaction proceeded in a divided cell equipped with a glassy carbon working electrode for both the cathode and anode in a mixture of CH_3CN: THF: CH_3OH/Et_4NBF_4 as a solvent/electrolyte, under a controlled potential. For this type of paired synthesis, focus should be put onto compatibilities and complementarities of the two half reactions, which should be selected carefully, taking care with the amount of voltage used. As an improvement, the authors suggested a plausible mechanism for this transformation (Scheme 2). The reaction at the anode started with the oxidation of Ce^{III} to Ce^{IV}. Then, the coordination of syringaldehyde 52 and o-phenylenediamine 51 with Ce^{IV} led to the desired product 53.



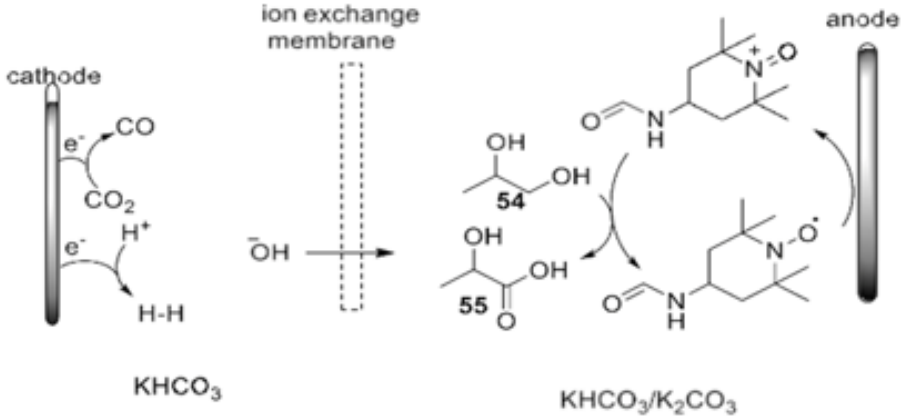
Scheme 1. Parallel paired electrolysis for the formation of 2-(3,5-dimethoxy-4-hydroxyphenyl)-benzimidazole and CO.



Scheme 2. Plausible mechanism for the parallel paired electrolysis for the formation of 2-(3,5-dimethoxy-4-hydroxyphenyl)-benzimidazole and CO.



Scheme 3. A parallel paired electrosynthesis of Lactic acid and CO.

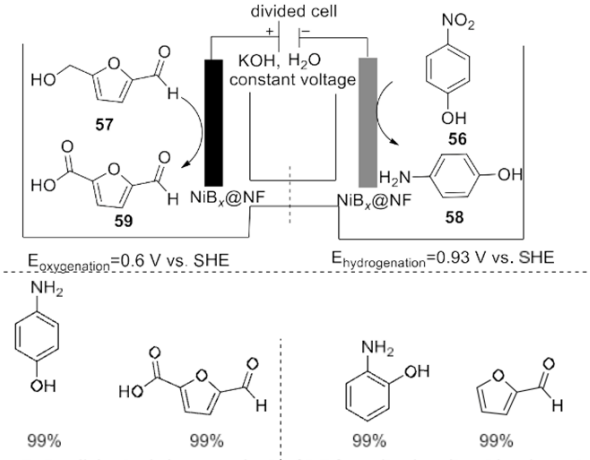


Scheme 4. Plausible mechanism for the parallel paired electrosynthesis of Lactic acid and CO.

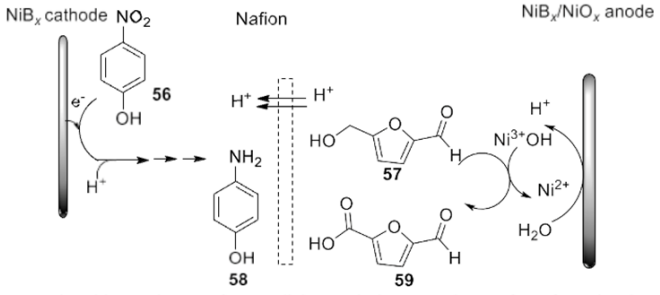
Meanwhile at the cathode, the reaction started by the reduction of Re^{I} to Re^{-I}, which catalysed the transformation of CO_2 to CO. Independently of Moeller's group, Goetheer and co-workers developed an excellent approach for a parallel paired electrosynthesis of Lactic acid 55 from the oxidation of 1,2-propanediol 54 using 4-acetamido-(2,2,6,6-tetramethylpiperidin-1-yl)oxidanyl (ACT-TEMPO) as a catalyst in parallel with the reduction of CO_2 to CO [47]. The reaction proceeded in a flow cell with two compartments, divided by an anionic exchange membrane, equipped with a gold plate as a cathode and carbon felt as the anode in a solution of $KHCO_3$ for the cathode compartment and a solution of $KHCO_3/K_2CO_3$ for the anode compartment, under a constant voltage (Scheme 3). The authors demonstrated that when the anodic half-reaction of oxygen evolution was replaced by alcohol oxidation, a lower energy consumption of ~35% and an increase in the total product value of the process would occur. Moreover, this resulted in a faradaic efficiency, for the combined cathodic and anodic processes, of up to 160% compared to the reduction of CO_2 in which oxygen is formed at the anode. As an improvement, the authors suggested a plausible mechanism for this transformation (Scheme 4). The cathodic reduction of CO_2 on the Au cathode provided CO. Concurrently, 4-acetamido-(2,2,6,6-tetramethylpiperidin-1-yl) oxidanyl (ACT-TEMPO) underwent anodic oxidation to form an oxoammonium ion ACT-TEMPO$^+$ whose further interaction with the substrate 54 provided the desired product 55.

In 2019 Sun and co-workers reported an excellent green method for parallel paired reactions to form 2,5-furandicarboxylic acid derivatives 59 and *p*-aminophenol derivatives 58 [48]. The reaction was conducted in a photoelectrocatalytic cell equipped with NiB_x@NF as a both electrodes, at a controlled potential, in water as the solvent (Scheme 5). Under mild conditions, using water as an oxygen and hydrogen source, a family of hydroxy-methylfural derivatives 57 and nitrophenol derivatives 56 were tolerated, affording the desired products 58 and 59 in excellent yields of up to 99%. The reaction combines two desirable half-reactions for the oxygenation of 5-hydroxy-methylfural 57 to 2,5-furandicarboxylic acid 59 in parallel with the hydrogenation of *p*-nitrophenol 56 to *p*-aminophenol 58 using NiB_x as a catalyst.

In this case, the theoretical maximum electron efficiency is 200%, twice that for conventional unpaired cells. Moreover, this paired synthetic process may lower operation costs by reducing the number of reactors or processing steps required. The newly designed electrochemical cell exhibited high conversions, selectivities, and Faradaic efficiencies at both the anode and cathode, simultaneously, without the involvement of oxygen and hydrogen. High conversions and selectivities were also observed when using furan-2-carbaldehyde, furan-2-ylmethanol, pyridin-4-ylmethanol, and 4- (hydroxymethyl) phenol, as the compounds undergoing oxygenation, and when *p*-NP was replaced with *m*-NP, *o*-NP, 4-nitrobenzonitrile, and 1-ethynyl-4-nitrobenzene. A mechanistic elucidation in (Scheme 6) showed that firstly, Ni^{2+} was electrooxidised with water to form the complex $Ni^{3+}OH$, accompanied by the liberation of a proton. Then the resulting Ni^{3+} species reacted with 5-hydroxy-methylfural 57 to provide 2,5-furandicarboxylic acid 59 and regenerate Ni^{2+}. Furthermore, the proton penetrated through to the cathode compartment to ensure the hydrogenation of p-nitrophenol 56 to *p*-aminophenol 58.

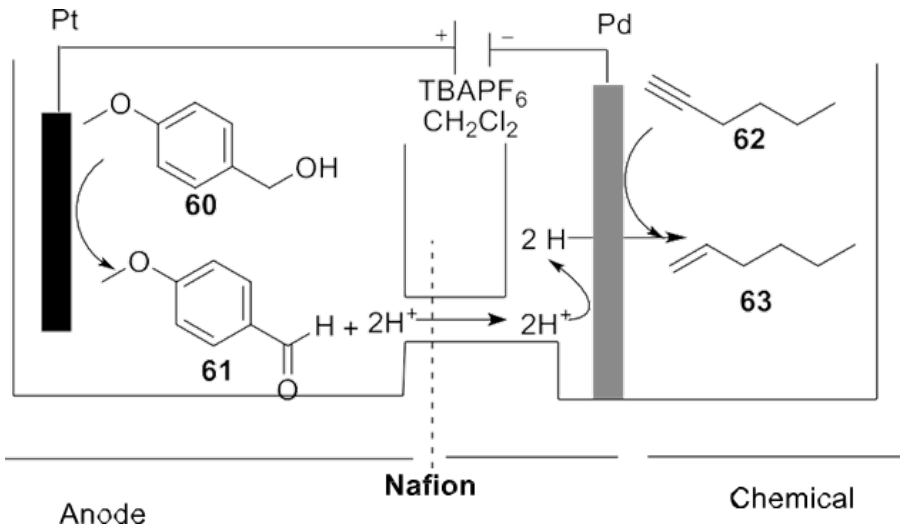


Scheme 5. Parallel paired electrosynthesis of 2,5-furandicarboxylic acid and p-aminophenol by using NiBx@ NF as both electrodes.

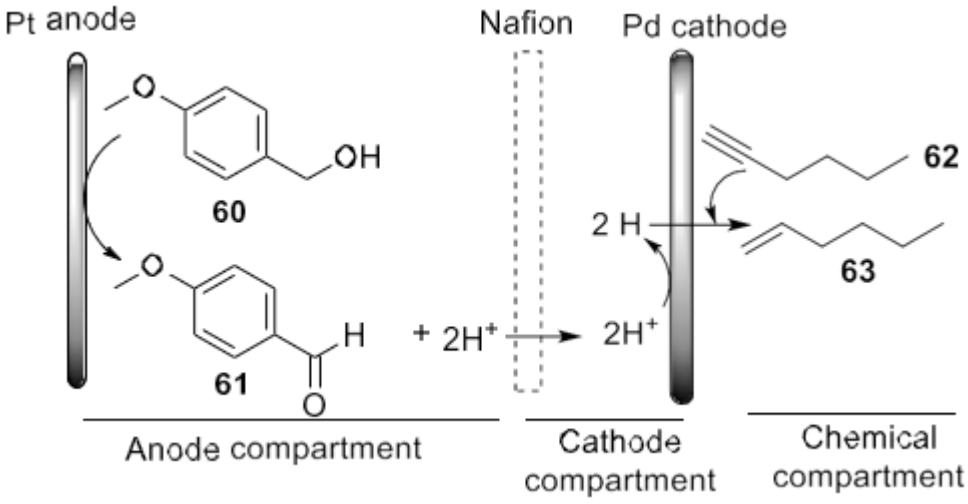


Scheme 6. Plausible mechanism for parallel paired electrosynthesis of 2,5-furandicarboxylic acid and *p*-aminophenol.

Non-Mediated Electrolysis: In 2018, Berlinguette and co-workers reported a good strategy, described by a paired parallel reaction, for the formation of 4-methoxy benzaldehyde 61 from the anodic oxidation of 4-methoxyalcohols 60 and the electro-formation of 1-hexene 63 from 1-hexyne 62 via an electrolytic palladium membrane reactor (Scheme 7) [49,50]. The reaction proceeds in a three-compartment cell, using Pt plates as the anode and Pd as the cathode in a system of CH_2Cl_2/ Tetrabutylammonium Hexafluorophosphate ($TBAPF_6$) as the solvent/electrolyte. The anode and cathode sides of the electrochemical compartment were separated by a Nafion membrane. The fundamental success of this device is the selectivity of the palladium foil which acts as a cathode for proton reduction and a hydrogen diffusion membrane. The authors reported that the adsorbed hydrogen must transition into the bulk of the palladium lattice, diffuse through the lattice and transition to the surface on the other side of the cell to react with the organic substrate. If the kinetics of hydrogen gas evolution on the electrochemical compartment of the palladium are faster than the competing absorption and diffusion steps, hydrogen would evolve only on the electrochemical side of the foil and would not hydrogenate the substrate on the chemical side. It was found that the majority of the total evolved hydrogen was detected on the chemical compartment of the foil at every applied current. Even at an applied current of 50 mA it was found that 90% of the hydrogen was detected on the chemical side of the foil, indicating that almost all the protons being reduced at the cathode are diffusing and recombining on the opposite side of the foil. These results confirm that palladium is acting effectively as a selectively permeable membrane for hydrogen atoms. A mechanistic elucidation in (Scheme 8) shows that firstly, alcohol 60 was oxidised at the anode to provide the corresponding aldehyde 61 with the liberation of a proton. Then, the protons cross a Nafion proton exchange membrane. These protons were reduced at the palladium foil cathode to form the adsorbed hydrogen atom, which easily reacted with alkyne 62 yielding the corresponding alkene 63.

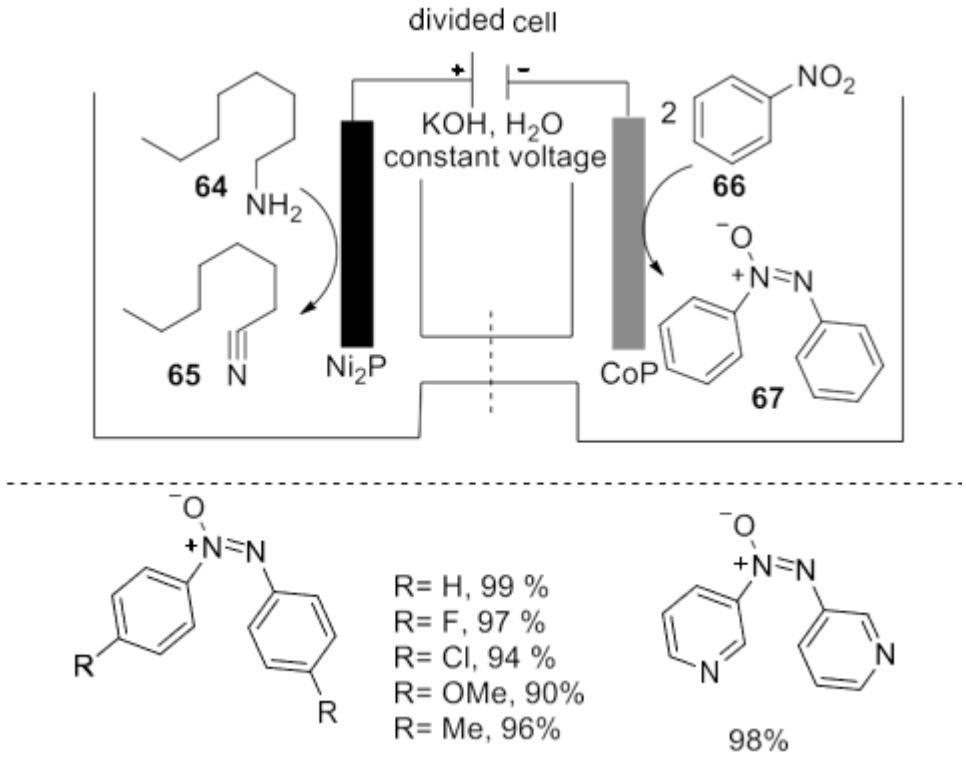


Scheme 7: Paired parallel reactions for the formation of 4-methoxy benzaldehyde1-hexene.

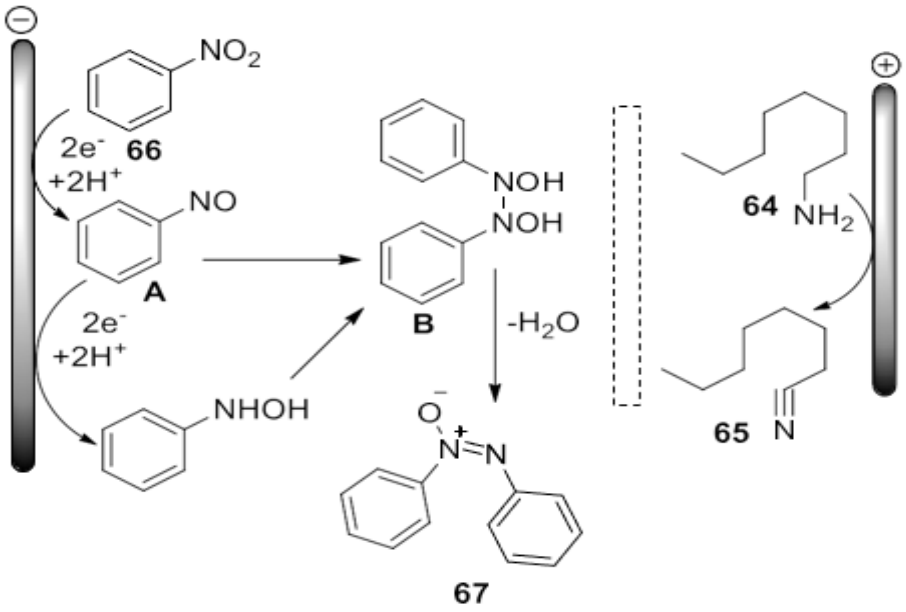


Scheme 8: Plausible mechanism for paired parallel reactions for the formation of 4-methoxy benzaldehyde and 1-hexene.

Very recently, Zhang and co-workers reported an excellent paired parallel reaction for the electroformation of azoxy-aromatics derivatives 67 from the cathodic reduction of nitrobenzene 66 and the electro-formation of nitriles 65 by the anodic oxidation of amine derivatives 64 (Scheme 9) [51] Different nitrobenzene derivatives 66 provide a broad scope of desired compounds in good to excellent yields of up to 99%. Substrates with electron-rich substituents such as Me, MeO, and electron-deficient groups such as F, Cl and Br have a good tolerance in this transformation. The reaction was performed in divided cell equipped with a NiP electrode at the anode and CoP at the cathode, in water (solvent) and KOH as an electrolyte, under a constant voltage for 6 h. The authors found that the required potential is only 1.25 V to achieve a current density of 20 mA cm^{-2}, which is lower than the potential 1.70 V of overall water splitting. Furthermore, these paired reactions can also be driven by 1.5 V batteries to synthesise nitrile and azoxybenzene with high selectivity. Additionally, the amount of KOH should be essentially 1.0 M because it will prevent unwanted hydrogenation of the two intermediates due to the trace number of protons at low potentials. A higher reductive potential result in more active *H which will be generated at the surface of CoP via water splitting, trapping phenylhydroxylamine to form aniline and inhibiting the condensation of nitroso benzene and phenylhydroxylamine. A mechanistic elucidation in (Scheme 10) shows that the cathodic reduction of nitrobenzene gave nitroso benzene A. After further reduction, the latter could provide intermediate B (or B can be formed directly from A). Finally, B led to the final product after losing one molecule of H_2O. Concurrently at the anode, amine 64 was directly oxidised to nitrile derivatives 65.

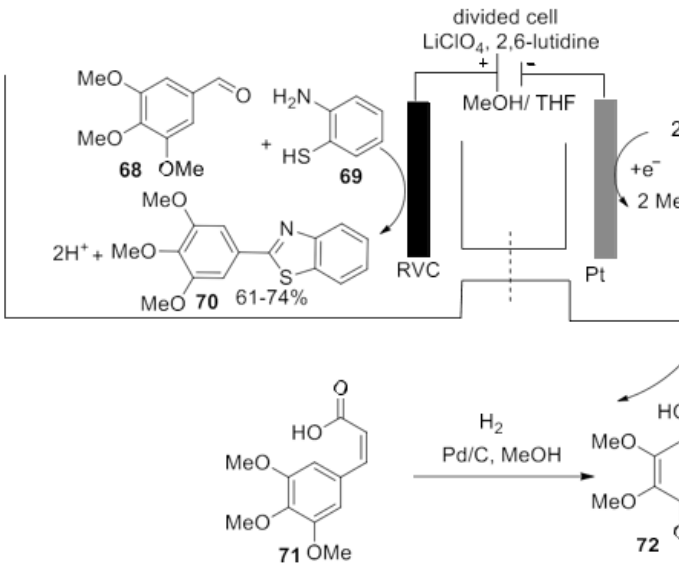


Scheme 9: Electro formation of azoxy-aromatics and nitriles derivatives.

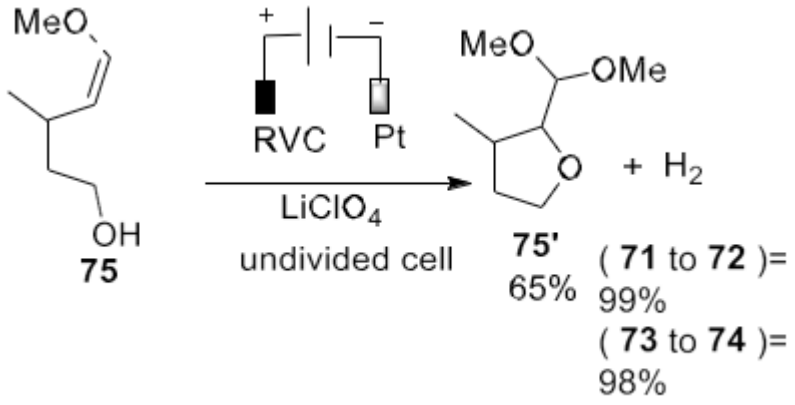


Scheme 10: Plausible mechanism for the electro formation of azoxy-aromatics and nitriles derivatives.

In 2019, Moeller and co-workers reported an important paired parallel reaction for the electro formation of 2-(3,4,5-trimethoxyphenyl) benzothiazole derivatives 70 from the anodic oxidation of 3,4,5-trimethoxybenzaldehyde derivatives 68 and 2-aminobenzenethiol 69 [27] The paired synthesis is combined with a hydrogenation reaction of trimethoxy phenyl-acrylic acid derivatives 71 (Scheme 11). The 4-((benzyloxy) methyl)-1,2-dimethoxybenzene derivative 73 were also tested for this transformation, and the hydrogenated product 74 was formed with a good yield of 90%. Here, the authors proved the environmental and financial cost benefit of producing H_2 gas during the oxidation reactions; the hydrogen gas can be synthesised easily by the cathodic reduction of MeOH. The key advantage of this technology is to transfer the gas, which was formed in the cathodic compartment, to a flask containing the unsaturated acid in the presence of Pd catalyst by the insertion of a cannula into the headspace of the cell. In a divided cell, equipped with a Pt plate as a cathode and an RVC anode in the system MeOH: THF (1:1)/ $LiClO_4$ as solvent/electrolyte. It was found that the use of different substrates for the oxidation reaction did not have any influence on the reduction reaction. This advantage allowed the authors to test the electrolysis in an undivided cell, for the main objective was a cyclisation reaction by anodic oxidation of 75 to form the cyclic product 75' (Scheme 12)



Scheme 11: A paired oxidative condensation with hydrogen generation in a divided cell.



Scheme12: Paired anodic cyclisation in an undivided cell.

Conclusion & Perspective

This review highlighted the recent advances of paired electrolysis, specifically concerning parallel paired reactions. All of these were presented with advantageous energy and atom economies, cost-efficiency, and done so in an environmentally friendly manner. Paired electrochemical reactions not only avoid the generation of toxic waste by-products but also allow a more efficient use of electrical current at both electrodes to achieve specific products. These examples illustrate the diversity, as well as the common features, of a paired electrosynthesis that are performed mainly under mild conditions with good selectivities. Some paired syntheses required the presence of a catalyst such as iodide, Ni, Mn, Br, TEMPO, Ag, Au, and Sn, which could be regenerated at the anode or cathode by the end of electrolysis, and some reactions were carried out in the absence of a catalyst. Paired reactions can start with one or two substrates to generate one or two products. Also, both types of cells under batch and continuous flow conditions (undivided or divided cell) can be employed in paired electrosynthesis. Paired electrosynthesis allows, theoretically, a current efficiency of 200%. However, this can be quite difficult to realise, and therefore, more attention is needed to expand organic transformations under ideal paired electrolysis to access value-added compounds. Industrial scale applications of paired electrolysis are rare; as such a much greater emphasis needs to be given to such developments. The first commercial process using paired electrosynthesis by BASF allowed the simultaneous generation of phthalide and tert-butyl benzaldehyde dimethyl acetal with 100% atom efficiency and 180% current efficiency. Paired electrosynthesis prevents unwanted side reactions and protects the electrodes from corrosion by reactive species. Increased work will be required to perform paired electrosynthesis with more systematic and rational approaches to achieve optimum products under paired conditions. New computational tools could be applied in this field [52] Such an approach can be effective to improve atom economies at the possible expense of energy economy.

Acknowledgements

None.

Conflict of Interest

None.

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