

Electrochemical double carboxylation of unsaturated C-C bonds with carbon dioxide: An overview

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ABSTRACT

In this review, we try to provide a comprehensive and updated overview of recent advances on the synthesis of dicarboxylic acids through electrochemical decarboxylation of unsaturated hydrocarbons with carbon dioxide. We have classified these reactions based on the type of starting materials. Thus, the review is divided into three major sections. The first section will cover dicarboxylation of alkenes. The second focuses exclusively on dicarboxylation of dienes. The third will discuss dicarboxylation of alkynes.

1. Introduction

Carbon dioxide (CO₂) is the primary greenhouse gas emitted through human activities and caused serious environmental problems [1]. On the other hand, it is an easily accessible, abundant, nontoxic, nonflammable, and renewable source of carbon [2-10]. Therefore, chemical fixation of carbon dioxide into valuable organic compounds is of great importance from the viewpoints of environmental and green chemistry. However, CO₂ is a thermodynamically and kinetically stable molecule which its activation typically requires large energy inputs (e.g., drastic reaction conditions and innovative catalytic processes) [11]. In fact, efficient chemical incorporation of CO₂ is specifically limited to the use of highly reactive molecules such as aziridines [12] and epoxides [13]. Therefore, development of novel strategies for truly efficient chemical transformations of CO₂ is an exceedingly attractive research subject in modern organic synthesis.

Organic electrosynthesis is an extremely versatile and powerful synthetic tool that circumvents the use of expensive catalysts, oxidants or ligands, thus being framed within the principles of green chemistry [14]. In this context, electrochemical methods have been widely used for the synthesis of various value-added chemicals utilizing CO₂ as a C1-building block [15]. Among various electrochemical CO₂-fixation reactions, carboxylation of

carbon nucleophiles by reaction of CO₂ has attracted much attention in recent years. In 2014, De Vos and co-workers highlighted this synthetic strategy in their interesting review paper entitled "electrocarboxylation: towards sustainable and efficient synthesis of valuable carboxylic acids" [16]. However, double carboxylation of unsaturated C-C bonds was omitted. In continuation of our reviews of new methodologies on chemical conversion of CO₂ [17] and modern organic synthesis [18], herein, we will highlight the most important developments on the electrochemical dicarboxylation of unsaturated C-C bonds. The review is divided into three major sections. The first section will cover dicarboxylation of alkenes. The second focuses exclusively on dicarboxylation of dienes. The third will discuss dicarboxylation of alkynes.

2. Double carboxylation of alkene

In 1992, Duñach and co-workers published one of the earliest examples of the dicarboxylic acids preparation through the electro-dicarboxylation of corresponding alkenes with CO₂ [19].

They showed that the treatment of a library of styrene derivative **1** with atmospheric CO₂ in the presence of a catalytic amount of Ni-PMDDTA in an undivided cell with magnesium anode and carbon fiber cathode in DMF

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produced the corresponding dicarboxylic acids **2** in poor to moderate yields (Scheme 1).

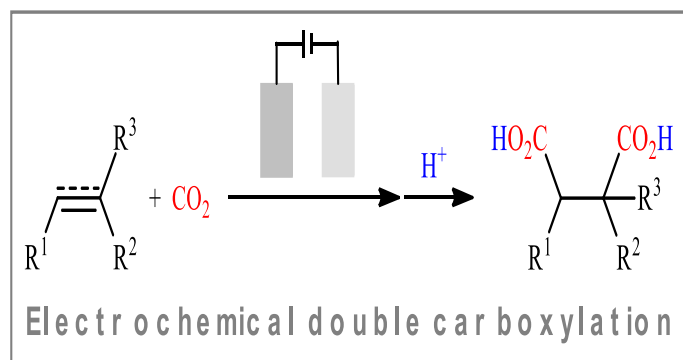
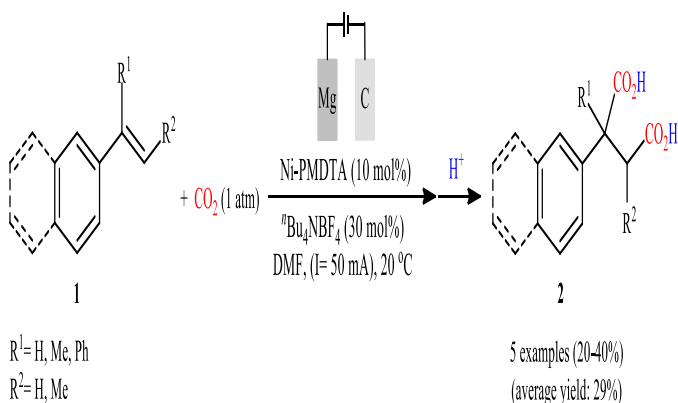


Figure 1. Electrosynthesis of dicarboxylic acids utilizing CO₂ as carboxyl source.

The results demonstrated that α -unsubstituted styrenes were more reactive than 1,1-disubstituted alkenes. Noteworthy, this protocol was not successful in the case of α,β -disubstituted styrenes.

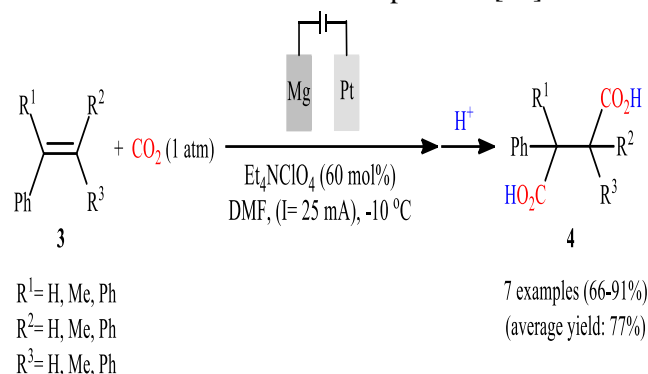


Scheme 1. Ni-catalyzed electrochemical double carboxylation of styrenes **1** with CO₂.

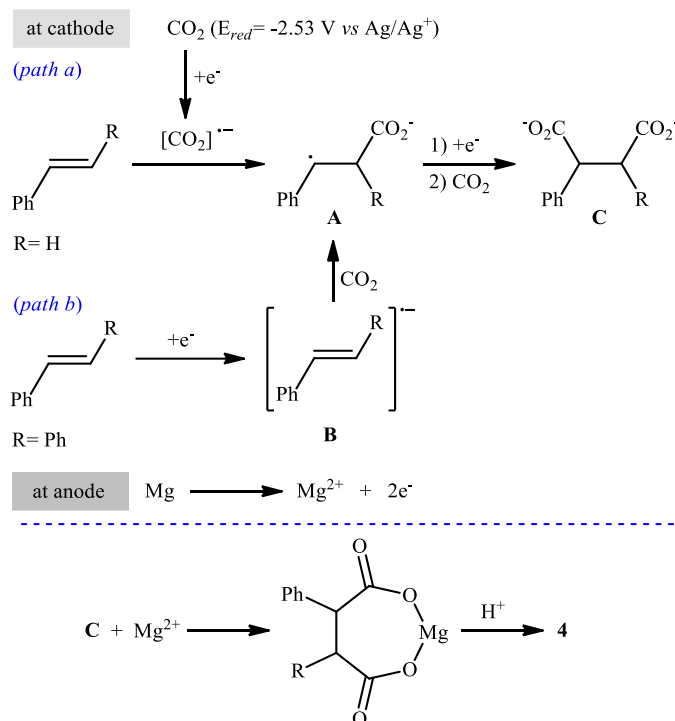
In 2001, Senboku, Tokuda, and co-workers described another efficient electrochemical method for dicarboxylation of alkenes using atmospheric CO₂ as the carboxyl source [20]. The electrolysis was performed with a magnesium rod anode and a platinum plate cathode in a DMF solution containing 0.1 M Et₄NClO₄ under a constant current of 25 mA (Scheme 2). The transformation was suitable for several phenyl-substituted terminal and internal alkenes **3** and the desired 1-phenylalkane-1,2-dicarboxylic acids **4** were obtained in good to excellent yields (7 examples, 66–91%). Cyclic voltammetry studies indicated that CO₂ and phenyl-substituted alkenes have similar reduction potentials: thus, two reaction pathways are possible (Scheme 3). For alkenes have more negative reduction potentials than that of CO₂, the reduction of CO₂ (−2.53 V vs Ag/Ag⁺) occurs preferentially whereupon the resulting CO₂ radical anion can add onto the alkene (Scheme 3, path a). Conversely, for alkenes whose reduction potentials are almost the same or slightly more positive than that of CO₂, alkene reduction predominates and the resulting carbanion can

be trapped by CO₂ (Scheme 3, path b). Both processes can be operative at the same time.

Six years later, Jiang's research group developed a similar protocol for the dicarboxylation of aryl-substituted alkenes **5** at room temperature [21].

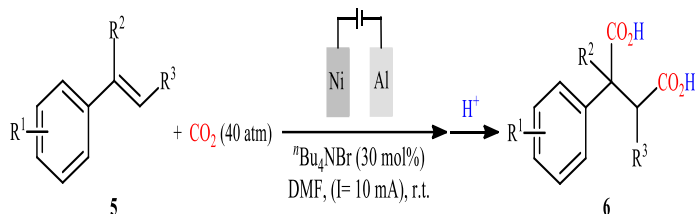


Scheme 2. Electrocatalytic metal-free dicarboxylation of phenyl-substituted alkenes **3** with atmospheric CO₂.



Scheme 3. Proposed mechanism for electrochemical dicarboxylation of alkenes **3** with atmospheric CO₂.

In an undivided cell assembled with an aluminum plate anode and nickel plate cathode, the best reaction conditions were achieved with DMF as the solvent and ⁿBu₄NBr as the electrolyte, with a constant current of 10 mA. Under the optimized condition, a library of 2-arylsuccinic acids **6** were synthesized in moderate to high yields (Scheme 4). However, a high pressure of CO₂ (40 atm) was required to furnish the required products. It should be mentioned that when *p*-chlorostyrene and β -bromostyrene used as substrates, one CO₂ molecule was preferentially added to the C–Cl or C–Br bond instead of the C=C double bond to produce mono-carboxylic acid as main product. The authors proposed a mechanism analogous to that of Senboku, Tokuda, and co-workers.



$R^1 = \text{H, 4-Me, 4-OMe, 4-F, 3,4-(CH=CH)}_2$

$R^2 = \text{H, Me}$

$R^3 = \text{H}$

$R^2 + R^3 = \text{-(CH}_2)_4$

7 examples (50-87%)

(average yield: 76.5%)

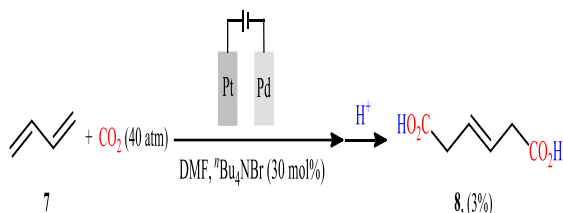
Scheme 4. Jiang's synthesis of 2-arylsuccinic acids **6**.

3. Double carboxylation of dienes

In 1997, the group of Grinberg investigated the possibility of synthesizing 3-hexenedioic acid **8**, a precursor of adipic acid, by cathodic double carboxylation of 1,3-butadiene **7** with CO_2 (Scheme 5) [22]. The electrolysis was performed with a platinum plate anode and a lead plate cathode in DMF with Bu_4NBr as the supporting electrolyte. However, the desired product was obtained in only 3% yield.

The author explained the poor efficiency of this reaction by the competing reductive debromodimerization and oligomerization of the *in situ* generated 1,4-dibromobut-2-ene. Although only one low yield example was disclosed, this paper represents the first example of dicarboxylation of dienes using CO_2 under electrochemical conditions.

According to the author proposed mechanism, this reaction proceeds through the bromination of starting 1,3-butadiene by Bu_4NBr on anode followed by carboxylation of generated 1,4-dibromobut-2-ene on cathode (Scheme 6).

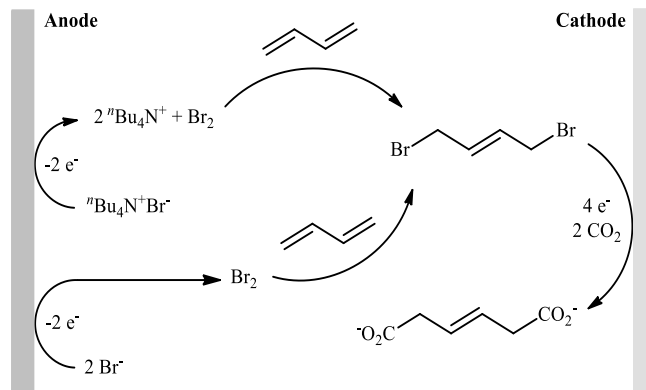


Scheme 5. Electrocarboxylation of 1,3-butadiene **7** with CO_2 .

In 2010, Wang and Lu along with their co-workers restudied Grinberg's discovery [23]. With stainless steel cathode and magnesium rod sacrificial anode using TBAF as a supporting electrolyte and DMF as the solvent, at -10°C the authors observed that under low current values (5 mA) the isoprene **9** was dicarboxylated with atmospheric CO_2 to 3-methylhex-3-enedioic acid **10** in fair yield (Scheme 7).

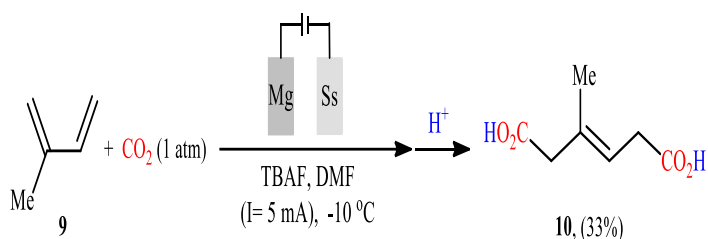
However, 2,3-dimethylbuta-1,3-diene failed to participate in this electrochemical reaction and methyl hexa-2,4-dienoate provided the corresponding mono-carboxylated product instead of the desired carboxylated product. Two possible mechanisms were proposed for this transformation (Scheme 8). In pathway A, CO_2 is

reduced to a reactive $\text{CO}_2^{\cdot-}$ radical anion by one-electron uptake from the cathode. With the addition of the $\text{CO}_2^{\cdot-}$ to C=C double bond, C-centered radical **A** is generated. This radical is converted to dicarboxylate anion **B** via another reductive carboxylation process. Finally, through the hydrolysis of intermediate **B**, the expected 3-methylhex-3-enedioic acid **10** is formed.

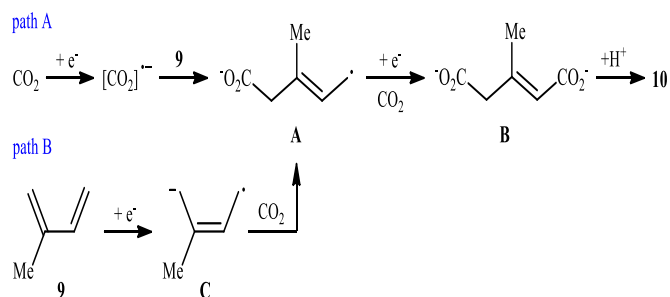


Scheme 6. Proposed mechanism for the formation of 3-hexenedioic acid **8**.

On the other hand, in pathway B through the cathodic reduction of isoprene **9**, the reactive species **C** is generated, which after carboxylation transformed to the radical **A**. It has been illustrated that both pathways may be operative at the same time. Later, Yuan, Jiang and co-workers improved the efficiency of this reaction by performing the process in an undivided cell equipped with a nickel cathode and an aluminum anode under constant current of 10 mA at room temperature [24]. Beside isoprene, other diene derivatives such as buta-1,3-diene, 2,3-dimethylbuta-1,3-diene, 1,4-diphenylbuta-1,3-diene, and cyclohexa-1,3-diene were also responded to this electrochemical reaction.

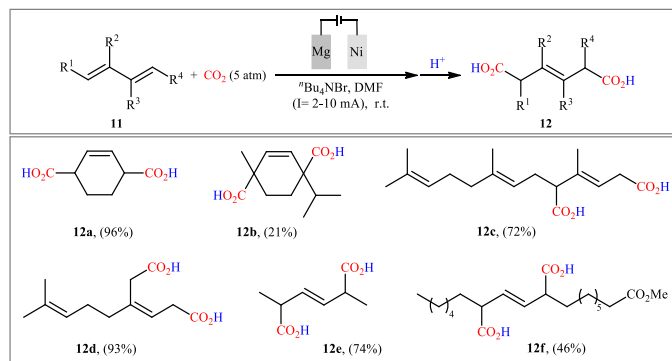


Scheme 7. Electrocarboxylation of isoprene **9** with atmospheric CO_2 reported by Wang and Lu.



Scheme 8. Mechanism that accounts for the formation 3-methylhex-3-enedioic acid **10**.

Following these works, De Vos and colleagues reported further examples of dicarboxylic acids synthesis through electro-carboxylation of the diene derivatives [25]. They showed that in an undivided cell assembled with a Ni cathode and Mg anode in the presence of ${}^n\text{Bu}_4\text{NBr}$ as the supporting electrolyte, various cyclic and acyclic dienes **11** underwent dicarboxylation with CO_2 to provide the respective dicarboxylic acids **12** in moderate in excellent yields (Scheme 9). Subsequently, another electrochemical method has also been developed by the same authors for double carboxylation reaction of the same set of dienes with CO_2 , albeit under milder conditions (atmospheric CO_2) [26].



Scheme 9. De Vos's electrosynthesis of dicarboxylic acids **12**.

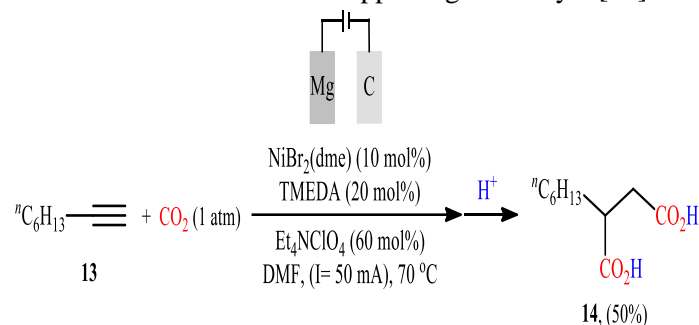
4. Double carboxylation of alkynes

In 1988, Labbé and co-workers reported one of the earliest methods for the direct dicarboxylation of alkynes with CO_2 under electrochemical conditions [27]. They showed that electrochemical carboxylation of 1-octyne **13** with atmospheric CO_2 in an undivided cell assembled with a magnesium sacrificial anode and a carbon cathode employing ${}^n\text{Bu}_4\text{NBF}_4$ as the supporting electrolyte and $\text{NiBr}_2(\text{dme})/\text{TMEDA}$ combination as a catalytic system, under 50 mA in DMF at 70°C , gave 2-hexylsuccinic acid **14** in a 50% yield (Scheme 10).

It should be mentioned that the outcome of this reaction was strongly dependent to the on the nature of catalyst and ligand. When the reactions were conducted in the presence of $\text{NiBr}_2(\text{dme})$ catalyst and DABCO ligand, non-2-enoic acid was obtained as the main product (50%), while performing the reaction in the presence of $\text{Ni}(\text{cyclam})\text{Br}_2/\text{cyclic tetraamine cyclam}$ combination as the catalytic system afforded non-2-ynoic acid as the main product (96%). Unfortunately, the authors did not investigate the substrate scope of this methodology. Moreover, they were silent regarding the possible mechanistic pathway of this transformation. Subsequently, the same authors extended the substrate scope of their methodology to internal alkynes using $\text{Ni}(\text{bipy})_3(\text{BF}_4)_2$ as the catalyst [28]. However, in all examples a mixture of mono- and di-carboxylated derivatives were obtained.

Two decades later, Jiang and colleagues devised a highly efficient electrochemical double carboxylation method of arylacetylenes employing CO_2 (30 atm) in an

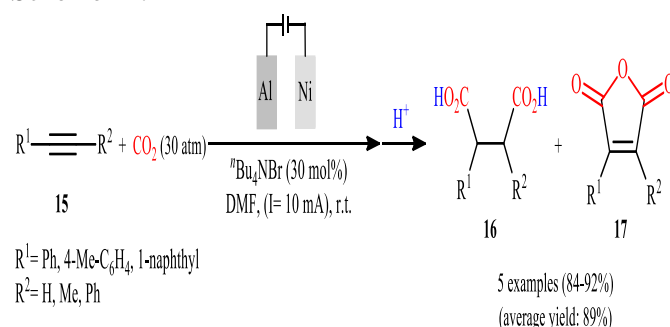
undivided cell using Ni as the cathode and Al as the anode with ${}^n\text{Bu}_4\text{NBr}-\text{DMF}$ as the supporting electrolyte [29].



Scheme 10. Nickel-catalyzed electrochemical dicarboxylation of 1-octyne **13**.

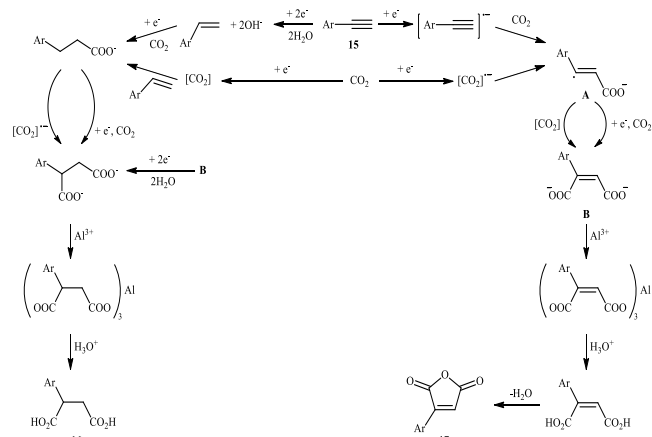
The reactions were conducted in the absence of any additional catalysts at room temperature, tolerated both terminal and internal alkynes **15**, and generally afforded the corresponding aryl-maleic anhydrides **16** and 2-arylsuccinic acids **17** in excellent total yields (Scheme 11).

Noteworthy, under anhydrous conditions, an unsaturated aryl-maleic anhydride as the main product was obtained, while the presence of water would lead to the formation of saturated 2-arylsuccinic acids. Based on some control experiments, the authors proposed a plausible mechanism for this reaction as depicted in Scheme 12.

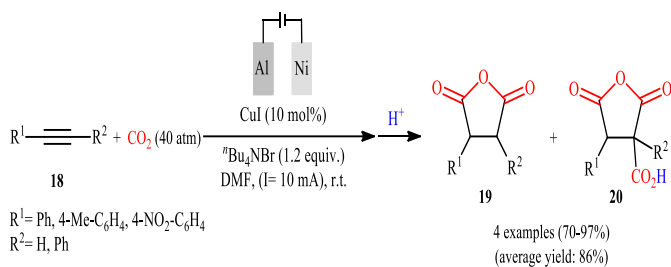


Scheme 11. Catalyst-free electrochemical dicarboxylation of alkynes **15**.

Following these works, the group of Huanfeng reported an interesting CuI -catalyzed electrocarboxylation of arylacetylenes with CO_2 at room temperature (Scheme 13) [30]. An undivided cell was assembled with an aluminum electrode as the anode and nickel electrode as the cathode. The transformation was performed under constant current of 10 mA and several terminal and internal aryl alkynes **18** were evaluated, providing the dicarboxylic acids **19** (7-27%) besides the saturated tricarboxylic acids **20** (63-83%) in high total yields. The results indicated that yield and ratio between di- and tri-carboxylated products were strongly influenced by catalysts and cathode materials. Therefore, it is possible with the development of suitable electrolytic cells and/or catalysts, restrict tricarboxylation and enhance dicarboxylic acids products.



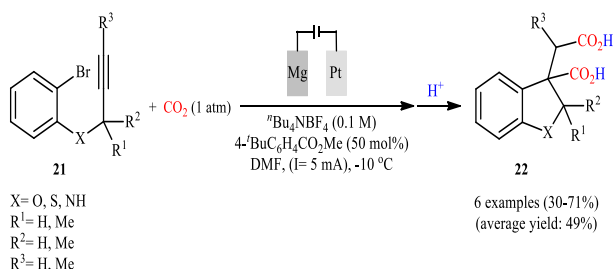
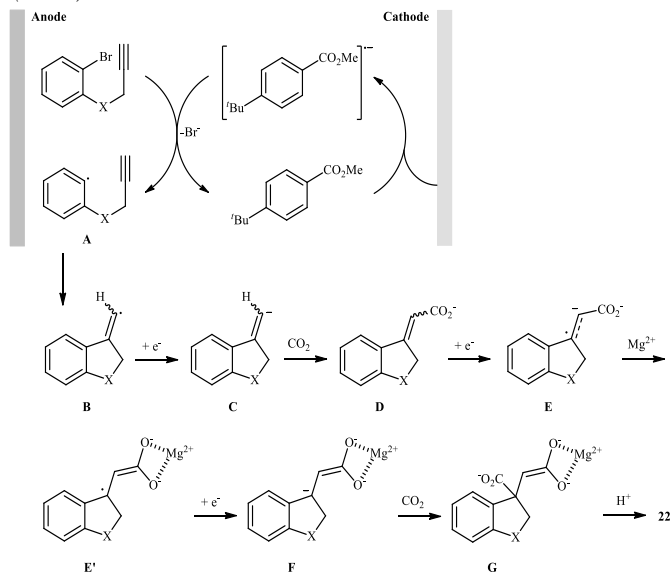
Scheme 12. Plausible mechanism for the reaction in Scheme 11.

Scheme 13. CuI-catalyzed electrocarboxylation of arylacetylenes **18** with CO_2 .

In 2016, Senboku and co-workers reported an interesting protocol for electrosynthesis of 2,2-ring-fused succinic acid derivatives **22** from the reaction between propargyl-substituted 2-bromo-phenol, -thiophenol, and -aniline derivatives **21** and carbon dioxide through an intramolecular radical cyclization/dicarboxylation sequential process (Scheme 14) [31]. The best conversion efficiency was obtained for the reactions which conducted in an undivided cell equipped with a Pt cathode and an Mg anode under constant current of 5 mA at -10°C .

It should be mentioned that presence of methyl 4-tert-butylbenzoate as an electron transfer mediator was crucial for the success on this tandem reaction. In the absence of an electron transfer mediator, two-electron reduction of aryl bromide competitively occurs at the cathode to generate the corresponding aryl anion species, directly producing carboxylated benzoic acid.

The author proposed mechanism for this tandem reaction is depicted in Scheme 15.

Scheme 14. Electrochemical tandem approach to 2,2-ring-fused succinic acid derivatives **22**.

Scheme 15. Probable reaction mechanism of the

electrosynthesis of 2,2-ring-fused succinic acid derivatives **22**. At the beginning, one-electron reduction of aryl bromide **21** by the *in situ* generated radical anion of methyl 4-tert-butylbenzoate produces the anion radical of **21** that, after a carbon-bromine bond cleavage affords aryl radical **A**. Subsequently, intermediate **A** undergoes an intramolecular 5-*exo* cyclization to give the corresponding cyclized vinyl radical **B** which after cathodic reduction converts to the corresponding vinyl anion **C**. Later, reaction of intermediate **C** with CO_2 generates α,β -unsaturated carboxylate ion **D** that, after one-electron reduction transforms to the radical anion intermediate **E**. In the next step, the newly formed anion **E** undergoes further one-electron reduction to form benzylic anion **F**. Afterwards, fixation of another molecule of CO_2 at the benzylic position of **F** gives dicarboxylate ion **G**. Finally, acidic workup of **G** provides the final product **22**.

5. Conclusion

Electrochemical CO_2 conversion represents a promising and eco-friendly strategy for CO_2 utilization and a green alternative for the synthesis of many industrially relevant chemicals. Electrocaraoylation of carbon nucleophiles and electrohydrocarboxylation of unsaturated hydrocarbons are continuously being reported. However, little progress has been made in the synthesis of dicarboxylic acids through electrochemical dicarboxylation of unsaturated C-C bonds. As illustrated, various unsaturated hydrocarbons such as alkenes, alkynes, and dienes can easily participate in this reaction and provide the corresponding dicarboxylic acids. This research topic has still further possibilities for growth and we anticipated that the highly versatile and extremely effective electrosynthesis of biologically important dicarboxylic acids utilizing CO_2 as a C1 source will be attainable in the near future.

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