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Review

Recent advances towards electrochemical transformations of α-keto acids

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ABSTRACT

As a kind of environmentally benign reagents, α-keto acids have been extensively employed as key starting materials in organic synthesis. Organic electrosynthesis has the advantages of reducing byproduct generation, improving the cost-efficiency of synthetic processes, and accessing reactive intermediates under mild conditions. Inspired by the merits of organic electrosynthesis, α-keto acids have shown many synthetic applications in electrochemical acylation, cyclization, and reductive amination reactions with improved efficiencies and selectivities. This review covers the recent breakthroughs achieved in the electrochemical transformations of α-keto acids, aimed at highlighting these electrochemical reactions' features and mechanistic rationalisations. Meanwhile, the practicalities and limitations of these transformations are also presented where possible.

1. Introduction

α-Keto acids are a class of organic compounds containing a keto function adjacent to a carboxylic acid group (Fig. 1). α-Keto acids are wildly occurred in the energy-supplying biochemical processes [1,2]. For example, pyruvate **1** is an important metabolite in the Krebs cycle, also called tricarboxylic acid cycle, which triggers a number of enzyme-catalyzed reactions to give adenosine triphosphate (ATP). Pyruvate **1** can also undergo condensation reaction with carbonic acid utilizing pyruvate carboxylase as the catalyst, affording dianion **2** which is another central metabolite in the Krebs cycle (Scheme 1). Considering the importance of α-keto acids in biochemical processes, the discovery of new chemical properties of α-keto acids is of great significance. Since Gooßen *et al*. reported the first example of decarboxylative coupling of aryl bromides with α-keto acids, the use of α-keto acids as green acyl surrogates has been extensively investigated since this type of reactions excluded nontoxic CO_2 as the by-product [3]. Recently, some nice reviews have highlighted the advances in metal- or photo-catalyzed decarboxylative couplings of α-keto acids [4,5].

Organic electrosynthesis employs electrons as the clean redox reagents, thus avoiding the use of harmful and dangerous chemical redox reagents [6–13]. Apart from the advantages of reducing byproduct generation and improving the cost-efficiency of synthetic processes, organic electrochemistry has the ability to access reactive intermediates under mild conditions, which are challenging for conventional approaches. Recently, organic electrosynthesis has experienced a renaissance after being overlooked by synthetic chemists for many years [14–25]. Inspired by the merits of organic electrosynthesis, many synthetic applications of α-keto acids under electrochemical conditions have been described in recent years. However, there is no systematic review dealing with this topic. Herein, we present the recent breakthroughs achieved in the electrochemical transformations of α-keto acids. This review can be classified into three categories: electrochemical decarboxylative acylation reactions, electrochemical decarboxylative cyclization reactions, and electrochemical reductive amination reactions. In addition to the description of advantages of these methodologies, we also want to point out the limitations to the readers.

2. Electrochemical decarboxylative acylation reactions

Minisci acylation reaction provides a facile route to obtain valuable carbonyl-substituted *N*-heterocycles, which is challenging for Friedel-Crafts acylation reactions [26–29]. Typically, Minisci acylation reactions employed Fe or Ag salts as the catalysts and $(NH_4)_2S_2O_8$ as the oxidant [30,31]. In 2017, we reported an

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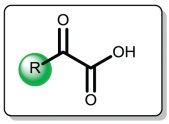
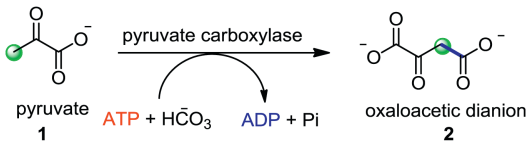


R = H, alkyl, (hetero)aryl

α-keto acid

Fig. 1. The structure of α-keto acids.

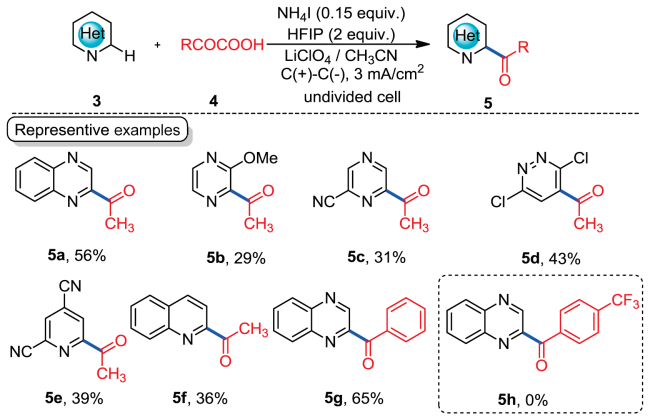


Scheme 1. Biochemical transformation of pyruvate.

electrochemical decarboxylative Minisci acylation reaction under metal- and chemical oxidant-free conditions with α-keto acids as acyl surrogates (Scheme 2) [32]. This reaction was carried out in an undivided cell with graphite as the electrodes, $LiClO_4/CH_3CN$ as the electrolytic solution, readily available NH_4I as the catalyst, and hexafluoroisopropanol (HFIP) as the additive under controlled-current electrolytic (CCE) conditions. A diverse of *N*-heterocycles such as quinoxaline (**5a**, **5g**), pyrazine (**5b**, **5c**), pyridazine (**5d**), pyridine (**5e**), and quinoline (**5f**) were all suitable substrates under the electrolytic conditions. Besides, aliphatic and aromatic α-keto acids were all well-tolerated under the standard conditions. However, this methodology still has some limitations: 1) most of the *N*-heterocycles showed moderate efficiencies; 2) electron-withdrawing α-keto acid did not work under the optimal conditions (**5h**).

One of the advantages of this electrochemical protocol is the controllable formation of acyl radical under a relative low concentration. However, for chemical oxidant-mediated Minisci acylation reactions, acyl radical formation was finished in a short time. The high concentration of acyl radicals in the reaction mixture would lead to radical decomposition and poor selectivities. To demonstrate the selectivity advantage of this electrochemical protocol over traditional versions, the Minisci acylation of **3i** was carried out under different conditions. As shown in Scheme 3, when the reaction was carried out under Ag/$(NH_4)_2S_2O_8$-mediated conditions, the acylation occurred at C2 and C3 positions; however, the acylation only occurred at C2 position under electrocatalytic conditions.

The plausible mechanism for the electrochemical Minisci acylation reaction was shown in Scheme 4. Initially, the reaction

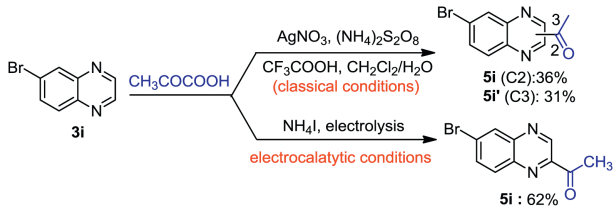


Scheme 2. NH_4I-catalyzed electrochemical Minisci acylation reaction.

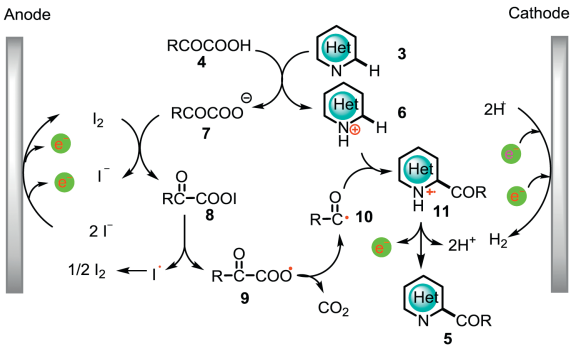
between *N*-heterocycle **3** and α-keto acid **4** gives protonated *N*-heterocycle **6** and carboxylate anion **7**. Then, carboxylate anion **7** reacts with anodically generated iodine to afford acyl hypoiodite **8**, which undergoes a homolytic cleavage to produce radical **9** and iodine radical. Aroyloxy radical **9** subsequently excludes CO_2 to give acyl radical **10**, which has a radical addition to **6** affording **11**. Finally, intermediate **11** undergoes a further oxidation and subsequently releases protons to give acylated product **5**. The controllable generation of acyl hypoiodite **8** as a mask of acyl radical is the key to the mono-selectivity of this electrochemical Minisci acylation reaction. The employment of HFIP as the additive not only assists the cathodic hydrogen evolution, but also stabilizes the generated radical species.

To overcome the substrate scope limitations of the above-mentioned methodology (Scheme 2), we want to develop a new catalytic system to facilitate the acyl radical formations from electron-deficient α-keto acids. In 2019, we reported the first example of Ni-catalyzed electrochemical Minisci acylation reaction with α-keto acids as acyl surrogates (Scheme 5) [33]. This reaction was carried out in an undivided cell equipped with Pt anode and graphite cathode with n-Bu_4NBF_4/DCM/CH_3CN as the electrolytic solution under CCE conditions. Electron-deficient α-keto acids and acridine were well-tolerated under this newly developed catalytic system. However, this methodology has its own limitations: 1) phenanthridine gave nonaromatic **14d** as the byproduct under the optimal conditions; 2) isoquinoline (**14h**) and benzothiazole (**14i**) did not work under the standard conditions.

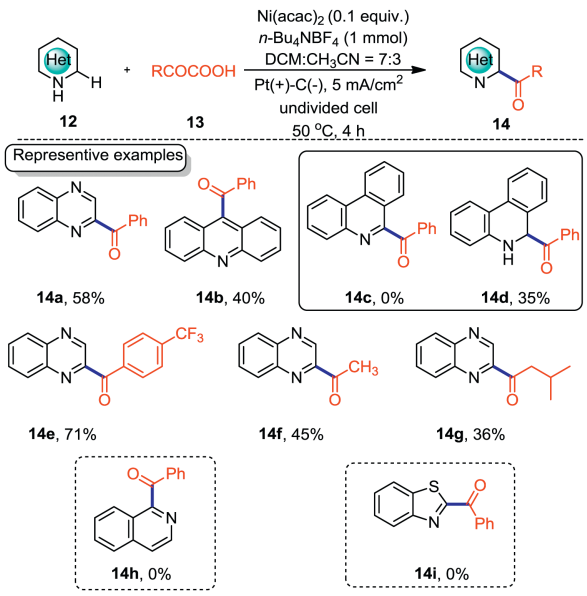
Control experiments showed that nickel catalyst is crucial for the decarboxylative formation of acyl radicals. Cyclic voltammetry (CV) experiments revealed that the oxidation potential of the complex of $Ni(acac)_2$ and 2-oxo-2-phenylacetic acid has a negative shift of 0.19 V compared with the oxidation potential of $Ni(acac)_2$. Based on the control experiments and CV studies, a plausible mechanism was shown in Scheme 6. The complex of $Ni(acac)_2$ and α-keto acids initially undergoes an anodic oxidation to give hypervalent nickel complex **15**, which has an inner-sphere electron transfer to produce aroyloxy radical **16**. Subsequent decarboxylation and radical addition afford radical cation **19**, which has



Scheme 3. Comparison of selectivities between conventional and electrochemical conditions.



Scheme 4. Mechanistic proposal for the formation of **5**.



Scheme 5. Nickel-catalyzed electrochemical Minisci acylation reaction.

further anodic oxidation followed by protons releasing to give valuable acylated *N*-heterocycle **14**. The hypervalent nickel complex **15** is a mask of acyl radical, which can afford acyl radical in a controllable concentration under electrolytic conditions.

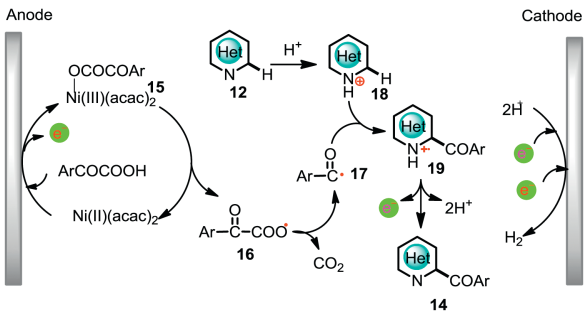
Enaminones are valuable structural motifs in natural products and pharmaceutically important compounds [34,35]. The construction of enaminone in a green manner is of great significance. In 2019, with acyl hypoiodite **8** as the mask of acyl radical as described in Scheme 4, Chen and Xu developed an electrochemical acylation of vinyl azides **20** with α-keto acids **21** (Scheme 7) [36]. This electrochemical protocol provides an environmentally benign route to enaminones, while previous report for the acylation of vinyl azides with α-keto acids needed CuI as the catalyst [37]. This decarboxylative coupling reaction was carried out in an undivided cell equipped with graphite anode and Pt cathode with *n*-Bu_4NI as the catalyst under CCE conditions. Under the optimal conditions, a diverse of electron-withdrawing vinyl azides were tolerated well to give enaminones in moderate yields (**22a**–**22c**). Heteroaryl substituted vinyl azides were also suitable substrates, affording the corresponding enaminones in moderate yields (**22d**–**22f**). Moreover, aliphatic α-keto acids underwent the acylation reaction smoothly with lower yields (**22g**–**22i**). However, the alkylated vinyl azide did not work under the standard conditions (**22j**).

The scale-up experiment demonstrated the practical utility of this electrochemical acylation reaction. As shown in Scheme 8, when the acylation reaction of **20a** and **21a** was carried out on a 10 mmol scale, the valuable acylated product **22a** was isolated in 81% yield.

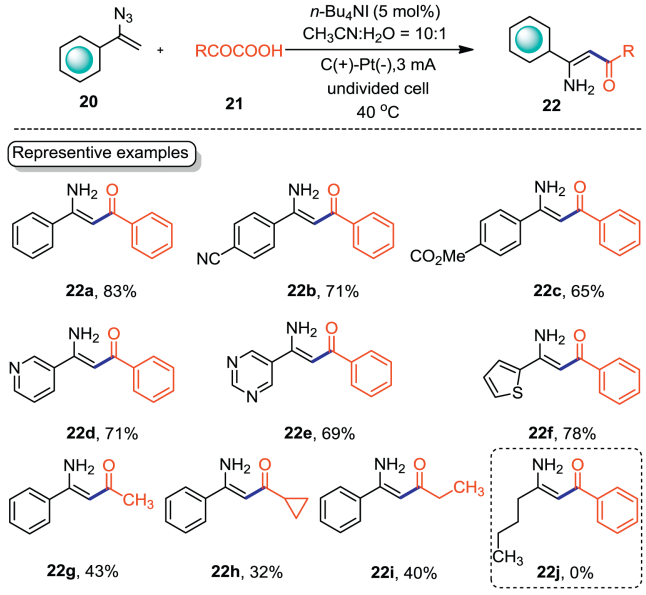
The plausible mechanism for the formation of **22a** was shown in Scheme 9. First, α-keto carboxylate anion **21a** reacts with anodically generated iodine to give acyl hypoiodite **23**, which undergoes homolytic cleavage and following decarboxylation to afford acyl radical **25**. Radical addition of **25** to vinyl azide **20a** gives radical **26**, which triggers an intramolecular hydrogen atom abstraction to produce carbon-centered radical **27**. Finally, further reduction of radical **27** followed by protonation affords the desired enaminone **22a**. Acyl hypoiodite **23** is a key intermediate for this electrochemical acylation reaction.

Formamides are always employed as important intermediates for the construction of value-added products in organic synthesis [38,39]. Besides, formamides are widely existed in many pharmaceutically relevant compounds [40,41]. As glyoxylic acid is readily available and stable, the decarboxylative coupling of glyoxylic acid with amines would provide an appealing alternative for the construction of formamides, since the reaction generates CO_2 as the sole byproduct. In 2018, Huang and co-workers realized an electrochemical *N*-formylation of diverse amines with the smallest α-keto acids, glyoxylic acid [42]. The electrochemical *N*-formylation reaction was carried out in an undivided cell equipped with Pt electrodes with Cu(II) and Ni(II) as the catalysts under CCE conditions. As shown in Scheme 10, this protocol could give various formamides in moderate to excellent yields. First, acyclic and cyclic secondary amines could be transformed into the corresponding formamides efficiently (**31a**–**31d**). Second, primary amines including aniline and heterocyclic amines were all compatible under the optimal conditions (**31e**–**31g**). However, aliphatic amines gave lower yields (**31d**, **31h**) owing to their low efficiencies of the corresponding condensation reactions with glyoxylic acid.

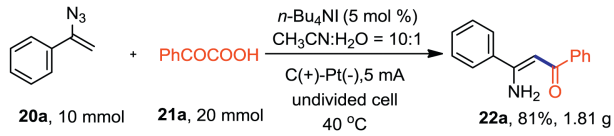
The plausible reaction pathway for the formation of formamide **31a** was shown in Scheme 11. Initially, the condensation of *N*-methylaniline **29a** with glyoxylic acid **30** gives intermediate **32**, which is oxidized by Cu(II) followed by decarboxylation to produce radical **34**. Meanwhile, Cu(I) is generated which can be re-oxidized to Cu(II) at the anode. Subsequently, radical **34** undergoes further single electron oxidation followed by proton releasing to give formamide **31a**. The Cu(II) species acts as an active oxidant which



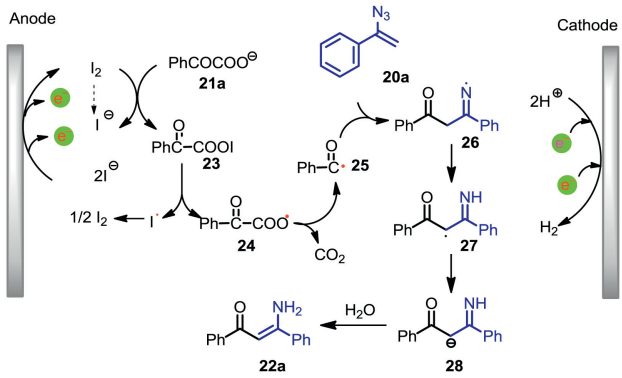
Scheme 6. Mechanistic proposal for the formation of **14.**



Scheme 7. NH_4I-catalyzed electrochemical acylation reaction.



Scheme 8. Scale-up experiment.

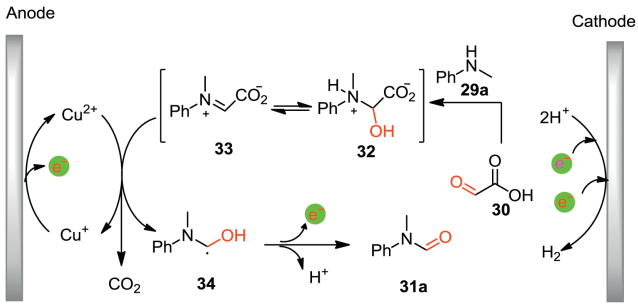


Scheme 9. A proposed reaction pathway for the formation of **22a**.

can be supported by the observation of catalytic current of Cu(II) catalyst in the CV experiments.

3. Electrochemical decarboxylative cyclization reactions

2-Substituted benzimidazoles are privileged building blocks for various natural products, and pharmaceutically important compounds [43,44]. Consequently, much effort has been devoted to the construction of 2-substituted benzimidazoles. In 2016, Huang and co-workers reported an electrochemical decarboxylative cyclization of diamines with α-keto acids to construct 2-substituted benzimidazoles (Scheme 12) [45]. This cyclization reaction was carried out in an undivided cell equipped with Pt electrodes with NH_4ClO_4/DMSO/H_2O as the electrolytic solution under CCE conditions. Aromatic α-keto acid underwent the decarboxylative cyclization smoothly to yield 2-substituted benzimidazole in excellent yield (**37a**). Aliphatic α-keto acids were also suitable substrates under the optimal conditions (**37c**, **37d**). Moreover, benzothiazole could also be obtained by this electrochemical protocol (**37e**). However, heteroaromatic α-keto acids as the

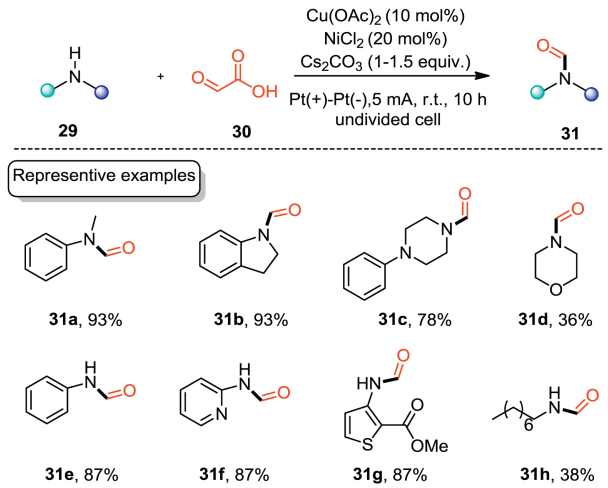


Scheme 11. A plausible reaction pathway for the formation of **31a**.

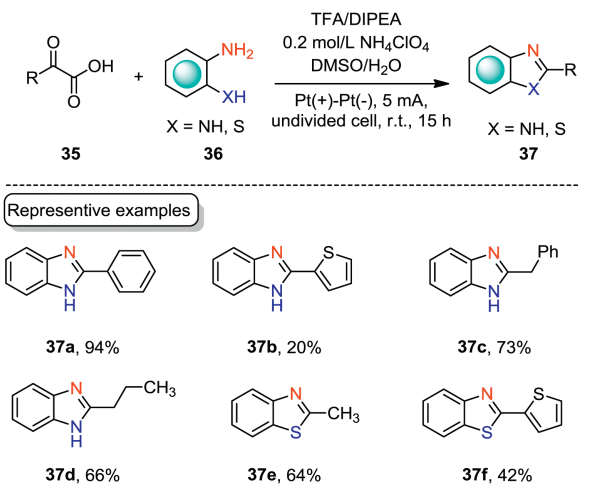
substrates only gave the corresponding products in low yields (**37b**, **37f**).

The plausible reaction pathway for the formation of benzimidazole **37a** was shown in Scheme 13. This decarboxylative cyclization proceeded *via* two pathways (path A and path B). For path A, 2-oxo-2-phenylacetic acid **35a** undergoes anodic oxidation and subsequent decarboxylation to give acyl radical **39**, which reacts with protonated diamine **36a** and then abstracts a hydrogen atom from anodically generated radical cation **40** to afford intermediate **42**. For path B, the condensation of **35a** and **36a** gives hemiaminal **46**, which undergoes decarboxylation and further H atom abstraction from anodically generated radical cation **40** to afford intermediate **42**. Then, intermediate **42** loses one molecule of proton followed by intramolecular cyclization to give **44**, which has further oxidation to produce benzimidazole **37a**. Previous report showed that the decarboxylation of hemiaminal **46** has a lower activation barrier than 2-oxo-2-phenylacetic acid **35a** [46]. Besides, iminocarboxylate **45** was detected during the electrolysis. Therefore, the authors believed that path B is preferable to path A.

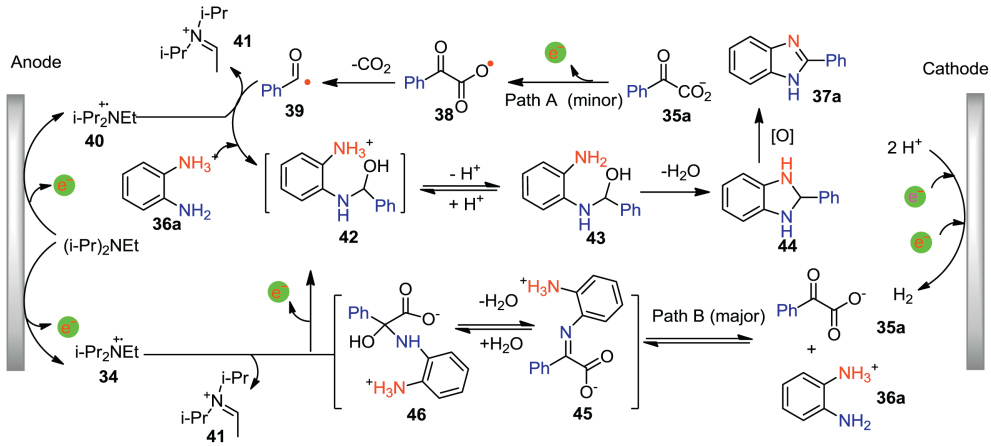
1,3,4-Oxadiazoles are widely existed in many pharmaceutically valuable agents and functional materials [47,48]. Thus, the green construction of 1,3,4-oxadiazoles has attracted much attention in recent years. In 2020, Gao, Zhang, Lei and co-workers developed an electrochemical decarboxylative cyclization of acylhydrazines with α-keto acids to furnish 1,3,4-oxadiazoles with high efficiencies (Scheme 14) [49]. The reaction was carried out in an undivided cell equipped with Pt electrodes with n-Bu_4NOAc/CH_3OH as the electrolytic solution under CCE conditions. For acylhydrazines, both aromatic and heteroaromatic acylhydrazines underwent the electrochemical cyclization smoothly to give the corresponding 1,3,4-oxadiazoles in good to excellent yields (**49a**–**49f**). Aliphatic



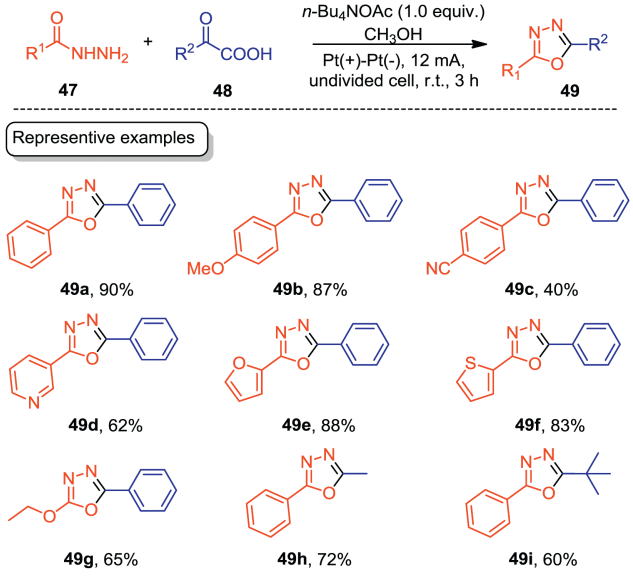
Scheme 10. Electrochemical *N*-formylation of amines with glyoxylic acid.



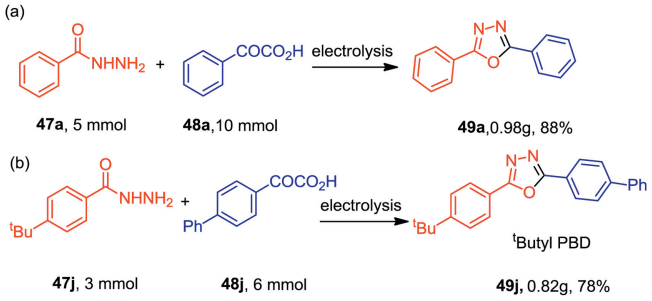
Scheme 12. Electrochemical decarboxylative cyclization of α-keto acids.



Scheme 13. A tandem reaction pathway for the formation of **37a**.



Scheme 14. Electrochemical synthesis of 1,3,4-oxadiazoles.



Scheme 15. Scale-up experiments.

acylhydrazine was also suitable substrate, giving 1,3,4-oxadiazole in 65% yield (**49g**). For α-keto acids, both aromatic and aliphatic α-keto acids were tolerated well under the optimal conditions. Compared with the previous reports dealing with 1,3,4-oxadiazoles syntheses, this protocol employed electron as the clean redox reagent, thus providing an appealing alternative for the green synthesis of 1,3,4-oxadiazoles.

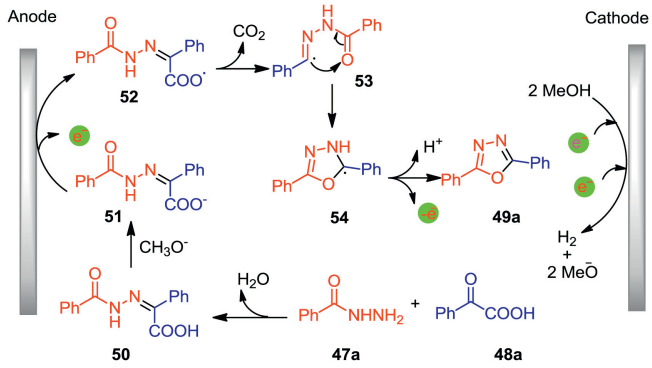
The practicability of this protocol was further demonstrated by the scale-up experiments. As shown in Scheme 15, the reactions can be carried out on a 5 mmol scale with high efficiencies. Moreover, compound **49j** which is important in the field of liquid scintillator neutrino detector can be obtained on a 0.82 g scale.

The plausible reaction pathway for the formation of 1,3,4-oxadiazole **49a** was shown in Scheme 16. First, the condensation of **47a** and **48a** gives imine **50**, which is deprotonated by cathodically generated MeO^- affording carboxylate anion **51**. Then, intermediate **51** has an anodic oxidation and subsequent decarboxylation to yield radical **53**, which undergoes further intramolecular cyclization to give intermediate **54**. Finally, intermediate **54** loses one electron and one molecule of proton to yield 1,3,4-oxadiazole **49a**. The proposed radical pathway and the formation of imine **50** have been demonstrated by the radical trapping experiments and control experiments.

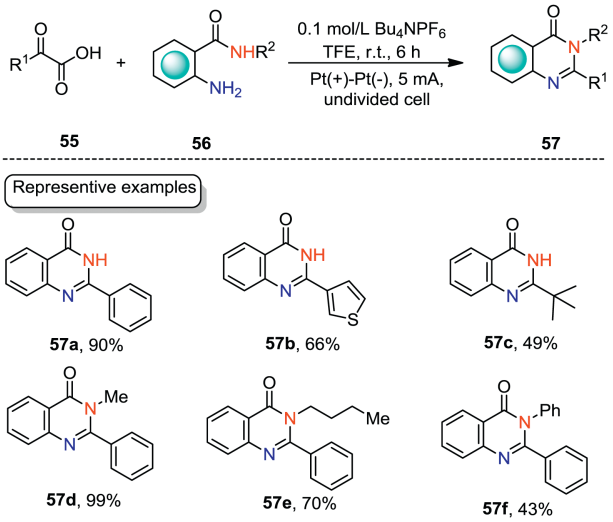
Quinazolinone skeleton represents one of the privileged structural motifs in natural alkaloids, and pharmaceutical reagents [50–52]. The construction of quinazolinones in an environmentally benign manner is one of the pursuits of organic chemists. In 2021, Wei and co-workers developed an electrochemical decarboxylative cyclization of 2-aminobenzamides with α-keto acids for the synthesis of quinazolin-4(3*H*)-ones under mild conditions (Scheme 17) [53]. The electrochemical cyclization was carried out in an undivided cell equipped with Pt electrodes with *n*-Bu_4NPF_6 in TFE as the electrolytic solution under CCE conditions. When R^2 = H, 2-oxo-2-phenylacetaic acid, 2-oxo-2-(thiophen-3-yl) acetic acid, and aliphatic α-keto acid were all suitable substrates, affording the corresponding quinazolin-4(3*H*)-ones with up to 90% yield (**57a**–**57c**). When 2-amino-*N*-substituted benzamides were employed as the substrates, the reactions could also underwent the cyclization reaction smoothly (**57d**–**57f**).

In addition to 2-aminobenzamide, 2-aminobenzensulfonamide **58** could also undergo decarboxylative cyclization with α-keto acid to give the desired product **59** in 67% yield (Scheme 18).

The plausible mechanism for the formation of **57a** was shown in Scheme 19. The condensation of **55a** with **56a** gives imine **60**, which releases one molecule of proton to yield carboxylate anion **61**. The anodic oxidation of **61** followed by decarboxylation leads to the formation of radical **63**, which undergoes an intramolecular cyclization to give carbon-centered radical **64**. Finally, further anodic oxidation of radical **64** and subsequent deprotonation affords quinazolin-4(3*H*)-one. Compared with previous reports on quinazolin-4(3*H*)-ones syntheses, this electrochemical protocol features metal-, base- and external oxidant-free conditions. However, the use of toxic TFE as the solvent is the drawback of



Scheme 16. A plausible reaction pathway for the formation of **49a**.

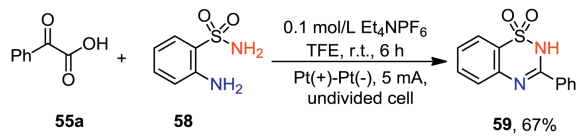


Scheme 17. Electrochemical synthesis of quinazolin-4(3*H*)-ones.

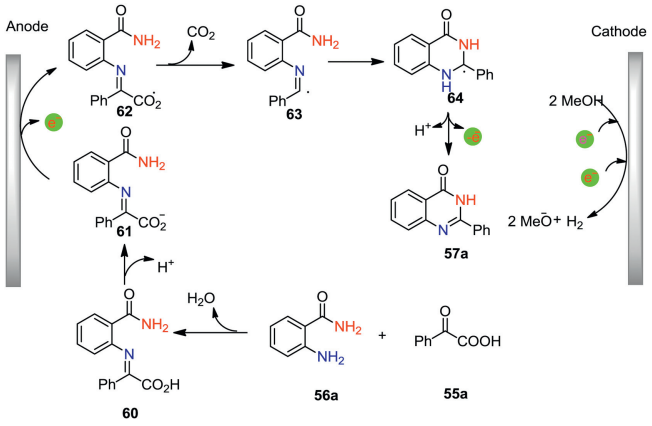
this protocol. Further investigations to avoid the use of toxic solvent would surely improve the synthetic potential of this methodology.

4. Electrochemical reductive amination reactions

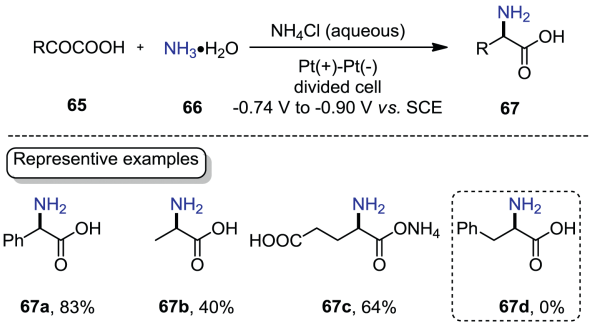
Amino acids play central roles as intermediates in metabolism and as building blocks in nature [54,55]. Considering the importance of amino acids, significant progress has been made on amino acids production. Among many synthetic protocols to produce amino acids, the most conventional approach for the chemical synthesis of amino acids is the Strecker reaction [56], which assembles amino acids from ammonia, cyanide, and aldehydes. However, the use of hazardous cyanide is the drawback of this reaction. To overcome this limitation, reductive amination of α-keto acids without consumption of hazardous reagents represents an appealing alternative to amino acid synthesis. Pioneered by Jeffery and co-workers [57,58], the electrochemical reductive amination of α-keto acids in aqueous ammonia has been



Scheme 18. Electrochemical cyclization of 2-aminobenzensulfonamide with α-keto acid.



Scheme 19. A mechanistic proposal for the formation of **57a**.

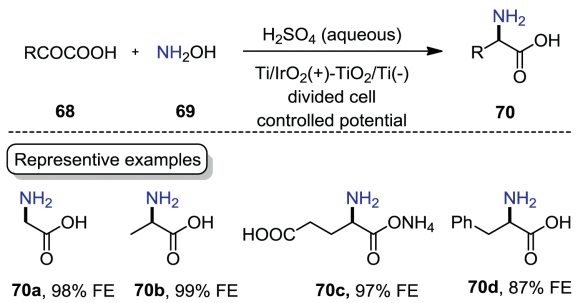


Scheme 20. Electrochemical synthesis of α-amino acids.

demonstrated to be a green approach for the synthesis of amino acids. As shown in Scheme 20, the electrolysis was carried out in a divided cell equipped with Pt electrodes in aqueous NH_3/NH_4Cl solution under constant potential conditions. 2-Oxo-2-phenylacetaic acid could give phenylglycine **67a** in 83% yield, while pyruvic acid gave alanine **67b** in lower yield. It is noteworthy that monoammonium glutamate **67c** could also be obtained in 64% yield under the optimal conditions. However, phenylalanine **67d** could not be produced by this method.

Even though the above-mentioned method could produce amino acids efficiently, the use of precious Pt electrodes limits its wide application. In 2019, Yamauchi and co-workers reported an electrochemical reductive amination of α-keto acids with NH_2OH on titanium dioxide [59]. As shown in Scheme 21, the reaction was carried out in a divided cell equipped with titanium dioxide electrode in aqueous H_2SO_4 under constant potential conditions. Glycine **70a**, alanine **70b**, and monoammonium glutamate **70c** were obtained with up to 99% Faraday efficiency (FE). Phenylalanine **70d** which cannot be obtained in previous report was synthesized with a 87% FE. This protocol features high FE values, which would reduce the cost-efficiency of synthetic process for amino acids production.

In conclusion, this review summarized the recent advances achieved in electrochemical transformations of α-keto acids. The introduction of electrochemistry further enriches the synthetic utilities of α-keto acids. α-Keto acids can be employed as green acyl surrogates in electrochemical acylation and cyclization reactions to afford value-added chemicals. Besides, electrochemical reductive amination of α-keto acids has been demonstrated to be an appealing alternative to produce amino acids, which are key building blocks in nature. Even though substantial progress has been made on electrochemical transformations of α-keto acids,



Scheme 21. Electrochemical reductive amination of α-keto acids on titanium dioxide.

some challenges still remain: 1) Further improvement of the efficiencies of heteroaromatic and aliphatic α-keto acids under electrochemical conditions would expand the synthetic applications of α-keto acids; 2) The electrosynthesis of amino acids *via* reductive aminations of α-keto acids in an undivided cell under CCE conditions would be another challenge in this avenue.

Declaration of competing interest

The authors report no declarations of interest.

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