



Electro-reductive carboxylation of C–Cl bonds in unactivated alkyl chlorides and polyvinyl chloride with CO₂

Li Li^{a,1}, Zhi-Xin Yan^{a,1}, Chuan-Kun Ran^{a,1}, Yi Liu^a, Shuo Zhang^a, Tian-Yu Gao^a, Long-Fei Dai^a, Li-Li Liao^{b,*}, Jian-Heng Ye^a, Da-Gang Yu^{a,*}

^a Key Laboratory of Green Chemistry & Technology of Ministry of Education, College of Chemistry, Sichuan University, Chengdu 610064, China

^b School of Chemistry and Chemical Engineering, Chongqing Key Laboratory of Chemical Theory and Mechanism, Chongqing University, Chongqing 400030, China

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ABSTRACT

The carboxylation of readily available organo halides with CO₂ represents a practical strategy to afford valuable carboxylic acids. However, efficient carboxylation of inexpensive unactivated alkyl chlorides is still underdeveloped. Herein, we report the electro-reductive carboxylation of C–Cl bonds in unactivated chlorides and polyvinyl chloride with CO₂. A variety of alkyl carboxylic acids are obtained in moderate to good yields under mild conditions with high chemoselectivity. Importantly, the utility of this electro-reductive carboxylation is demonstrated with great potential in polyvinyl chloride (PVC) upgrading, which could convert discarded PVC from hydrophobic to hydrophilic functional products. Mechanistic experiments support the successive single electron reduction of unactivated chlorides to generate alkyl anion species and following nucleophilic attack on CO₂ to give desired products.

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It is highly desirable to synthesize useful and sought-after compounds from readily available starting materials under mild conditions with ease of operation. Various investigations have shown that the production of valuable carboxylic acids using carbon dioxide (CO₂) as C1 source is an attractive strategy due to the abundance, availability, sustainability, and non-toxicity of CO₂ [1–23]. Among diverse methods to produce carboxylic acids, carboxylation of organo halides with CO₂ is considered as one of the most attractive and practical approaches [24–31]. One representative strategy is conversion of organo halides to organometallic reagents, including Grignard, organolithium and organozinc reagents, which could react with CO₂ to give carboxylic acids [32–47]. However, the practical application of this method is limited by the preparation, storage and use of the air and moisture-sensitive organometallic reagents. Meanwhile, transition metal-promoted or -catalyzed carboxylation of organo halides with CO₂ has emerged as an efficient and highly promising strategy to generate a variety of valuable carboxylic acids [48–59]. Compared with the widely investigated carboxylation of alkyl bromines and iodides with CO₂, those transformations with unactivated alkyl chlorides, which are more read-

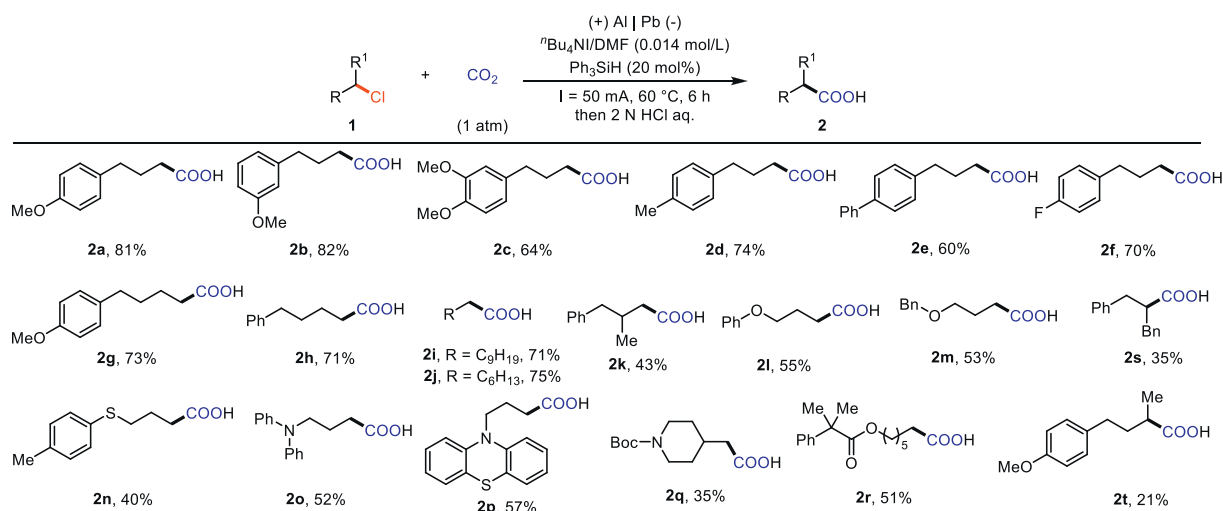
ily available and inexpensive, were much less investigated due to the higher bond energy of C(sp³)–Cl cleavage. Notably, Martin reported a leading work in this field on nickel-catalyzed carboxylation of alkyl chlorides with CO₂ under mild reaction conditions (Scheme 1A) [60]. However, excess of Mn powder was used as reductant, which might cause safety issue in large-scale synthesis. Therefore, it is still highly desirable to develop other sustainable strategies to realize safe and user-friendly carboxylation of unactivated alkyl chlorides.

As well-known, electrosynthesis has proven to be an efficient and environmentally friendly method and is increasingly pursued as a sustainable synthetic technology [61–102]. The direct electro-reductive or the integration of electrochemistry with transition-metal catalysis has been developed as an effective strategy to realize the carboxylation reactions [103–129], including those of activated C(sp³)–Cl bonds in benzyl chlorides and allyl chlorides (Scheme 1B) [130–142]. However, despite recent advances, the electro-reductive carboxylation of unactivated alkyl chlorides remains challenging. In addition to the high bond dissociation energy (~350 kJ/mol) of the C(sp³)–Cl bonds, many side reactions, such as radical involved dehydrochlorination [143], radical-radical coupling, and reductive protonation [144–145], might occur to result in low selectivity. To the best of our knowledge, there is only one isolated example for carboxylation of unactivated C–Cl bonds to give 25% yield of carboxylation products *via* successive single-electron

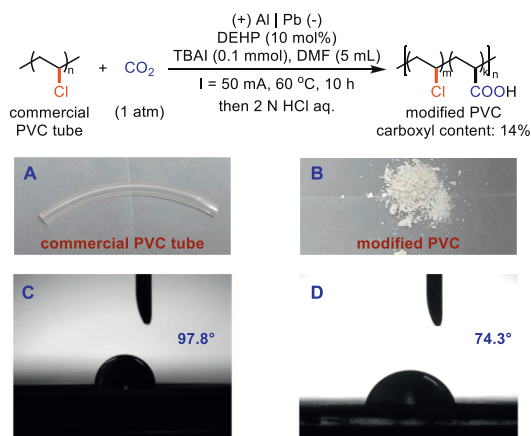
* Corresponding authors.

E-mail addresses: liao11@cqu.edu.cn (L.-L. Liao), dgyu@scu.edu.cn (D.-G. Yu).

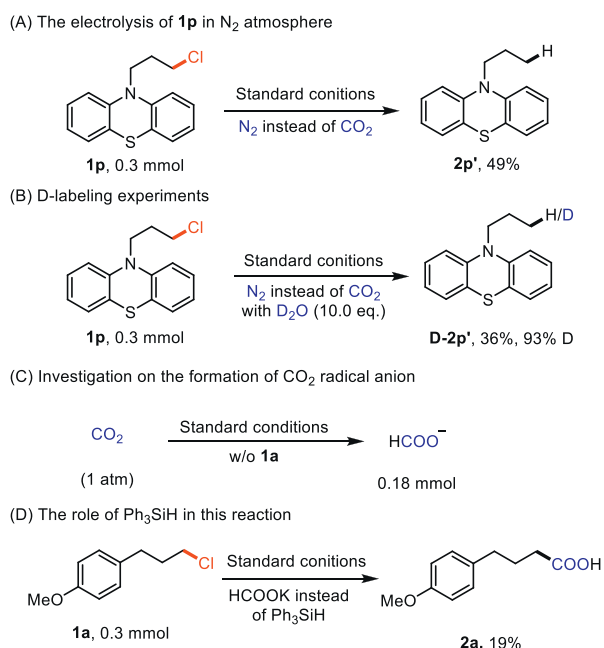
¹ These authors have contributed equally to this work.



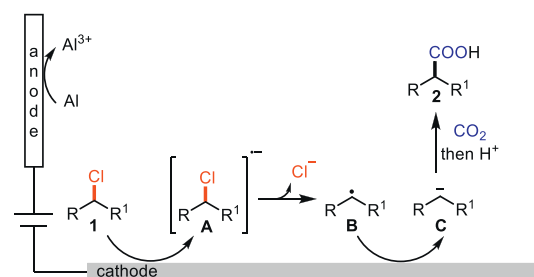
Scheme 2. Substrate scope of unactivated alkyl chlorides. The same reaction conditions as Table 1, entry 1. Isolated yields.



Scheme 3. Carboxylation of PVC. (A) The photo of commercial PVC tube. (B) The photo of modified PVC. (C) Contact angle test image of commercial PVC tube. (D) Contact angle test image of modified PVC.



Scheme 4. Mechanistic investigation.



Scheme 5. Proposed reaction mechanism.

tion product **d-2p'** in 36% isolated yield with 93% deuterium incorporation, which suggested that the alkyl anion might exist in this reaction. Additionally, the detection of formate when conducting experiment in the absence of **1a** verified CO_2 radical anion might be generated *via* reduction of CO_2 at the cathode. According to previous work [162], the reaction of hydrosilane, carboxylates, and CO_2 could give a mixture of formate and silanolate. When we conducted the control experiment of **1a** using formate instead of Ph_3SiH , we found the yield of **2a** was decreases drastically to 19%, which indicates that the role of silanes is not to provide formate. Further control experiment validates the role of silanes in our laboratory.

On the basis of the experimental results and previous reports [145], we proposed the following plausible mechanism for this electro-reductive carboxylation (Scheme 5). First, at the cathode, single-electron transfer (SET) reduction of **1** takes place to generate the corresponding radical anion **A**, which further undergoes C-Cl bond cleavage to release chloride anion and the alkyl radical **B**. Further SET reduction of **B** at the cathode forms the alkyl carbanion **C**, which reacts with CO_2 to give the corresponding carboxylate. At this stage, the single-electron reduction of **1** by CO_2 radical anion to give **A** [163] and direct radical coupling of **B** with CO_2 radical anion cannot be excluded.

In conclusion, we have developed an efficient and practical strategy for electrochemical carboxylation of unactivated alkyl chlorides with CO_2 *via* C-Cl bond cleavage. Both primary and secondary alkyl chlorides could undergo selective carboxylation with CO_2 . This method features mild reaction conditions, low electrolyte concentration, broad substrate scope and good functional group tolerance. Notably, this strategy is a promising and feasible method to recycle and reuse waste PVC, which could convert the PVC

into hydrophilic functional products containing carboxylate groups. The preliminary mechanistic studies indicate that alkyl radical and alkyl anion might be involved in this reaction.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Li Li: Writing – original draft, Methodology, Investigation. **Zhi-Xin Yan:** Writing – original draft, Methodology, Investigation. **Chuan-Kun Ran:** Writing – original draft, Methodology, Investigation. **Yi Liu:** Investigation. **Shuo Zhang:** Investigation. **Tian-Yu Gao:** Methodology, Investigation. **Long-Fei Dai:** Methodology. **Li-Li Liao:** Writing – review & editing, Supervision, Conceptualization. **Jian-Heng Ye:** Writing – review & editing, Supervision. **Da-Gang Yu:** Writing – review & editing, Validation, Supervision, Funding acquisition, Conceptualization.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.ccl.2024.110104.

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