Chinese Chemical Letters 34 (2023) 108489

Contents lists available at ScienceDirect

Chinese Chemical Letters

journal homepage: www.elsevier.com/locate/cclet

Probing the effect of nitrate anion in CAN: An additional opportunity to reduce the catalyst loading for aerobic oxidations[☆]

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A R T I C L E   I N F O

Article history:
Received 8 February 2023
Revised 10 April 2023
Accepted 20 April 2023
Available online 23 April 2023

Keywords:
Nitrate
Alkene
Oxidation
C=C bond cleavage
Ceric
Molecular oxygen

A B S T R A C T

Catalyzed by cerium ammonium nitrate (CAN), the oxidative cracking reaction of alkenes occurred to produce carbonyls in good yields under mild conditions. The reaction employed molecular oxygen (O_2) as the safe and clean oxidant. The catalyst dosage was reduced to as low as 0.5 mol%, while no additive was required. Thus, it may afford a generally green synthetic approach for introducing oxygen into organic molecules as well as the biomass degradation and the resource recycling from the C=C bond-containing waste polymers. X-ray photoelectron spectroscopy (XPS) analysis and control experiments demonstrated that the process proceeded *via* a single electron transfer (SET) reaction-initiated free radical reaction mechanism. In the process, both Ce and NO_3^- acted as the oxygen carrier to promote the oxidation reaction. The application of the abundantly existed nitrate in CAN was found to be the key for reducing the catalyst loading.

Oxidative cracking reaction of alkenes can allow syntheszing carbonyl compounds, deprotecting key functional groups, and degrading large molecules, especially for those from the biomass [1–11]. Thus, it is a significant transformation in many fields such as organic synthesis and biomass utilization, and has attracted comprehensive interests from academia and industry. So far, a series of protocols have been developed. For example, oxidative C=C bond cleavage of alkenes to carbonyl compounds was conventionally achieved using stoichiometric oxidants such as $NaIO_4$, $PhI(OAc)_2$, *meta*-chloroperoxybenzoic-acid (*m*-CPBA), or $PhIO/HBF_4$ (Scheme 1, method a) [3–6]. However, these methods may generate large amounts of wastes, with some being highly hazardous to the environments. Then, reactions using greener oxidants such as TBHP or H_2O_2 were developed [7,8], but these methods still have their own limitations for requiring high loadings of transition-metal catalysts/ligands, among which some are expensive and toxic. Classical ozonolysis is another frequently employed method (Scheme 1, method b). However, it not only suffers from the unsafe issues, but also suffers from the high cost of the equipments, low ozone generation efficiency associated with the energy waste problem, and the generation of massive wastes during work-up processes [9,10]. Moreover, C=C bond cleavage can also be achieved by enzyme-catalyzed oxidative reactions (Scheme 1, method c), but these methods require rather long reaction times and complicated procedures, which lead to low efficiency of the methods [11].

In recent years, catalytic aerobic oxidations have attracted much attention because the abundant molecular oxygen (O_2) can be used as cleaner oxidant [12–23]. The technique has also been applied in alkene cracking reactions (Scheme 1, method d). Various transition metals were found to be active catalysts, but high pressure of O_2, high loadings of the complex and expensive metal catalysts/ligands were usually mandatory conditions [17,18]. Methods employing transition metal-free catalysts such as azodiisobutyronitrile (AIBN), 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO), *N*-hydroxyphthalimide (NHPI), CBr_4 and ArSH were also reported [19–23]. Due to the easy decomposition features of most of the organocatalysts, the methods usually require high loadings of the catalysts, which may reduce the practicality and prevent further applications in large scale. Therefore, there is still a great demand in the field to develop practical and efficient methods for aerobic oxidative C=C cleavage that can use a low loading of inexpensive and readily accessible catalysts under mild conditions.

In our cases, we have investigated the oxidative cracking reaction of alkenes by using selenium catalysts, but it requires the use of H_2O_2 oxidant or relatively harsh reaction conditions, or suffer from the incompletely converted substrates [24]. Cerium(IV) ammonium nitrate (CAN)-catalyzed aerobic oxidation of alkenes was also reported, but the substrates were majorly styrenes or methylenecyclobutanes, and the use of 5–200 mol% of CAN was required in those works [25,26]. Recently, we unexpectedly found that, the nitrate anion (NO_3^-) in CAN was also a strong catalyst

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[☆] Dedication to Prof. Lixin Dai on the Occasion of His Centenary Birthday.

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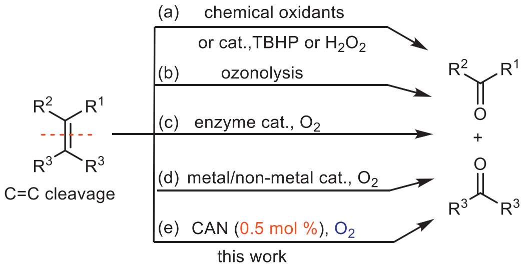
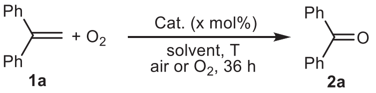
https://doi.org/10.1016/j.cclet.2023.108489



Scheme 1. Methods for oxidative alkene cracking reactions.

Table 1
Condition optimizations[a].



Entry	Solvent	T (°C)	Cat[b]	[O]	**2a** (%)[c]
1[d]	1,4-Dioxane		$FeCl_3$ (5)	Air	<3
2[d]	1,4-Dioxane	80	CuI (5)	Air	<3
3[d]	1,4-Dioxane	80	$CuSO_4$ (5)	Air	45
4	1,4-Dioxane	80	$Cu(OAc)_2$ (5)	Air	60
5[d]	1,4-Dioxane	80	CAN (5)	Air	66
6[d]	H_2O	80	CAN (5)	Air	12
7[d]	DMF	80	CAN (5)	Air	41
8[d]	Cyclohexane	80	CAN (5)	Air	14
9[d]	1,4-Dioxane	70	CAN (5)	Air	58
10[d]	1,4-Dioxane	90	CAN (5)	Air	53
11[d]	1,4-Dioxane	80	CAN (1)	Air	68
12[d]	1,4-Dioxane	80	CAN (0.5)	Air	74
13[d]	1,4-Dioxane	80	CAN (0.4)	Air	73
14[d]	1,4-Dioxane	80	CAN (0.3)	Air	68
15[e]	1,4-Dioxane	80	CAN (0.5)	O_2	93

[a] 0.5 mmol of substrate **1** and 2 mL of solvent were employed; the reactions were heated with bubbled air or O_2 (0.88 cm^3/s).

[b] Molar ratio of catalyst *vs.* **1a** inside the brackets (mol%); without special instruction, metal salts used in the whole text were the ones containing regular amount of crystal water.

[c] Isolated yield of **2a** based on **1a**.

[d] Reactions uncompleted.

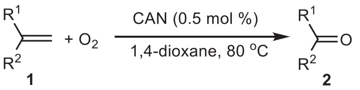
[e] Reaction finished within 24 h.

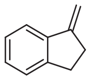
and it could even catalyze the oxidative C=C cracking reaction, so that the CAN catalyst loading could be reduced to be as low as 0.5 mol% (Scheme 1, method e). Herein, we wish to report our findings.

1,1-Diphenyl ethene (**1a**) was chosen as the model substrate and $FeCl_3$ was initially used as the metal catalyst. However, almost no reaction occurred when heating **1a** and $FeCl_3$ in 1,4-dioxane with air (Table 1, entry 1). CuI was also ineffective for the reaction (Table 1, entry 2), but by using $CuSO_4$ or $Cu(OAc)_2$ as catalyst, the desired reaction occurred to produce ketone **2a** in 45%–60% yields (Table 1, entries 3 and 4). CAN was then found to be an even better catalyst, affording **2a** in enhanced yield (Table 1, entry 5). Besides 1,4-dioxane, other solvents, such as water, DMF and cyclohexane were tested, but all of them led to decreased **2a** yield (Table 1, entries 6–8). Reducing or enhancing the reaction temperature both led to decreased product yields (Table 1, entries 9, 10 *vs.* 5). Using less catalyst did not reduce the product yield and with 0.5 mol% of CAN, the reaction could produce **2a** in the highest yield (Table 1, entries 12 *vs.* 5, 11, 13, 14). In the above reactions, substrate **1a** was not completely converted and could be observed in thin layer chromatography. Thus, pure O_2 was then employed as a stronger oxidant instead of air. We were very glad to find that, the reaction was accelerated and could complete within 24 h to produce **2a** in 93% yield (Table 1, entry 15).

Substrate scope of the reaction was then examined under the optimized conditions in Table 1, entry 15. Results in Table 2 showed that the method could be widely applied for the oxidative cracking reactions of a variety of 1,1-disubstituted ethenes. Both electron-enriched and -deficient 1,1-disubstituted ethenes such as **1a**–**1m** could be smoothly oxidized to produce ketones under mild conditions (Table 2, entries 1–13). Introducing an electron-donating group (EDG) into the substrate obviously reduced its activity for the reaction, giving ketone products in decreased yields (Table 2, entries 3, 4 *vs.* 1, 2; 7, 8 *vs.* 5, 6). In cases of the alkenes **1d** and **1h** bearing MeO- as the strong EDG, the reactions were retarded and extended reaction time (48 h) was required (Table 2, entries 4 and 8). Bulky substituents in substrate could also slow down the reaction and only 44%–76% of **1i**–**1k** were converted after 48 h of reactions, but since the unconverted starting material could be recovered, the isolated yields of **2i**–**2k** based on converted substrates were still good (73%–81%, Table 2, entries 9–11). The method was also fit for the exocyclic C=C system in 1-methylene-2,3-dihydro-1*H*-indene **1l**, giving 2,3-dihydro-1*H*-inden-1-one **2l** in 76% yield (Table 2, entry 12). It also showed some degree of tolerances for the strained rings in substrate and the 16 h reaction of (1-cyclopropylvinyl)benzene **1m** could produce the desired cyclopropyl-contained ketone **2m** in 65% yield (Table 2, entry 13).

Table 2
CAN-catalyzed oxidation of 1,1-disubstituted ethenes[a].



Entry	1	R^1, R^2	t (h)	**2** (%)[b]
1	**1a**	bis-Ph	24	**2a**: 93
2	**1b**	bis-4-ClC_6H_4	24	**2b**: 96
3	**1c**	bis-4-MeC_6H_4	24	**2c**: 80
4	**1d**	bis-4-$MeOC_6H_4$	48	**2d**: 72
5	**1e**	Ph, Me	24	**2e**: 85
6	**1f**	4-ClC_6H_4, Me	24	**2f**: 86
7	**1g**	4-MeC_6H_4, Me	24	**2g**: 73
8[c]	**1h**	4-$MeOC_6H_4$, Me	48	**2h**: 91 (74)
9[c]	**1i**	Ph, c-C_6H_{11}	48	**2i**: 73 (76)
10[c]	**1j**	1-$C_{10}H_7$, Ph	48	**2j**: 81 (64)
11[c]	**1k**	1-$C_{10}H_7$, Me	48	**2k**: 79 (44)
12	**1l**		24	**2l**: 76
13[d]	**1m**	Ph, c-C_3H_5	16	**2m**: 65

[a] The mixture of **1** (0.5 mmol) and the catalyst CAN (0.5 mol%) in 1,4-dioxane (2 mL) was heated at 80 °C with bubbled pure O_2 (0.88 cm^3/s).

[b] Isolated yield of **2** based on converted alkenes **1** outside the parenthesis; conversion ratio of alkenes **1** inside the parenthesis (100% conversion ratio as default).

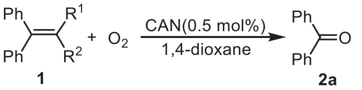
[c] Reactions uncompleted.

[d] Using air as oxidant.

The reactions of tri- and tetra-substituted ethenes were also tested (Table 3). Introducing a methyl reduced the reactivity of the substrate, and the reaction of **1n** led to **2a** in decreased yield (Table 3, entry 1 *vs.* Table 2, entry 1). By enhancing the reaction temperature and extending the reaction time, the reaction of tri-substituted ethene could be improved. For example, heating **1n** at 100 °C in O_2 for 48 h led to **2a** in 80% yield (Table 3, entries 2 *vs.* 1). Besides, the ethyl- (**1o**), *n*–butyl (**1p**) or even phenyl substituted substrates (**1q**) could lead to **2a** in good yields after a 48 h reaction at 100 °C (Table 3, entries 3–5). The reactions of **1r-t** produced **2a** in 68%–75% yields and the electron-enriched substrate **1t** was obviously less reactive and was not completely converted (Table 3, entries 8 *vs.* 6 and 7). 1,1,2,2-Tetraphenylethene **1u** showed poor reactivity for the reaction, affording very low substrate conversion ratio (Table 3, entry 9).

Table 3

CAN-catalyzed oxidation of tri- and tetra-substituted ethenes[a].



Entry	**1**	R^1	R^2	*T* (°C)	*t* (h)	**2a** (%)[b]
1[c]	**1n**	Me	H	80	24	81 (82)
2	**1n**	Me	H	100	48	80
3	**1o**	Et	H	100	48	78
4	**1p**	*n*-Bu	H	100	48	70
5	**1q**	Ph	H	100	48	72
6	**1r**	4-ClC$_6$H$_4$	H	100	48	75
7	**1s**	4-MeC$_6$H$_4$	H	100	48	68
8[c]	**1t**	4-MeOC$_6$H$_4$	H	100	48	70 (82)
9[c]	**1u**	Ph	Ph	100	48	84 (15)

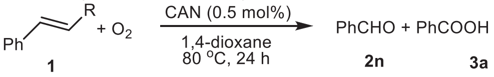
[a] The mixture of **1** (0.5 mmol) and the catalyst CAN (0.5 mol%) in 1,4-dioxane (2 mL) was heated at 80–100 °C with bubbled pure O_2 (0.88 cm^3/s).

[b] Isolated yield of **2a** based on converted alkenes **1** outside the parenthesis; conversion ratio of alkenes **1** inside the parenthesis (100% conversion ratio as default).

[c] Reactions uncompleted.

Table 4

CAN-catalyzed oxidation of styrene and its derivatives[a].



Entry	**1**	R	**2n** (%)[b]	**3a** (%)[b]
1	**1v**	H	52	40
2	**1w**	Me	56	31
3[c]	**1x**	*n*-Bu	58 (73)	37 (73)
4[c]	**1y**	Ph	70 (24)	23 (24)
5[c]	**1z**	CO$_2$Et	No reaction	

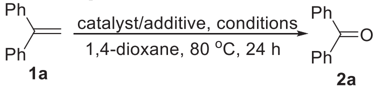
[a] The mixture of **1** (0.5 mmol) and the catalyst CAN (0.5 mol%) in 1,4-dioxane (2 mL) was heated with bubbled pure O_2 (0.88 cm^3/s).

[b] Isolated yield of **2n** or **3a** based on converted alkenes **1** outside the parenthesis; conversion ratio of alkenes **1** inside the parenthesis (100% conversion ratio as default).

[c] Reactions uncompleted.

Table 5

Control experiments[a].



Entry	Catalyst/Additive[b]	Conditions[b]	**3a** (%)[c]
1[d]	CAN (0.5)	N_2	Trace
2[d]	Ce(SO$_4$)$_2$ (0.5)	O_2	70 (69)
3[d]	Ce(SO$_4$)$_2$ (2)	O_2	72 (69)
4	Ce(SO$_4$)$_2$ (0.5), KNO$_3$ (3)	O_2	88
5[d]	Ce(NO$_3$)$_3$ (0.5)	O_2	90 (95)
6	Ce(NO$_3$)$_3$ (0.5), KNO$_3$ (1.5)	O_2	94
7	Ce(NO$_3$)$_3$ (1)	O_2	90
8[d]	Fe(NO$_3$)$_3$ (0.5)	O_2	85 (95)
9	Fe(NO$_3$)$_3$ (0.5), KNO$_3$ (1.5)	O_2	86
10[d]	Cu(NO$_3$)$_2$ (0.5)	O_2	90 (96)
11	Cu(NO$_3$)$_2$ (0.5), KNO$_3$ (2)	O_2	90
12[d]	Mn(NO$_3$)$_2$ (0.5)	O_2	87 (69)
13[d]	Mn(NO$_3$)$_2$ (0.5), KNO$_3$ (2)	O_2	98 (86)
14[d]	KNO$_3$ (3)	O_2	Trace
15	HNO$_3$ (3)	O_2	75
16	KNO$_3$ (3), H$_2$SO$_4$ (1.5)	O_2	80 (60)
17	Ce(SO$_4$)$_2$ (0.5), HNO$_3$ (3)	O_2	89 (93)
18[d]	CAN (0.5), TEMPO (100) or HQ (100)	O_2	Trace
19[e]	CAN (0.5)	O_2	91

[a] **1** (0.5 mmol) and 1,4-dioxane (2 mL) were employed.

[b] Molar ratio of catalyst/additive *vs.* **1a** inside the brackets (mol%).

[c] Isolated yield of **3a** based on converted alkenes **1** outside the parenthesis; conversion ratio of alkenes **1** inside the parenthesis (100% conversion ratio as default).

[d] Reactions uncompleted.

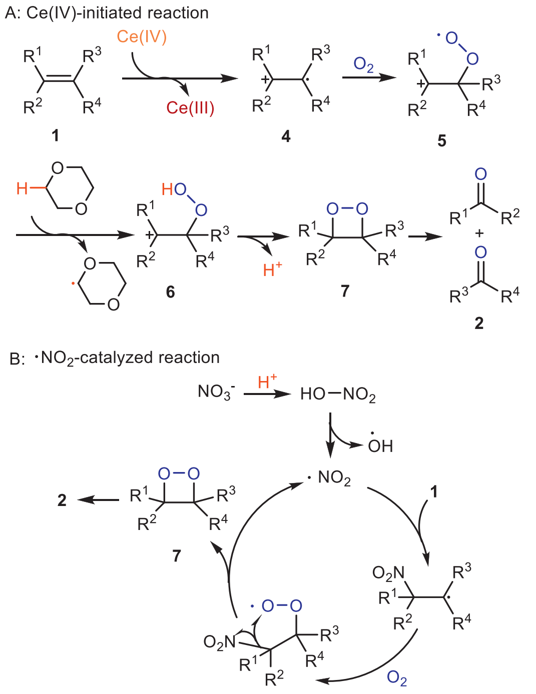
[e] **1** (5 mmol) and 1,4-dioxane (20 mL) were employed.

The reaction of styrene produced benzaldehyde (**2n**) in 52% yield, while benzoic acid (**3a**) was also generated in 40% yield as the unavoidable deep oxidation by-product of **2n** (Table 4, entry 1). After introducing a methyl on the terminal carbon of styrene, the substrate reactivity reduced and produced **2n** in 56% yield, while the by-product **3a** yield decreased (Table 4, entry 2). For the reactions of **1x** and **1y** bearing larger alkyl or aryl group, the substrate was not completely converted (Table 4, entries 3 and 4). No reaction occurred when electron deficient substrate **1z** was employed (Table 4, entry 5).

Moreover, this CAN-catalyzed oxidative C=C bond cleavage method could be used for the purpose of polyene pollutant degradation (Fig. S1 in Supporting information). The reaction of β-carotene was chosen as an example to illustrate this idea for its intuitive reaction phenomenon. Heating β-carotene in the presence of 0.5 mol% of CAN with O_2 flow, the color of the reaction liquid faded away gradually (Fig. S1a), while the absorption peak of the samples in UV–vis spectra shifted to the shortwave field (Fig. S1b). These results demonstrated that the C=C bonds in β-carotene were successfully cut off in this CAN-catalyzed reaction.

Mechanisms were our next concern and a series of control reactions were performed to get the mechanistic insights. The control reaction under N_2 was performed, but only traces of **3a** were obtained, while most of the substrate was unconverted, indicating that O_2 was the crucial oxidant for the reaction (Table 5, entry 1). Using Ce(SO$_4$)$_2$ as catalyst resulted in decreased product yield (Table 5, entry 2), which was hardly enhanced even with elevated Ce(SO$_4$)$_2$ amount (Table 5, entry 3). Interestingly, after a supplementary of 3 mol% of KNO$_3$ that contained the same amount of nitrate as in CAN, the reaction occurred smoothly and produced **3a** in excellent yield (Table 5, entry 4). The reaction with 0.5 mol% of Ce(NO$_3$)$_3$ catalyst produced **2a** in 90% yield with 95% substrate conversion (Table 5, entry 5). It could be further improved by adding KNO$_3$ or using increased amount of Ce(NO$_3$)$_3$ (Table 5, entries 6, 7). In the later reaction (Table 5, entry 7), the increased product yield probably attributed to the enhanced NO_3^- amount other than Ce^{4+}, in accordance with the results in Table 5, entries 2–4. Besides, the nitrate salts of Fe, Cu and Mn, which were all conventional SET catalysts, could catalyze this reaction as well (Table 5, entries 8–13), and the product yields were enhanced after adding KNO$_3$ as an additional nitrate source (Table 5, entry 8 *vs.* 9; 10 *vs.* 11; 12 *vs.* 13). KNO$_3$ alone could not catalyze the reaction (Table 5, entry 14). By using 3 mol% of HNO$_3$ as catalyst, the reaction could produce **2a** in 75% yield (Table 5, entry 15) and it could also be catalyzed by the KNO$_3$/H$_2$SO$_4$ system (Table 5, entry 16). The HNO$_3$-catalyzed reaction could be improved by adding Ce(SO$_4$)$_2$ (Table 5, entry 17). The reaction could be restrained by free radical scavenger such as TEMPO or HQ (hydroquinone), indicating that free radicals were involved during the process (Table 5, entries 18) [27–29]. In addition, gram-scale reaction was also conducted, and it was found that the yield of this reaction did not decrease significantly after amplification (Table 5, entry 19).

X-ray photoelectron spectroscopy (XPS) analysis of the reaction mixtures showed that Ce(IV) in CAN was converted into Ce(III) after reaction (Fig. S2a in Supporting information). Thus, it could be deduced that the reaction was catalysed by transition metals with variable valences *via* the single electron transfer (SET) mechanisms [30,31]. Results in Table 5 indicated that nitrate could also promote the activity of the catalytic system, and this could well explain why the catalytic activity of CAN was so high that it could be employed at very low loading (0.5 mol%). In order to explore whether there is hydroxyl radical formation in the reaction process, we designed a verification experiment by taking advantage of the property that



Scheme 2. Possible mechanism of the reaction.

salicylic acid can trap hydroxyl radical, and the results obtained were shown in Fig. S2b (Supporting information). After adding the reaction liquid into the salicylic acid sample, a strong adsorption at around 490–650 nm (540 nm at maximum) was observed in the UV–vis spectra (red curve *vs.* black curve), which was the characteristic peak of the adducts of hydroxyl radical with salicylic acid (*i.e.* 2,3-dihydroxybenzoic acid and 2,5-dihydroxybenzoic acid) reflecting the existence of hydroxyl radical [32].

On the basis of the above experimental results as well as the reported literatures [26,33–38], a plausible mechanism was supposed (Scheme 2). The alkenes **1** were initially oxidized by Ce(IV) to give the active free radical cation **4** [33]. As being confirmed by the XPS spectrum illustrated in Fig. 2, Ce(IV) was reduced into Ce(III) after reaction. In the catalysis cycle, the Ce(III) species could be oxidized by O_2 to regenerate Ce(IV). Oxidation of **4** with O_2 led to the intermediate **5** [34], which grabbed a proton from the 1,4-dioxane solvent and produced the intermediate **6** [35]. Cyclization of **6** afforded the 1,2-dioxetane **7** [26] and released a proton. Finally, decomposition of **7** produced carbonyls as the reaction product. Moreover, the proton generated in the previous step could enhance the acidity of the reaction liquid to facilitate the use of nitrate as an additional catalyst in the HNO_3 form, which could oxidize alkenes and the generated low valent nitrogen speceis could be reoxidized by O_2 to produce HNO_3 and restart the HNO_3 catalysis cycle B [36–38]. The proton releasing during the transition metal catalysis processes was considered to be the key for activating nitrate anion, *i.e.* convert NO_3^- into its highly active and oxidative HNO_3 form. This is why KNO_3 alone can not catalyze the reaction (Table 5, entrey 14). Therefore, it can be deduced that, CAN is an efficient low-loading (0.5 mol%) catalyst for the oxidative cracking reaction of alkenes because both Ce and nitrate in it are active catalytic species.

In conclusion, we have developed a novel method to split the C=C bond in alkenes by using molecular oxygen as the cheap and clean oxidant. The reaction was catalyzed by low-loading CAN (0.5 mol%) free of any additives. Mechanism studies demonstrated that the ultrahigh catalytic activity of CAN attributed to the fact that both Ce and nitrate may participate the reaction as catalyst *via* the coupled Ce(IV)-Ce(III) circle and nitroxide circle respectively. The catalyitic activity of anions such as NO_3^- is a novel finding. It is surprising that the catalytic activity of NO_3^- is so strong that it can even catalyze the oxidative alkene cracking reaction, which consumes a lot of reaction energy for the high C=C bond dissociation energy (*ca.* 609 kJ/mol). This work not only provides an efficient method for carbonyl synthesis, but also leads to a new pathway for biomass degradation, as well as the recycling of the C=C bond-containing waste polymer. It might also inspire new ideas for catalytic system design: the significances of anions in the system should be noticed. Further investigations on the design and application of Ce catalysts are ongoing in our laboratory.

Declaration of competing interest

The authors declare no conflict of interest.

Acknowledgments

This work was financially supported by Priority Academic Program Development of Jiangsu Higher Education Institutions and Postgraduate Research & Practice Innovation Program of Jiangsu Province (Yangzhou University) (No. KYCX21_3205).

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2023.108489.

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