Comparison of N-nitrosodiethylamine degradation in water by UV irradiation and UV/O3: Efficiency, product and mechanism

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N-Nitrosodiethylamine (NDEA) is a member of nitrosamines, which is strong carcinogenic. In order to explore an effective treatment method for NDEA removal from water, sole UV irradiation and UV/O3 were carried out in this study. The removal efficiency, degradation products and pathways were compared between those two processes. Results showed that NDEA removal efficiency achieved 99% within 15 min by both UV and UV/O3 Degradation reaction well followed pseudo-first-order kinetics. Water pH had different effect on NDEA degradation in those two processes. Acidic and neutral conditions were good for NDEA degradation by sole UV irradiation. However, NDEA underwent rapid degradation under various pH conditions in the UV/O3 process. Though the ozone introduction in the UV/O3 process had little effect on NDEA degradation efficiency, it had significant effect on its degradation products and pathways. Methylamine, dimethylamine, ethylamine and diethylamine were observed as aliphatic amine products of NDEA degradation in both two processes. They were assumed to arise due to N–N bond fission under UV irradiation, or due to the reaction of NDEA and hydroxyl radicals in the UV/O3 process.

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1. Introduction

Nitrosamines are a class of mutagenic, teratogenic and carcinogenic chemicals in the environment as by-products of various manufacturing, agricultural and natural processes [1,2]. There are nearly 300 types of nitrosamines, of which 80–90% are carcinogenic [3]. The carcinogenicity of nitrosamines interrelates with their chemical structures [3]. The carcinogenic risk of the nitrosamine increases with the decrease of aliphatic chain length. As a special case, the carcinogenicity of N-nitrosodiethylamine (NDEA) is greater than that of N-nitrosodimethylamine (NDMA). According to previous researches, the maximum admissible concentration of NDMA and NDEA in drinking water is 7 ng L\(^{-1}\) and 2 ng L\(^{-1}\), respectively [3]. The California Department of Public Health has established a notification level of 10 ng L\(^{-1}\) for both NDMA and NDEA [4].

Previous studies about N-nitrosamines mainly focused on their occurrence in food, consumable and air pollution [5]. Since NDMA was observed to be an important disinfection by-product in chlorination or chloramination, N-nitrosamines in drinking water and wastewater were of great concern [6–7]. Recently, NDMA has been selected as a typical representative to carry out related researches. Its formation mechanisms [8–15], detection methods [16–20] and treatment methods [21–26] have been extensively investigated. However, there were a few studies on NDEA in drinking water and wastewater.

One study utilized Fenton reagent for decomposition of NDMA and NDEA in water [27]. Their results suggested the interaction of the N-nitrosamines with Fenton reagent, and only focused on the formation of nitric oxide. The absolute rate constants and degradation efficiencies for hydroxyl radical reaction with NDEA were evaluated by Landsman et al. [28] using the combination of electron-pulse radiolysis/absorption spectroscopy and steady-state radiolysis/GC–MS. Plumlee et al. [29] removed NDMA, NDEA and other nitrosamines by using reverse osmosis and UV treatment. They also determined the rates of direct and indirect photolysis, and quantum yields of those pollutants under simulated natural sunlight conditions [30]. However, NDMA was selected for further investigation to assess the photodegradation ability and identify the photodegradation products.

NDEA is one of the most important environmental carcinogens, which is known as the hepatic carcinogenic agents [31]. Moreover, it is also suspected to be involved in gastrointestinal tumors in humans [32]. NDEA is occurring in a variety of products that would...
result in human exposure, such as tobacco, meat, whiskey and so on [33]. It is inevitable to be discharge with municipal wastewaters, resulting in water pollution and elevated healthy risk. In recent years, the occurrence of NDEA in source water and drinking water treatment plant has been reported. NDEA present in most primary influent (21 plants) samples at concentrations 25 ng L\(^{-1}\) [34]. If high chlorine dosage applied in disinfection, the concentration of NDEA was much higher [35]. Accordingly, elevated concentrations of NDEA in natural aquatic environment should be of great concerns because of its especial toxicological characteristic and widespread occurrence.

In this study, both sole UV irradiation and UV/O\(_3\) were used to degrade NDEA in water. The objectives of this study were to assess sole UV irradiation and UV/O\(_3\) as alternative treatment technologies for NDEA degradation, and compare the removal efficiency, degradation products and pathways between those two processes. This study would help to explore a better degradation method for NDEA removal in water.

2. Materials and methods

2.1. Reagents

NDEA, methylamine (MA), dimethylamine (DMA), ethylamine (EA), diethylamine (DEA), N-methylmethylamine, benzensulfonyl chloride and 9-fluorenylmethylocchloroformate (FMOCS) were all purchased from Sigma–Aldrich, Fluka, USA, with purity higher than 98%. Dichloromethane and acetonitrile were obtained from Dikma, USA. Other chemicals (NaNO\(_2\), NaNO\(_3\), NaHCO\(_3\), anhydrous Na\(_2\)SO\(_4\), diethylamine (DEA), and N-(EA), diethylamine (DEA), and N-(EA), diethlylamine, benzensulfonyl chloride (95%), dichloromethane and acetonitrile were all purchased from Sigma–Aldrich, Fluka, USA, with purity higher than 98%. Dichloromethane and acetonitrile were obtained from Dikma, USA. Other chemicals (NaNO\(_2\), NaNO\(_3\), NaHCO\(_3\), anhydrous Na\(_2\)SO\(_4\), ammonia, borate, NaOH and H\(_2\)SO\(_4\)) used in the experiments were of analytical grade and were used without further purification. All stock solutions were prepared in Milli-Q water.

All glassware used in the experiments was soaked in H\(_2\)SO\(_4\)–K\(_2\)Cr\(_2\)O\(_7\) solution over a night, and then washed several times in both tap water and distilled water.

2.2. Experimental procedure

The NDEA degradation experiments using sole UV irradiation and UV/O\(_3\) were performed in a cylindrical quartz photoreactor (Fig. 1). The valid sample bulk of the reactor was 700 mL. NDEA solution was exposed to UV irradiation using a low-pressure Hg lamp (8 W, emission at 253.7 nm, Tianjin Xjing Co. China), of which light quantum flow rate was 3.3 × 10\(^{-6}\) Einstein L\(^{-1}\) s\(^{-1}\). In the UV/O\(_3\) process, ozone was produced by a lab ozonizer (DHX–SS–1G, Harbin Jiuju Electrochemistry Engineering Ltd. China), which had a maximum ozone production at 9 g h\(^{-1}\), and used pure oxygen as feed gas. Dissolved ozone concentration in the reactor was controlled by varying the oxygen flux, the voltage of the ozone production and the time at which ozone was introduced into the reactor. Ozone was introduced into the reactor in a single addition before the start of the experiment. Then, the stock solution of NDEA was dropped into the reactor immediately, and the low-pressure Hg lamp was turned on at the same time simultaneously. Water pH was adjusted by H\(_2\)SO\(_4\) and NaOH solution (0.1 mol L\(^{-1}\)). Samples were collected at predetermined time intervals, and were analyzed quickly after sampling. The ozone oxidation reaction was quenched with Na\(_2\)SO\(_3\) solution (0.1 mol L\(^{-1}\)). Each experiment was carried out for three times, and average value was shown in this study.

2.3. Analysis

NDEA was analyzed by high performance liquid chromatography (HPLC, LC–10A, Shimadzu, Japan) with UV detection at 230 nm. The separation was performed with a Venusil-MP–C\(_18\) column (150 mm × 4.6 mm, 5 μm, Agela Technologies Inc.) using isocratic elution of 95% water and 5% methanol at a flow rate of 1.0 mL min\(^{-1}\). The detection limit was approximately 150 ng L\(^{-1}\), and the relative standard deviation was approximately 1.1%.

Quantitative analysis of EA and DEA were carried out followed a modified derivatization method described by Sacher et al. [36]. First, samples were derived with benzensulfonyl chloride, and then were analyzed by gas chromatography–mass spectrometry (GC–MS, Agilent 6890/5973N, USA), which was equipped with a column (DB–1MS, 30 m × 0.25 mm i.d. × 0.25 μm), J & W Scientific, Folsom, CA). Sample (20 mL) was diluted to 200 mL in a 250 mL round-bottomed flask. Both NaOH solution (8 mL, 10 mol L\(^{-1}\)) and benzensulfonyl chloride (2 mL) were added into the flask. The flask was sealed, and then was stirred for 10 min at room temperature. Afterwards, another 10 mL NaOH solution (10 mol L\(^{-1}\)) was added, and the mixture was stirred for another 10 min at 80 °C to hydrolyze the excess of derivatization reagent. Subsequently, the mixture solution was cooled down with ice water, and was acidified to pH 5.5 using 18.5% HCl solution. The mixture was extracted twice with 10 mL dichloromethane. The organic phase was combined, washed with 15 mL NaHCO\(_3\) solution (0.05 mol L\(^{-1}\)), and was dried with anhydrous Na\(_2\)SO\(_4\) for analysis.

The samples were analyzed by GC–MS. The following oven temperature program was used: an initial oven temperature was 80 °C (held for 5 min), increased to 150 °C (held for 10 min) at 10 °C min\(^{-1}\), and then increased to 300 °C (held for 1 min) at 50 °C min\(^{-1}\). The splitless injection was used with an injection port temperature of 290 °C, and the volume of injection was 1 μL. High purity He gas (purity > 99.999%) was used as carrier gas, and the column flow rate was 0.5 mL min\(^{-1}\). The ion source of mass spectra was operated in the electronimpact mode (EI mode, electron energy 70 eV, 230 °C). Full-scan mass (m/z 40–500) were recorded for identification of analytes at the high concentration. Identification of trace chemicals was achieved by using selected ions scan mode (SIM mode). As the qualitative ions, m/z 77 and m/z 141 were selected for both DEA derivative and EA derivative. The quantitative ion was m/z 198 for DEA derivative and m/z 170 for EA derivative, respectively. The detection limit of EA and DEA was approximately 100 nmol L\(^{-1}\) and 70 nmol L\(^{-1}\), respectively. The relative standard deviation for EA and DEA was approximately 5.4% and 3.1%, respectively.

Qualitative analysis of aliphatic amine products generated in photodegradation was carried out by a modified derivatization method described by Lopez et al. [37]. Samples were derived with 9-fluorenylmethylocchloroformate (FMOCS), and were analyzed by HPLC (LC–10A, Shimadzu, Japan) with fluorescence detector. Samples (1 mL) were mixed with 0.5 mL borate buffer (pH 8.5) and 0.5 mL FMOCS solution (0.02 mol L\(^{-1}\)), shaking for 2.5 min reaction. After that, 0.5 mL ammonia (25%) was added. The mixture reacted for 2.5 min. The solution, consisting of 50% acetonitrile and 50% water
(2.5 mL), was finally added before the solvent injected into HPLC. Fluorescence detection was performed at an excitation wavelength of 265 nm, monitoring the emission wavelength of 310 nm with a Venusil M-Pc18 column (150 mm × 4.6 mm, 5 μm, Agela Technologies Inc.), using isocratic elution of 65% acetonitrile and 35% water at a flow rate of 0.8 mL min⁻¹.

The anions NO₂⁻ and NO₃⁻ were analyzed by ion chromatography (ICS-3000, Dionex, USA) with an Ionpac AS-HC (Dionex, USA) ion-exchange column, using KOH solution (30.0 mmol L⁻¹) as the eluent at a flow rate of 1.2 mL min⁻¹. The detection limit of NO₂⁻ and NO₃⁻ was approximately 1.0 μmol L⁻¹ and 5.0 μmol L⁻¹, respectively. The relative standard deviation for NO₂⁻ and NO₃⁻ was approximately 2.5% and 4.3%, respectively.

The aqueous ozone concentration was measured with the indigo method [38]. The incident irradiance value (μW cm⁻²) was measured in separated experiments by a ferrioxalate actinometry experiment as described by Murov et al. [39].

3. Results and discussion

3.1. NDEA degradation by UV irradiation and UV/O₃

The results of NDEA removal by different treatment methods, i.e. sole ozonation, sole UV irradiation and UV/O₃, are shown in Fig. 2. By sole ozonation, NDEA removal efficiency only achieved 10%, suggesting this method was not effective for NDEA removal in water. However, both sole UV irradiation and UV/O₃ were effective for NDEA degradation. NDEA underwent a rapid direct photolysis by sole UV irradiation because of its photolabile property. Its concentrations decreased sharply within the first 5 min, and the removal efficiency achieved over 99% at longer times. Compared with sole UV irradiation, the trends of NDEA degradation in the two different processes. In sole UV irradiation, NDEA was almost completely removed within 15 min. NDEA initial concentrations increasing, and they all were higher than that of the highest oxidation-reduction potential, NO₃⁻ and NO₂⁻ reacted with H₃O⁺ resulting in faster degradation rate in the UV/O₃ process.

The reason was complicated due to the combination of UV and ozone. Ozone may attack on the pollutants via two different reaction pathways: (1) the direct ozonation by the ozone molecule, which occurred at low pH condition, and (2) the radical oxidation by the highly oxidative free radicals, e.g. hydroxyl radicals, which resulted in faster degradation rate in the UV/O₃ process.

3.2. Effect of initial concentration on NDEA degradation by UV and UV/O₃

The NDEA degradation experiments using UV and UV/O₃ were carried out under various NDEA initial concentrations (shown in Fig. 3). Both sole UV irradiation and UV/O₃ can efficiently remove NDEA from water. In the sole UV irradiation, NDEA was almost completely removed within 15 min. NDEA initial concentrations had little effect on its degradation under investigated initial concentrations, i.e. 0.01–0.10 mmol L⁻¹. Though the rate constant obviously decreased at initial concentration of 0.20 mmol L⁻¹, the final removal efficiency was still greater than 99%. Compared with sole UV irradiation, UV/O₃ also presented good trends for NDEA degradation in all cases. The rate constants decreased with the initial concentrations increasing, and they all were higher than that in sole UV irradiation process under each same condition.

Depending on the NDEA initial concentration, a semi-logarithmic plot of the actual concentration versus irradiation time was linear, indicating the degradation reaction of NDEA by sole UV irradiation and UV/O₃ followed pseudo-first-order kinetics. The reaction kinetic parameters are presented in Table 1.

3.3. Effect of water pH on NDEA degradation by UV and UV/O₃

Water pH is one of the most important factors influencing contaminants degradation. Fig. 4 shows the effect of water pH on NDEA degradation in the two different processes. In sole UV irradiation, NDEA was almost completely degraded within 15 min under various pH conditions. The degradation rates sharply decreased with pH increasing. As being amphipathic, nitrosamine exhibited different species for special chemical property under different acid–base pH conditions. They were protonated at low pH, and the protonated species of excited-state nitrosamine were assumed more photolabile than unprotonated species. Accordingly, the rate of NDEA degradation was high in acidic and neutral solutions, but was low in alkaline solution. This was consistent with the previous studies on NDMA photodegradation [40,41].

A quite different result was observed in the UV/O₃ process (Fig. 4). Water pH had little effect on NDEA degradation by UV/O₃. NDEA underwent a rapid degradation under various pH conditions. The reaction was complicated due to the combination of UV and ozone. Ozone may attack on the pollutants via two different reaction pathways: (1) the direct ozonation by the ozone molecule, which occurred at low pH condition, and (2) the radical oxidation by the highly oxidative free radicals, e.g. hydroxyl radicals, which...
were formed by the ozone decomposition at high pH condition [42]. Ozonation was more selective and predominated in acidic solution. However, hydroxyl radical oxidation was less selective and predominated in alkaline solution. When ozone combined with UV irradiation, ozone decomposition was enhanced by UV irradiation, resulting in producing more hydroxyl radicals. Alkaline condition was suitable for ozone decomposition to generate hydroxyl radicals. Accordingly, a more rapid NDEA degradation was observed in UV/O3 process at high water pH. Though ozone decomposition was still observed because of its strong photolability to UV irradiation, ozone decomposition was enhanced by UV irradiation, resulting in producing more hydroxyl radicals. Alkaline condition was still observed because of its strong photolability to UV irradiation. The results suggested that the presence of hydroxyl radicals enhanced NDEA removal effectiveness in UV/O3 at alkaline condition than that in the sole UV irradiation.

3.4. Products of NDEA degradation by UV and UV/O3

During NDEA degradation, two nitrogen atoms of amino and nitroso group were going to form organic amine products and inorganic nitrogen products, respectively. Then some organic amine products can further transform to inorganic nitrogen products. According to previous studies about degradation products of NDMA [43,44], EA, DEA, NO2− and NO3− were proposed as main products of NDEA due to the similar chemical structure. To certify this hypothesis, those four products were analyzed quantitatively in this study.

Fig. 5 shows the concentrations of NDEA degradation products by UV and UV/O3 under various pH conditions. Water pH significantly influenced the yields of products. The yields of DEA and NO2− increased with pH increasing from acidic to neutral solution, but rapidly decreased in alkaline solution in both UV and UV/O3 process. Yields of EA, DEA, NO2− and NO3− produced in UV and UV/O3 processes were quite different under the same reaction condition. The yield of DEA produced in the UV/O3 process was less than that in the UV process under the same pH condition. As compared with sole UV irradiation, the yield of DEA reduced 54%, 52%, 37% and 22% in the UV/O3 process at pH 3.2, 6.0, 7.1 and 10.8, respectively. The NO2− yield also decreased quickly in the UV/O3 process as compared with sole UV irradiation.

It was worth noting that DEA and NO2− can contribute the generation of NDEA [5]. In nitrosation process, NDEA is formed by the reaction of DE and nitrosating reagent, which often exists in the form of NO-X (such as, HNO, N2O3, N2O4, etc.). Although NDEA cannot be formed by simply mixing the solution contained DEA and nitrite, nitrite could transformed in to nitrosating reagent in certain circumstance (such as acidic condition) which leads to NDEA formation. Therefore, the presence of DEA and nitrite might be contributed to NDEA regeneration. In the UV/O3 process, the yield reduced, and NO2− replaced NO3− to be the major inorganic nitrogen product. These indicate that UV/O3 was suitable to inhibit NDEA regeneration in water.

Nitrogen imbalance took place in both the sole UV irradiation and UV/O3 according to Fig. 5. EA and DEA account for only about 30% of the initial NDEA concentration in all cases. This may be due to some other organic nitrogen products that were not determined generated. The losses of inorganic nitrogen were proposed due to the generation of gas products, i.e. N2, N2O, or NO, under some conditions. However, the sum of NO2− and NO3− was by 40% higher than the initial concentration of NDEA in UV/O3 under water pH 7.1. This may be caused by the additional transformation from some organic nitrogen generated to NO2− and NO3−. Based on the nitrogen imbalance, the generation of some other products contained nitrogen in sole UV irradiation and UV/O3 was confirmed. Com-

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**Table 1**

<table>
<thead>
<tr>
<th>Reaction system</th>
<th>Initial concentration (mmol L−1)</th>
<th>Kinetic formula</th>
<th>Reaction rate constant (min−1)</th>
<th>Correlation coefficient</th>
<th>Half-life (min)</th>
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<td>UV irradiation</td>
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<td>0.9984</td>
<td>0.98</td>
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<td>0.05</td>
<td>Ln(C/C0) = 0.1035 − 0.6770t</td>
<td>0.6770</td>
<td>0.9961</td>
<td>1.02</td>
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<tr>
<td></td>
<td>0.10</td>
<td>Ln(C/C0) = 0.0195 − 0.5597t</td>
<td>0.5597</td>
<td>0.9998</td>
<td>1.24</td>
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<td>0.20</td>
<td>Ln(C/C0) = 0.1326 − 0.5446t</td>
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<td>0.9903</td>
<td>1.27</td>
</tr>
<tr>
<td>UV/O3</td>
<td>0.01</td>
<td>Ln(C/C0) = 0.3522 − 1.0303t</td>
<td>1.0303</td>
<td>0.9811</td>
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<td></td>
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<td>0.9970</td>
<td>0.87</td>
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**Fig. 4.** Effect of solution pH on NDEA degradation by the sole UV irradiation and UV/O3. [NDEA]0 = 0.10 mmol L−1, [O3]0 = 6.64 mg L−1, pH 6.0, irradiation 1000 μW cm−2.

**Fig. 5.** Effect of pH on products of NDEA degradation by UV (a) and UV/O3 (b) [NDEA]0 = 0.10 mmol L−1, [O3]0 = 6.64 mg L−1, irradiation 1000 μW cm−2.
pared with sole UV irradiation, nitrogen losses in UV/O3 were more notable, indicating a further degradation.

To identify other aliphatic amine products in both UV and UV/O3, HPLC equipped with fluorescence detector was employed. According to Fig. 6, MA, EA, DMA and DEA were observed as aliphatic amine products of NDEA degradation in both two processes. Nevertheless, another peak with retention time at 14.6 min only emerged in the UV/O3, suggesting that new aliphatic amine product generated. This result also indicated the difference of NDEA degradation pathways between sole UV and UV/O3.

3.5. Pathways of NDEA degradation by UV and UV/O3

N-Nitrosamines were known to undergo photoelimination, photoreduction, photoaddition, and photohydrolysis under weakly acidic conditions when exposed to UV light [41]. Two photolytic pathways were suggested as the primary pathway of the N-nitrosamines according to the previous studies [41,43]. The first was the homolytic cleavage of N–N bonds, which produced the corresponding aminium radical and nitric oxide. The second was the heterolytic cleavage of N–N bonds, which produced parent secondary amines and nitrite [43].

Accordingly, the primary pathway of NDEA degradation by sole UV irradiation was proposed (Fig. 7). The products of NDEA photodegradation were produced because of N–N bond fission. In the sole UV irradiation process, the homolytic cleavage of N–N bonds produced the diethylaminium radical. Two diethylaminium radical could transform to DEA. The intermediate formed from the reaction of diethylaminium radical and nitric oxide underwent acid-catalyzed hydrolysis to generate EA and aldehyde. When the
heterolytic cleavage of N–N bonds took place, DEA was produced because of the nucleophile attack in acidic solution.

The mechanism of NDEA degradation in UV/O₃ process was complicated due to the presence of UV, ozone and hydroxyl radicals. On one hand, NDEA would degrade by UV irradiation. EA, DEA, NO₂⁻ and NO₃⁻ could be produced due to the N–N bond fission as discussed above. On the other hand, NDEA would also react with hydroxyl radicals generated by the combination of UV irradiation and ozone (Fig. 7b). The reaction of hydroxyl radicals and NDEA was proposed to occur by hydrogen atom abstraction from one of its methylene groups to form carbon-centered NDEA radical. Then the intermediate generated from the self-decomposition of the carbon-centered NDEA radical was further degraded to EA, DEA and other amines.

Though the primary pathways of NDEA degradation by UV and UV/O₃ were proposed, the nitrogen imbalance of NDEA was still unclear. Further investigations on NDEA transformation need to be carried out.

4. Conclusions

Both sole UV irradiation and UV/O₃ were effectice methods to degrade NDEA in water. Though introduction of ozone into UV process enhanced the removal efficiency faintly, it had great effect on degradation products and pathways. As the main products, DEA and NO₂⁻ decreased sharply in UV/O₃ as compared with sole UV irradiation. Water pH had significant effect on NDEA degradation in UV process. Both acidic and neutral pH conditions were suitable for the NDEA degradation in the UV process. However, pH has little effect on NDEA degradation by UV/O₃. The products of NDEA degradation were produced because of N–N bond fission under UV irradiation. In addition, the reaction of hydroxyl radicals and NDEA was proposed to be another pathway of NDEA degradation in UV/O₃ process.

The results showed that UV/O₃ process had many advantages on NDEA removal in water compared with sole UV irradiation: (1) it was a fast, effective and feasible method for water treatment; (2) it would not be influenced by solution pH; (3) it was suitable for reducing the yields of DEA and NO₂⁻ and inhibiting the NDEA regeneration.

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