Perspective

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Heterogeneous AOPs from Mineralization Rate to Organic carbon transfer Process: OCTP systems and Emerging Regeneration Indicators

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Abstract: In wastewater treatment, heterogeneous advanced oxidation processes (AOPs) are often evaluated by the degradation rate of pollutants, but this may overlook a problem: pollutants may not be completely mineralized but instead transfer and accumulate on the surface of the catalyst as degradation intermediates. Recently, the organic carbon transfer process (OCTP) proposed by Xing et al. in Nature Water indicates that partial degradation intermediates can mask active sites and lead to catalyst deactivation, but this deactivation is reversible. Based on this, we introduce OCTP as a question of where the carbon of heterogeneous AOPs goes, pointing out that the properties of the oxidant and the choice of the reaction path will determine whether the pollutants in the system move towards deep mineralization or surface accumulation, thereby affecting stability and long-term effectiveness. Based on this logic, we propose the index of catalyst regeneration extent (CRE) to quantify the recoverable effective life after cleaning and advocate designing corresponding strategies according to different situations.

Key words: advanced oxidation processes; organic carbon transfer process; catalyst deactivation; catalyst regeneration

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Introduction

Advanced oxidation processes (AOPs) in water treatment are used to remove refractory organic pollutants. In heterogeneous AOPs, the catalyst is an activator for oxidants and serves as the reaction site: Pollutants are adsorbed on the catalyst and form degradation intermediates. Therefore, the degradation rate in the aqueous phase does not fully show where the pollutant carbon finally goes. It also cannot fully explain why catalyst performance declines. This problem should be noticed when research targets shift from laboratory scale to the development of practical catalytic technologies. Stability, maintainability, secondary waste and safety often determine the feasibility of the technology. The organic carbon transfer process (OCTP) proposed by Xing et al.[1] holds importance: This method transfer the focus of the field from "the degradation rate of pollutants" to "where the organic carbon ultimately goes and its impact". In this Perspective, we clarify our unique contribution in three aspects. Firstly, using OCTP as a framework to track the distribution of carbon provides a mechanistic basis for explaining catalyst deactivation and regeneration in heterogeneous AOPs. Secondly, a practical reporting scheme is proposed to characterize carbon distribution and reversible fouling. Finally, CRE is introduced as a quantitative indicator to evaluate regeneration performance and support a regeneration-oriented testing strategy. In summary, these elements move AOP assessment from short-term kinetics toward maintainability and safety-relevant lifecycle considerations.

OCTP: A New Path for Heterogeneous AOPs

OCTP describes a new path: in the heterogeneous AOPs treatment process, the partial degradation intermediates generated have evolved physical properties such as polarity and solubility, and are preferentially enriched on the catalyst surface rather than mineralized. One of its key contributions is to clearly quantify the carbon-related characterization quantities in the reaction solution and the catalyst: simultaneously determining the COD of the aqueous phase and the COD of the solid phase, the authors transformed "catalyst surface contamination" from a qualitative suspicion into a quantifiable organic carbon index[1]. It is worth noting that in complex industrial wastewater, OCTP has been reported to account for the major proportion of carbon, the source of pollutants related to the catalyst surface, showing that a reduction in COD in the solution can be accompanied by a significant increase in solid-phase COD. As shown in Fig. 1a, initially, phenol is hydroxylated by the $^1O_2$ produced in the reaction system, which helps to

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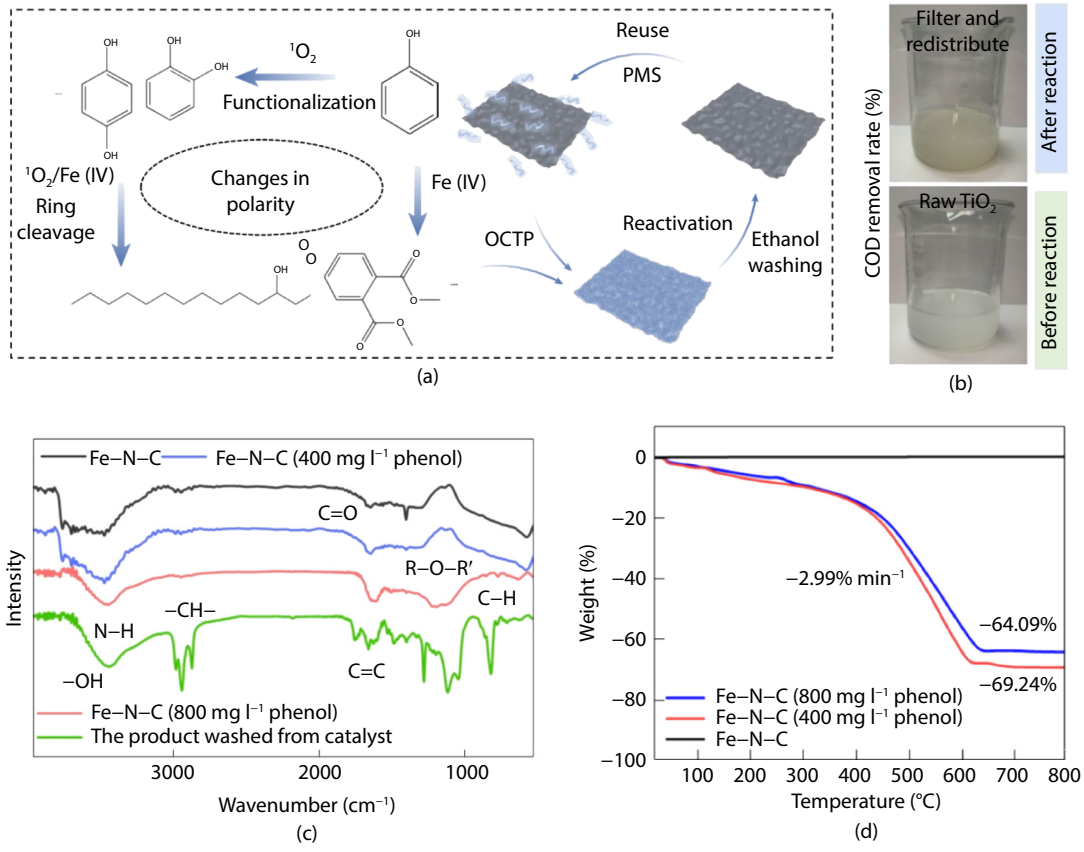

Fig. 1. Evidence and mechanistic illustration of the organic carbon transfer process (OCTP). a) Proposed OCTP mechanism during phenol degradation in the Fe–N–C/PMS system. b) TiO_2 before and after reaction, indicating catalyst-surface accumulation of reaction-derived organic residues. c) FTIR of Fe–N–C before and after the reaction and ethanol washings of Fe–N–C after the reaction. d) TG analysis of Fe–N–C before and after the reaction. For measuring the COD of catalyst, 50 ml of the reaction mixture was membrane filtered, and the powder was collected and ultrasonically redispersed in an equal volume of pure water. Subsequently, 2 ml of the dispersion was used as the sample for measuring the COD. Reproduced with permission.[1] 2025, Springer Nature. Copyright © 2025.

reduce the ionization potential (IP, eV) of pollutants and makes them more prone to oxidation. Subsequently, these low-IP pollutants are further oxidized into various organic pollutants through complex reactions with $^1O_2$ or high-valent iron. High-valent iron itself can also directly oxidize phenol without the need for $^1O_2$, mainly in the early stage of the reaction. During this process, the polarity and water solubility of the pollutant degradation intermediates constantly change, and pollutants accumulate on the surface of the catalyst. The feature that the active sites on the surface of this catalyst are masked by contaminants can also be reflected by the changes before and after the TiO_2 reaction: when TiO_2 is added as the aggregation center of low-polarity degradation intermediates in the Co^{2+}/PMS system and then filtered, the COD removal rate is 45.5%, and the TiO_2 turns brown significantly after the reaction (Fig 1b). The FTIR in Fig. 1c shows that after the reaction cycle, both the catalyst surface and the deposited substances therefrom exhibited and significantly enhanced typical organic functional group signals such as –OH/N–H, –C–H, C=O, C=C, and R–O–R′, and these signals became more pronounced as the phenol content increased, indicating that the organic intermediates/products were enriched on the surface. The TGA in Fig. 1d provides further quantitative evidence: before the reaction, Fe–N–C remained almost weightless during the nitrogen temperature rise (intrinsic stability), but after the reaction, it showed a significant weight loss of over 60%, indicating that a large amount of organic substances accumulated on the surface that could be thermally decomposed/carbonized under an inert atmosphere. The two aspects, namely "functional group characteristics" and "mass changes", mutually confirm each other, thus strongly supporting the conclusion that organic substances transferred to the catalyst surface and formed a deposition layer during the reaction process.

OCTP is not a simple restatement of the traditional adsorption concept. During the adsorption process, the captured substances are usually the parent pollutants or the equilibrium distribution results under the given conditions. The system often reaches adsorption equilibrium relatively quickly. A certain degree of reversible desorption then can be achieved through dilution or simple rinsing. In contrast, the core of OCTP lies in the partial oxidation step. During this process, reaction intermediates evolve in polarity, solubility, and surface affinity, making them more likely to accumulate on the catalyst surface. These species may further aggregate or condense, forming a deposition layer that is difficult to remove. Therefore, the surface accumulation related to OCTP continues to increase with the activation of the oxidant and the progress of the reaction time, and is related to the reaction path selection and interface microenvironment, rather than solely determined by adsorption parameters such as specific surface area or surface charge. At the operational level, the deactivation caused by

such deposition often requires solvent cleaning or regeneration steps to be significantly restored, and as the degree of "solidification/condensation" of the deposition increases, it may exhibit partial irreversibility. Based on these differences, we will discuss OCTP as an independent "carbon accumulation" process, and its design and evaluation significance is therefore different from simple adsorption. It is worth noting that current research tends to design catalyst systems with non-free radical or high-valent paths (such as single-atom catalysts)[2-6]. Although these pathways demonstrate outstanding selectivity, whether they will lead to stronger surface solidification deposition is context-dependent: under conditions where the oxidation process tends to remain at the oxygen-containing intermediate stage, and these intermediates further evolve into low solubility/high surface affinity species (and may undergo aggregation or condensation), it is indeed possible to promote the transfer of organic matter to the solid phase and cause surface passivation, thereby converting the short-term kinetic advantage into a stability issue on a full lifespan scale; however, when there are conditions for continuous deep transformation or the oxidation ability is sufficient to drive subsequent mineralization, these pathways can also achieve low residue and better long-term stability[7-8]. This does not deny the value of non-radical pathways, but emphasizes that a clear strategic choice must be made: whether to enhance deep oxidation to inhibit OCTP for mineralization, or to use it as a controllable carbon transfer to collect specific products, and to complement it with a systematic regeneration and solid-phase carbon flow management plan. The most engineering-enlightening aspect of this perspective is that the deactivation caused by OCTP is reversible - Xing et al. could restore catalytic activity by removing surface deposits through methods such as ethanol washing, indicating that deactivation often results from masking rather than destruction. This distinction is crucial: reversible deactivation can be managed through a catalyst regeneration strategy, while irreversible structural damage or poisoning requires a completely different approach.

Durability, regeneration and safety

If the passivation of the catalyst surface caused by OCTP is a common phenomenon, then the assessment of the long-term performance of the catalyst cannot rely solely on the initial kinetics. A highly efficient catalyst that performs well in a short period of time will cause a series of problems if it rapidly accumulates organic carbon or contaminates intermediates during operation: overall system maintenance, reagent consumption and management, generation of secondary waste, and unpredictable performance degradation. These problems that are easily overlooked in single-batch experiments are precisely the key factors determining the feasibility of continuous flow processes. Therefore, an increasing number of studies have begun to incorporate recyclability and stability in practical water-treatment conditions into the core evaluation framework[9-10]. OCTP reveals that the surface properties of the catalyst directly regulate the direction of reaction intermediates. By regulating these interface properties (such as constructing a specific acidic microenvironment[11]), OCTP can be suppressed and the cycling stability of the material can be enhanced. The design concept of catalysts should then shift from a simple "activity-first" approach to a coordinated optimization of "activity, anti-pollution performance and renewability". A effective coping strategy is to establish a feasible reporting standard to enhance the comparability among different studies. This at least includes reports on the aqueous and solid carbon indicators, as well as the long-term cycling performance under actual water quality conditions; and provides the performance after catalyst regeneration. These requirements are also related to safety. The transfer of carbon alters the existence form and location of pollutants. When toxic intermediate products accumulate in solid waste, a change in the treatment strategy is necessary. An ideal OCTP research report should include an assessment of these toxins, taking into account the subsequent treatment and resource recovery of the discarded catalysts.[12].

For quantitative reporting, COD reflects oxygen demand rather than direct carbon content. Solid-phase COD can be affected by digestion strength and matrix effects. It is recommended to use TOC or TC as the primary basis for carbon allocation, with COD reported as an operational proxy under clearly defined protocols. For solid residues, solvent extraction of spent catalysts (e.g., ethanol or acetonitrile) followed by TOC or COD analysis of the extract can quantify extractable organic matter. Appropriate pretreatment, such as removal of inorganic carbon, should be applied. The selected methods (Thermogravimetric analysis and supplementary surface spectroscopy) and QA/QC procedures, including blanks, recovery tests, and repeated digestion or extraction conditions, should be clearly disclosed to reduce inter-study variability. For carbon-based catalysts (e.g., Fe–N–C), baseline correction or isotope labeling can further improve the specificity of carbon deposition quantification.

A Regeneration-Oriented Metric: Catalyst Regeneration Extent

Here, to supplement the OCTP rate and quantify its indexability, the degree of CRE is proposed as a new conceptual indicator that can evaluate the recoverability after a standardized cleaning process:

$$\mathrm{CRE} = \frac{\mathrm{Num.CDAEW}}{\mathrm{Num.ICD}} \times 100\%$$

Among them, Num. ICD is the number of cycles to initial catalyst deactivation and Num. CDAEW is the number of cycles to catalyst deactivation after ethanol washing. CRE answered a question related to OCTP: After carbon transfer or pollutant clogging occurs, how much lifespan can the catalyst regain The closer the CRE value is to 100%, the more reversible the inactivation is. A lower value indicates strong polymerization of the sediments, insufficient cleaning or catalyst damage induced by the regeneration process. Because CRE can be reported as a concise scalar along with the data presented by long-term experiments, it can enhance the comparability without the need for dedicated instruments.

To ensure transparency and repeatability, the CRE report should clearly state:

(i) Inactivation criterion: such as when the activity drops to a certain proportion;

(ii) Cycling scheme: Batch/continuous, oxidant dosage, pH, substrate;

(iii) Regeneration plan: Solvent type, contact time, temperature, number of washes, and any post-treatment activation steps.

CRE ranges from 0 to 100% and reflects lifecycle recoverability. For comparison, it should be reported together with Num. ICD and Num. CDAEW. As a reference, CRE ⩾ 80% indicates good regenerability with mainly removable residues; 50–80% suggests mixed reversible and irreversible loss; < 50% indicates poor regenerability, such as strong deposits or regeneration damage. CRE applies when deactivation occurs within the test window. For large ICD, a threshold-based CRE defined by a preset drop in K_{obs} or removal efficiency may be used, with conditions clearly disclosed. For rapid screening, the K_{obs} ratio can serve as a simplified proxy, while full CRE testing can be reserved for benchmark studies.

Since different reaction pathways and by-products lead to different intermediates and deposition behavior, pollutant selection should be standardized to improve the comparability and reproducibility of CRE. A two-layer strategy is recommended: use shared probe pollutants (e.g., phenol) for cross-study comparison, and select application-related pollutants for practical relevance. The probe should generate representative partially oxidized intermediates, have measurable residual potential, cover different pathway sensitivities, and allow reliable analysis of aqueous transformation and solid residues. At minimum, pollutant type, initial concentration, key properties, and reaction conditions should be reported, and it should be clarified whether CRE is pollutant-specific or consistent within the probe group to promote standardized evaluation.

Conclusions and Outlook

The proposal of OCTP has pointed out a new direction for the optimization of heterogeneous AOP. Future designs should focus on long-term stability and safety research. A feasible method is to inhibit OCTP through the coordinated regulation of "reaction pathway - interface characteristics". This can be achieved by balancing the free radical pathway and the non-free radical pathway, or promoting the complete mineralization of key intermediates. Reducing carbon deposition by means of surface polarity regulation should be considered as well. Complex reaction environment make deep mineralization difficult to achieve. OCTP can be considered to be directed towards a controlled capture and regeneration strategy. By enriching intermediates on the catalyst and then achieving cycle operation through regular mild regeneration can be achieved. However, this requires that the corresponding waste treatment solution must be reliable and environmentally friendly. This approach further drives us to directly integrate the regeneration stage into the process chain. This can be treated as part of the operation cycle rather than a post-event remediation. Ultimately, This framework supports a full evaluation of resource use, emissions, and solid waste toxicity, and promotes a closed-loop approach from reaction design to safe disposal to ensure effective treatment with controlled environmental risks.

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Conflict of Interest

The authors declare no competing interests.

Ethical Approval

The English language of the article was improved with ChatGPT, developed by OpenAI. After using the tool, the authors thoroughly reviewed and revised the material as needed and assume full responsibility for the final content of the publication.

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