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Stability and regeneration of metal catalytic sites with different sizes in Fenton-like system

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ABSTRACT

Metal-based catalysts with different site sizes (*e.g.*, metal nanoparticles (NPs) and single atom catalysts (SACs)) demonstrated outstanding catalytic activities in versatile Fenton-like reactions. However, the surface/structural instability is a critical issue, which will result in rapid passivation in Fenton-like reaction and fail in long-term operation. The catalytic stability of the catalysts with different metal sizes considering versatile peroxides (H_2O_2, peroxymonosulfate (PMS), and peroxodisulfate (PDS)) should be analyzed. In addition, strategies for catalyst regeneration and recyclability improvement are also important to realize the metal-based catalysts for practical applications. In this review, catalytic stability of catalysts with different metal sizes in the backgrounds of versatile peroxides and water matrixes in Fenton-like reactions were first evaluated. Regeneration of metal catalytic sites with different methods were also reviewed. Finally, major challenges and development of methods concerning the stability and regeneration of metal catalytic sites with different sizes were discussed to understand the future researches of metal catalytic sites in Fenton-like reactions.

1. Introduction

Recently, the design of cost-effective and stable Fenton-like based catalysts with highly active metal sites for the environmental remediations is critical. The activations of various Fenton-like systems using different peroxides (*e.g.*, H_2O_2, peroxymonosulfate (PMS), and peroxodisulfate (PDS)) *via* the heterogeneous metal-based catalysts have received more and more attentions due to their high-efficiency in oxidizing versatile refractory organics [1–4]. Substantial effort has been devoted recently to exploiting the highly active metal-based catalysts. Since the metal centers are the dominant active sites for Fenton-like reactions, a series of metal-based nanoparticles (NPs) have been developed for the activations of various peroxides. Various metal NPs catalysts (*e.g.*, Co_3O_4, $FeMnO_n$, $FeCoO_n$, Fe^0) as well as their carbon hybrids have showed extraordinary excellent catalytic performances towards various peroxides [5–7]. However, most metal NPs catalysts exhibited a wide range of particle size distributions and surface features; this posed the serious challenges to the rational designs of active metal sites for different Fenton-like reactions. In addition, since most of the metal atoms in the metal NPs catalysts are embedded inside the catalysts, the atomic utilization of metal NPs catalysts was mainly controlled on the surface of catalysts [5,6]. Therefore, many recent studies have focused on the downside of particle size and engineering surface topography to obtain more surface metal catalytic sites.

Single-atom catalysts (SACs) with fully exposure of metal sites have showed superior selectivity and efficiency in Fenton-like reactions as compared with those of metal NPs catalysts [8–10]. The superior catalytic activities of SACs were mainly ascribed to the atomically-dispersion of metal atoms. In addition, the geometric, electronic properties and metal-support interactions of SACs were closely related to the single-atom metal sites and supporting matrixes, and ultimately affect their activation pathways towards different peroxides and catalytic performances [11–14].

Although metal NPs and SACs demonstrate outstanding catalytic activities in versatile Fenton-like reactions, the surface/structural instability of both metal NPs and SACs is a critical issue, which will result in rapid passivation in catalysis and fail in long-term operation. Previous reviews always focused on the catalytic activities and mechanisms of different metal catalytic sites in various Fenton-like systems. The catalytic stability of the catalysts with dif-

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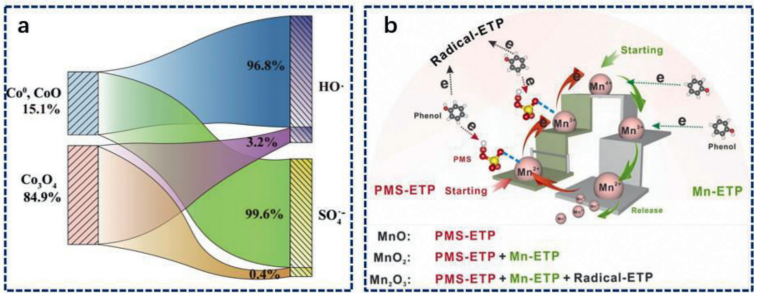

Fig. 1. (a) Contributions of low-valent Co species and Co_3O_4 to $HO^{\bullet}/SO_4^{\bullet-}$ generation. Reprinted with permission [5], Copyright 2020, Elsevier. (b) Catalytic mechanism of MnOx with PMS activation for phenol removal. Reprinted with permission [6], Copyright 2020, Elsevier.

ferent metal sizes considering the versatile peroxides (H_2O_2, PMS, and PDS) were not considered previously, which can be well analyzed to understand the potential relationships. In fact, the water matrixes (*e.g.*, coexisting anions) were also the important factors that affected the catalytic stability of different metal catalytic sites. In addition, strategies for catalyst regeneration and to improve the recyclability are also important to realize the metal-based catalysts for practical applications, but they were also less reviewed.

2. Stability of different metal catalytic sites for versatile peroxides

Multiple peroxides (H_2O_2, PMS and PDS) should be considered when analyzing the effect of metal size on catalyst stability [1–4]. The difference in the catalytic stabilities between the SACs and metal NPs was relatively lower for H_2O_2 activation as compared with those for PMS/PDS activation [2–4]. The H_2O_2 activated by the metal-based catalysts (metal NPs and SACs) was always based on the $HO^{\bullet}$-based pathways, which mean the high mineralization degree of organic pollutants with less intermediate products. Therefore, the coverage of catalytic sites by reaction intermediates was retarded in the metal-based catalysts/H_2O_2 systems leading to the less inactivity of catalysts. In contrast, both radical or nonradical pathways always occurred in the PMS/PDS systems with lower mineralization of organic pollutants for both metal NPs and SACs. For example, low-valent Co species (*e.g.*, Co^0 and CoO) and high-valent species (Co_3O_4) showed significant different contributions to the $HO^{\bullet}/SO_4^{\bullet-}$ generation *via* PMS activation (Fig. 1a) [5]. Catalytic mechanism of MnO_x with PMS activation for phenol removal showed three kinds of complicated mechanisms (Fig. 1b), which resulted in different catalytic capacities and stabilities [6]. PMS/PDS can be activated into nonradical/radical reactive species by the metal NPs, which were regulated by their physicochemical features, such as crystalline structure and defective sites (oxygen and iodine vacancies). In fact, PMS/PDS activated by SACs always showed more complicated mechanisms since these SACs can be anchored onto the various substrate matrixes, which exhibited both metal and substrate properties in PMS/PDS catalytic systems [1,9,11–13]. In addition, the intermediate products would be generated and adhered onto the catalytic sites of both metal NPs and SACs, which resulted in the catalyst poisoning [11,13]. Recent studies also reported that some catalytic sites would form the complexes with PMS/PDS, which would further hinder the recovery of catalytic sites [1,11,13].

Considering that both metal NPs and SACs can be used in versatile peroxide systems, their superiorities in stability can be summarized. A comparison of the catalytic stability of metal NPs catalysts and SACs in H_2O_2 activation and PMS/PDS activation system was summarized in Fig. 2. It was obvious that the recovery efficiency of most SACs in PMS/PDS activation system can be stable over 90% (Fig. 2), while most metal NPs counterparts only achieved 50%−80% of recyclability in PMS/PDS system [15]. Qi *et al.* fabricated both Co-SACs and Co NPs catalyst by using lignin as carbon precursor, and compared their catalytic stabilities in PMS activation system [1]. The Co-SACs showed excellent recyclability after four cycles with only 3% loss of catalytic activity. This is in stark contrast to the Co NPs catalyst that the efficiency of Co NPs catalyst decreased by over 30% after running four times (Fig. 3a). As a result, the SACs exhibit their superiorities in the high stability, which can be attributed to the high dispersion of single metal sites in the supporting matrixes as well as the strong metal-support interactions, which endowed the metal sites with high catalytic activity and protected the active sites from metal leaching (Fig. 3b). Although the catalytic stabilities of SACs as well as their structural stabilities (*e.g.*, metal leaching, substrates decomposition) seemed to be inevitable in most reported work and almost 2%−10% of single-atom metal leaching can be observed in some cases [16–20]. This can be ascribed to several well-known factors, summarized in Fig. 4 [21]. As for the metal NPs, although their recyclability was lower than those of SACs, the relatively mature catalytic membrane immobilized with metal NPs can effectively improve the stability of the catalytic system, while single-atom catalytic membranes are still in their infancy [9].

Based on the above analysis, increasing the density of metal active sites in the SACs or stabilizing the atomically dispersed metal sites could effectively improve the stability of SACs in versatile peroxide systems [19]. Li *et al.* reported that the SACs with 8.0 wt% of single-atom Co loading could achieve almost 100% of recovery after 5 cycles [22]. To obtained the stabilized catalytic sites, Zuo *et al.* showcased a strategy to stabilize the atomically dispersed Fe catalysts by anchoring the Fe atoms in a C_3N_4-rGO double-layered support (C_3N_4-Fe-rGO) [23]. The double-layered support could significantly prohibit metal leaching and agglomeration of Fe atoms while maintaining activity of the reaction sites. The layered C_3N_4-Fe-rGO exhibited stable catalytic efficiency (>95%) for several reaction cycles, while the efficiency of single-layered C_3N_4-Fe decreased by over 50% only after five cycles. They also found that the populations of the Fe sites as well as their reductive and oxidative potentials were essentially unaltered, indicating the stable bonding environment for Fe atoms in the ternary C_3N_4-Fe-rGO. As for the metal NPs, the promotion of catalytic stability of metal NPs catalysts can be achieved by stabilizing the catalytic sites with the supports after specific modification [24]. For example, Fe_3O_4 magnetic nanoparticles stabilized on the carbon substrates after poly(catechol) modification exhibited exceptional high stability and catalytic activity towards refractory organics [24]. The poly(catechol) could not only stabilize the Fe_3O_4 to avoid the loss of metal sites, but also promoted the regeneration of Fe(II) *via* the strong metal-support interaction to promote the activation of peroxides. As a result, negligible recovery loss were observed after 8

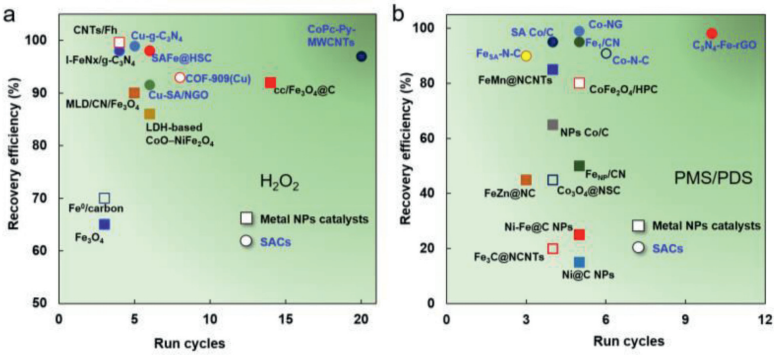

Fig. 2. Comparison of the catalytic stability of metal NPs catalysts and SACs by (a) H_2O_2 activation and (b) PMS/PDS activation.

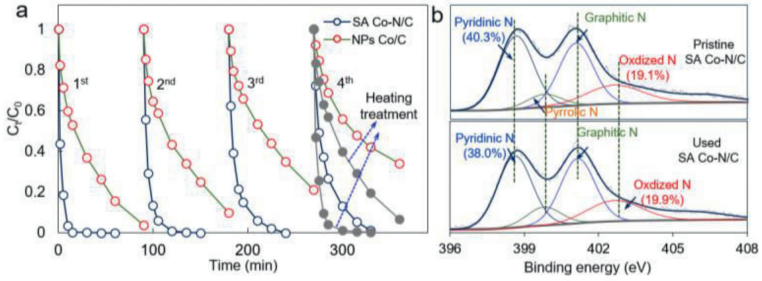

Fig. 3. (a) Stability of SA Co-N/C and NPs Co/C. (b) N 1s of original and used SA Co-N/C. Reprinted with permission [1], Copyright 2020, Elsevier.

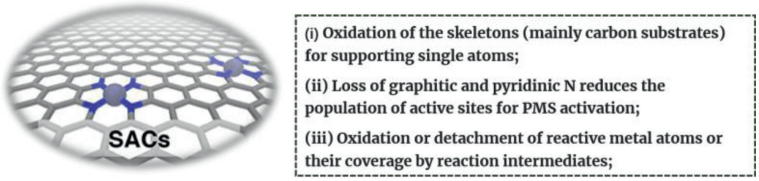

Fig 4. Factors affecting the catalytic stabilities of SACs.

cycles with little iron leaching during all running cycles [24]. This also provides a strategy for improving the stabilities of both metal NPs and SACs.

3. Stability of different metal catalytic sites for water matrixes

Inorganic anions (*e.g.*, $H_2PO_4^-$, CO_3^{2-}, HCO_3^-, NO_3^-, Cl^-) and natural organic matters (NOMs) that exist in water bodies have a great influence on the activation of peroxides [25–27]. These substances could react with $HO^•$ and $SO_4^{•-}$ to form weaker radicals or compete with pollutant molecules with the active sites, resulting in the inferior degradation stability [27,28]. As a result, effect of water matrixes on the catalytic stability of metal-based catalysts (metal NPs and SACs) can be analyzed. A comparison of the catalytic performance of SACs/PMS and metal NPs/PMS systems with different types of coexisting ions and NOMs indicated that SACs show stronger anti-interference performance to coexisting substances than that of metal NPs [29,30]. Luo and co-workers reported that the single iron atom anchored in C_3N_4 (Fe_{SA}/CN) was more stable than the Fe_{NP}/CN during the activation of PMS with coexisting inorganic ions, and NOM [18]. Because the Fe_{SA}/CN only produced $^1O_2$ *via* activating PMS, which exhibited strong resistance to environmental factors with excellent cycle stability [18]. In contrast, radicals-dominated pathways induced by Fe_{NP}/CN can be easily quenched by the anions or NOMs in the PMS systems [1,31]. Similarly, Qi *et al.* reported that degradation of NPX by the Co-SAC/PMS system with the background of $H_2PO_4^-$ and NO_3^- can be restrained due to the quenching of active radicals to form the secondary radicals with lower activity [1]. The inhibition caused by humic acid was most likely attributed to the attachment with catalytic sites on surface of Co-SAC catalyst as well as the competition with NPX for catalytic sites, thus hindering the ROS formation in Co-SAC/PMS system [27,32].

Generally, the coexisting CO_3^{2-} and HCO_3^- could transform the high-oxidative radicals ($HO^•$ and $SO_4^{•-}$) into the lower oxidative carbonate radicals (*e.g.*, $HCO_3^•$, and $CO_3^{•-}$), which reduced the catalytic activity in the radical-dominated systems [33,34]. However, the $HCO_3^•$, and $CO_3^{•-}$ were more selective than $HO^•$ and $SO_4^{•-}$ for targeted pollutants with electron-rich moieties [35–37], and recent studies reported that the nonradical-dominated reaction systems induced by both metal NPs and SACs were insensitive to the coexisting CO_3^{2-} and HCO_3^- [35–38]. It was also reported the existence of dual-effects of Cl^- for PMS activation *via* metal NPs and SACs [1,38]. Low-concentrations (2–5 mmol/L) of Cl^- had a slight inhibitory effect on the degradation of organics, which can be attributed to the reaction between Cl^- and free radicals ($HO^•$ and $SO_4^{•-}$), producing the $Cl^•$ and $Cl_2^{•-}$ with lower redox potentials [2,39]. In contrast, catalytic process can be greatly facilitated with high concentrations (500 mmol/L) of Cl^-, which was due to the generation of high HOCl as well as the nonradical pathways insensitive to the Cl^- [1,38]. Such dual-effects were mainly affected by the concentrations of Cl^- existed in the water matrixes rather than the sizes of metal sites. As a result, the dual-effects of Cl^- on the PMS activation were universal and have been reported in both metal NPs/PMS and SACs/PMS systems [2,38]. Peng *et al.* reported the ultrafast paracetamol (PCM) degradation (0.534 min^{-1}) in the

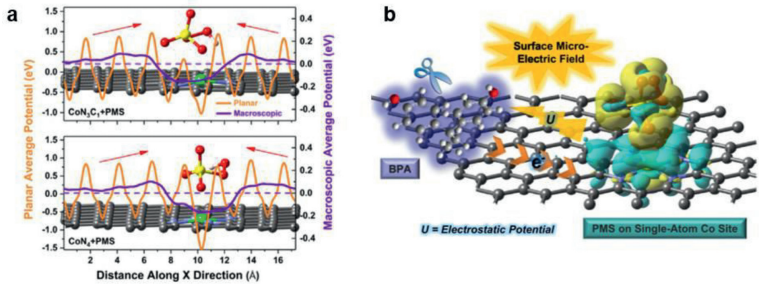

Fig. 5. (a) Macroscopic average electrostatic potentials of different SAC/PMS complexes. (b) Mechanism of surface micro-electric fields on BPA oxidation by PMS activation. Reprinted with permission [40], Copyright 2020, Elsevier.

seawater (salinity of 3.6%) by Co_3O_4-based catalyst as compared with that (0.162 min^{-1}) in tap water [2]. Oxidization of NPX by Co-SACs/PMS system with coexisting 50 mmol/L of Cl^- was 2.5 times higher than that of lower Cl^- background (5 mmol/L) [1]. Such results unveiled the potential applications of oxidization of refractory organic pollutants in saline waters for both metal NPs and SACs.

Although the stability of metal catalytic sites can be affected by coexisting anions no matter their sizes, many studies have further evaluated the decomposition of organics by the SACs and metal NPs catalysts in different in actual water bodies [30,40]. Peng and co-workers reported the strong anti-interference Fenton-like reaction due to the construction of surface micro-electric fields on hollow Co-SAC (Fig. 5) [40]. Zou and co-workers reported the negligible impact of organics degradation operated in Poyang lake water and Yangtze river water for PMS activation by Fe_{SA}/CN [18], indicating its potential application for practical waters. There are also observations that the Fenton-like activities of SACs decreased in the actual water bodies, mainly due to the coverage and blockage of the catalytic sites by the degradation intermediates and impurities. In particular, most reported metal NPs catalysts always showed a higher loss of degradation activity in the actual water environments [41,42], which further indicated the superior catalytic stability of SACs over metal NPs catalysts.

4. Regeneration of metal catalytic sites

Regeneration is another major concern for the metal-based catalysts (metal NPs catalysts and SACs) to be available in practical applications. The reduction of recyclability of catalysts was mainly due to the deactivation of the catalytic sites resulted from the coverage of product intermediates and unreacted peroxides, and the loss of catalytic sites. Therefore, the regeneration of deactivated catalysts can be achieved by a variety of methods (*e.g.*, calcination, solvent washing, metal impregnation, and UV irradiation), which were universal to both metal NPs catalysts and SACs [1,30,43–48].

Generally, calcination of the deactivated catalysts at a medium temperature (350–500 °C) is the most widely used method for catalyst regeneration and shows a very promising recovery capacity [11,13,38]. This is partially due to the thermal decomposition of the product intermediates attached on the surface of catalysts, which could re-expose the catalytic sites for use. Shang *et al*. reported the severe deactivation (75% of catalytic performance loss) of Fe_3C@NCNTs catalysts after four running cycles. The exhausted catalysts were calcinated at 350 °C and the catalytic activity could be recovered from 25% to more than 98%. Ma and co-workers also achieved 100% of recovery of the biochar loaded with $CoFe_2O_4$ nanoparticles (80% of performance left after 5 cycles) *via* calcining at 400 °C [44]. Calcination of the exhausted ACs also showed almost complete recovery of the catalytic activity [1]. Deactivated single-atom cobalt sites anchored on the lignin-based carbon frameworks can be effectively regenerated by heating treatment at 400 °C with the same kinetic rate as the original sample [1,30,43]. In addition, the morphologies of these recovered catalysts remain intact after calcination, indicating that the heat-treatment was a promising method for exhausted catalysts with both high recovery capacity and high stability.

The facile solvent washing can achieve the recovery of some exhausted metal-based catalysts [46,48,49]. Pan and co-workers reported that the Fe_2O_3-based catalyst was slightly deactivated after five consecutive MB degradation due to the cumulative degradation products occupying the active sites [49]. The catalytic activity could be fully recovered after ethanol washing. The same group further examined the reusability of the single-atom Fe anchored carbon nanotubes for MB degradation [46]. Methanol washing was also proved to be efficient to recover the metal NPs and SACs. In addition to these organic solvents, Oladipo *et al*. reported the desorption of the attached dye intermediates from the exhausted CoO–$NiFe_2O_4$ by using 0.01 mol/L of NaOH [48]. The Fenton-like performance of the NaOH-eluted hybrid catalyst after six cycles can be recovered ~90%. However, the solvent method has a narrow application, since it can only be used in catalytic systems in which the degradation products can be dissolved by these solvents.

Since catalyst deactivation can also be ascribed to the loss of metal catalytic sites, some attempts have been made to re-impregnate the metal sites onto the used catalysts [47,50,51]. Successful attempt to regenerate an exhausted iron-coated Fenton catalyst was described by Akinremi *et al*. [47]. The novel iron-based catalyst was deactivated due to the iron loss caused by acid hydrolysis in a Fenton-like system. The deactivated catalyst was modified by a hydroxylamine reaction and then the iron(III) salt was impregnated onto the hydroxylaminated iron-based catalyst. Li *et al*. reported the recovery of the V and W active sites in the V_2O_5-WO_3/TiO_2 catalyst *via* the impregnation of V and W elements from the salts of ammonium metavanadate (NH_4VO_3) and ammonium paratungstate hydrate ($H_{40}N_{10}O_{41}W_{12}{\cdot}xH_2O$) [51]. The catalytic performance of regenerated V_2O_5-WO_3/TiO_2 catalyst can be almost completely recovered as compared with the fresh catalyst. Although the preliminary results suggest that this method can be used for catalyst recovery, the regeneration process is relatively complicated and can only be used in cases of severe metal sites loss. Other regeneration methods, *e.g.*, ultrasonic regeneration, UV irradiation, are sporadically reported in some cases [45,52], which can be used in both metal NPs and SACs.

5. Conclusions and perspectives

Metal-based catalysts with both metal NPs and SACs can be used in versatile peroxide systems, while the difference in the catalytic stabilities between the SACs and metal NPs was relatively lower for H_2O_2 activation as compared with those for PMS/PDS activation. The H_2O_2 activated by the metal-based catalysts was always based on the $HO^{\bullet}$-based pathways, which mean the high

mineralization degree of organic pollutants with less intermediate products. In contrast, both radical or nonradical pathways always occurred in the PMS/PDS systems with lower mineralization of organic pollutants. Recovery efficiency of most SACs in PMS/PDS activation system can be higher than those of most metal NPs counterparts. Basically, the high stability of the SACs can be attributed to the high dispersion of single metal sites in the supporting matrixes as well as the strong metal-support interactions, which endowed the metal sites with high catalytic activity and protected the active sites from metal leaching. In addition, the stability of metal catalytic sites can be affected by coexisting anions no matter their sizes, while most reported metal NPs catalysts always showed a higher loss of degradation activity in the actual water environments, which further indicated the superior catalytic stability of SACs over metal NPs catalysts.

The regeneration of deactivated catalysts can be achieved by a variety of methods (*e.g.*, calcination, solvent washing, metal impregnation, and UV irradiation), which were universal to both metal NPs catalysts and SACs. In future, more efforts can be donated to understand the deactivation pathways of catalysts in different reaction environments. With increased understanding of deactivation mechanisms, effective strategies to prolong the lifetimes of highly active and selective catalysts can be expected. Developing catalysts with self-healing abilities is another intriguing approach to addressing these stability issues.

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.

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