Original Research

Enhanced utilization efficiency of peroxymonosulfate via water vortex-driven piezo-activation for removing organic contaminants from water

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The efficient activation and utilization of peroxymonosulfate (PMS) in PMS-based advanced oxidation processes is a high-priority target for the removal of organic contaminants. This work introduces a water vortex-driven piezoelectric effect from few-odd-layered MoS2 into the PMS activation to remove benzotriazole (BTR) and other organic contaminants from the water. Approximately 91.1% of BTR can be removed by the MoS2 piezo-activated PMS process with a reaction rate constant of 0.428 min−1, which is 2.69 times faster than the sum of the individual MoS2, water vortex, and piezocatalysis rates. Meanwhile, the PMS utilization efficiency reached 0.0147 in the water vortex-driven piezo-activation system, which is 3.97 times that of the sum from the vortex/PMS and MoS2/PMS systems. These results demonstrate that the presence of MoS2 under a water vortex can trigger a piezoelectric potential and generate abundant free electrons to activate PMS to generate various active species for degradation of organic contaminants.

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1. Introduction

Peroxymonosulfate (PMS)-based advanced oxidation processes (AOPs) have aroused immense interest for in-situ chemical oxidation for remediation of contaminated wastewater [1–5]. For this application, PMS, with its asymmetric structure (HO−O−SO3)2−, needs to be activated by the input of energy or by the electron transfers indicated in Eqs. (1) and (2) to break the superoxide O−O bond. This then generates strong reactive oxygen species (ROS) to oxidize organic contaminants in water [6–11]. Thus, the decomposition and utilization efficiency of PMS are extremely important factors to be considered in water treatment applications.

\[
\text{HSO}_5^- + \text{energy} \rightarrow \text{SO}_4^{2-} + \text{HO}^* \quad (1)
\]

\[
\text{HSO}_5^- + \text{e}^- \rightarrow \text{SO}_4^{2-} + \text{OH}^- \text{ or } \text{SO}_3^{2-} + \text{HO}^* \quad (2)
\]

A wide variety of strategies have been employed for PMS activation, such as the use of metal ions, ultraviolet (UV), heating, or ultrasonication (US) [8,12–16]. However, the introduction of transition metals not only consumes a large amount of chemicals but also brings some drawbacks with regards secondary pollution from the leaching of metal ions or more difficult recovery of the catalyst. Moreover, UV, heating, or US will cause a high energy consumption [17], thus limiting their application for industrial scale wastewater treatments (Scheme 1). Therefore, it is imperative to find an environmentally friendly, simple yet effective method for PMS activation to enhance PMS utilization efficiency.

The piezoelectric effect is gaining much attention as a physical phenomenon that converts mechanical vibrations into electrical energy [18,19]. In this process, an external mechanical force induces the deformation and polarization of the piezoelectric material to form a piezo-potential to drive the transfer and migration of intrinsic free electrons [20]. The piezoelectric effect has been applied for the elimination of contaminants and the activation of PMS [21–24]. However, most of these applications required external energy from ultrasonic vibration with a relatively high frequency (kHz) to trigger deformation of the piezoelectric materials, which also caused high energy consumption and limited their
of MoS2 compared with other traditional activation methods.

2. Materials and methods

All materials and reagents used in this study, the synthesis and characterization of materials, and analysis methods are described in detail in the supplementary material (Texts S1–4).

3. Results and discussion

3.1. Piezoelectric characteristics of MoS2

The few-odd-layered MoS2 was hydrothermally synthesized [26], and the phase, composition, and morphology characterized via X-ray diffraction, X-ray photoelectron spectroscopy, and transmission electron microscopy, respectively (see Figs. S1–3). To investigate the piezoelectric nature of the as-prepared MoS2, the piezoelectric force microscope (PFM) technique was carried out under an applied voltage on the tip of the sample. Fig. S4a reveals the quantitative height signal and the petal-like structure, indicating a rough surface of the prepared MoS2. Obvious polarization rotation of 180° was observed in the phase-voltage loop, and the amplitude-voltage loop exhibited a typical butterfly-shape curve (Fig. S4b), thus demonstrating the nonzero remnant polarization and piezoelectric properties of the MoS2.

The piezoelectric current was investigated and electrochemical impedance spectroscopy (EIS) used to elaborate on the separation and transfer of the piezo-induced electrons. From Fig. S5a, the samples showed a prompt and repeatable piezoelectric current response during the consecutive on-off stirring cycles. Fig. S5b shows that the semicircular Nyquist diagram of the MoS2/vortex was smaller than that obtained under static conditions, indicating a smaller resistance. These results suggest that the vortex from water can deform MoS2 to generate a piezoelectric potential, which not only promotes the separation of electrons and holes, but also accelerates local electron discharge to the surrounding medium, resulting in an increase in current response and a decrease in resistance [27,28].

3.2. Performance of PMS piezo-activation for BTR degradation

The piezocatalytic performance of the few-odd-layered MoS2 was studied by PMS activation for BTR removal. Fourier-transform infrared (FTIR) spectra of PMS, MoS2, and MoS2/PMS were first explored to investigate the interaction between the piezo-process of MoS2 and the decomposition of PMS. From Fig. 1a, the wave-lengths at approximately 1631 and 3384 cm⁻¹ could be ascribed to the peaks of water, and the bands at approximately 1107 and 1274 cm⁻¹ were attributed to the S–O stretching vibration of either HSOS or SO4²⁻ [29,30]. The intensity of the 1107 and 1274 cm⁻¹ bands were clearly reduced in the MoS2/vortex/PMS system, demonstrating that the PMS molecules were decomposed and activated in the MoS2 piezo-process.

Moreover, electron paramagnetic resonance (EPR) experiments were conducted to confirm the existence of ROS in the PMS activated systems by MoS2 piezocatalysis. From Fig. 1b, a typical 1:1:1:1 triplet signal of TEMP-1O2 was found, demonstrating the existence of 1O2 in the MoS2/vortex/PMS system. The characteristic signal of DMPO-OH, with intensities of 1:2:2:1, indicated the presence of HO²-. No signal from DMPO-SO4 was observed because of rapid transformation toward other radicals [31,32]. Furthermore, DMPO-O2 was also found in the MoS2/vortex/PMS system, suggesting the generation of O2⁻-. These results indicated the piezoelectric process of MoS2 induced the decomposition of PMS to generate various ROS capable of attacking organic pollutants in water.

The BTR degradation performance and PMS utilization efficiency were investigated for the PMS systems activated by MoS2 piezocatalysis. From Fig. 1c and d, in the absence of MoS2, the individual PMS showed limited BTR degradation (2.3%) after adsorption for 10 min in the static condition. In the presence of MoS2, PMS showed a slight oxidation (4.8%) for BTR in the static condition. In the co-presence of MoS2 and hydromechanical energy from the vortex, the BTR degradation efficiency reached 66.1% with a reaction rate constant (k) of 0.189 min⁻¹, owing to the fact the piezo material MoS2 was deformed by the vortex to produce a piezo-effect, thus inducing the piezo-catalytic redox process for BTR degradation. Significantly, the efficiency increased to 91.1% after introducing PMS into the piezo-process of MoS2. The corresponding k value increased to 0.428 min⁻¹, which was 2.09 times faster than the sum of the MoS2/PMS, vortex/PMS, and MoS2/vortex, exhibiting an obvious synergetic effect. This is because the formed piezoelectric potential in MoS2 under an external vortex can drive the separation and transfer of piezo-induced carriers. These electrons participated in the PMS activation to break the O–O bond to generate various active species which then attacked the BTR molecules [33].

During the MoS2 piezo-activated PMS for BTR degradation, one key concern is whether the oxidant is fully utilized. To clarify this,
the PMS decomposition and utilization efficiency were evaluated by measuring the PMS concentration and calculating the reaction stoichiometric efficiency. Fig. 1e shows that the PMS decomposition efficiencies reached 62.11% in MoS2/vortex/PMS, 2.31% in MoS2/ PMS, and 1.41% in the vortex/PMS system. More PMS molecules were decomposed in the MoS2 piezocatalytic system, indicating the O–O bond of PMS was broken through electron transfers from the piezo-process. Moreover, the PMS utilization efficiency of MoS2/ vortex/PMS reached 0.0147, which was 3.97 times greater than the sum of the vortex/PMS (0.0016) and MoS2/PMS (0.0021) systems (Fig. 1f). This is because the external hydromechanical energy from the vortex triggered the piezo-response of MoS2 to generate more electrons for PMS activation to then produce various active species. This showed a competitive PMS utilization efficiency than a previous study [34].

The influences of MoS2 and PMS dosage in the MoS2 piezo-activated PMS process were further studied. From Fig. S6a, the BTR degradation efficiency gradually increased with an increase in MoS2 content. Similarly, an increase in the PMS dosage from 0 to 0.5 g L⁻¹ also significantly promoted BTR degradation, but the elevation was insignificant when the PMS dosage was greater than 0.5 g L⁻¹ because of the self-consumption of SO₄²⁻ by excessive PMS (Fig. S6b) [35]. These results suggest that only a suitable dosage of catalyst and PMS are beneficial to the reactive species generation and PMS utilization.

Moreover, the mineralization degree of BTR was determined using total organic carbon (TOC) analysis. From Fig. S7, approximately 24% of TOC was removed within 60 min in the MoS2/vortex/PMS system, suggesting that some organic intermediates were generated during the process.

3.3. Effects of different operational parameters

The effects of different operational parameters on pollution degradation were further evaluated in the piezo-activated PMS process, including scavenger molecule concentration; stirring speed; catalysts, pollutant, and ion types; water matrixes; and the catalyst reusability. To elucidate the contribution of each ROS on the BTR degradation in the MoS2/vortex/PMS system, experiments were conducted on the existence of different radical scavengers, including AgNO₃ (scavenger of e⁻), EDTA-2Na (scavenger of h⁺), methanol (MeOH, scavenger of HO·/SO₄²⁻), SOD (scavenger of O₂⁻), tert-butanol (TBA, scavenger of HO·), and histidine (His, scavenger of ¹O₂) [36]. From Fig. S8, the introduction of His provided a dramatic inhibitory effect, and the BTR degradation efficiency decreased from 91.1% to 4.5%. With the addition of AgNO₃, EDTA-2Na, MeOH, TBA, and SOD, the BTR removal efficiencies slightly reduced from 91.1% to 74.4%, 70.3%, 71.1%, 83.5%, and 81.9%, respectively. The results indicated that ¹O₂ played a dominant role in BTR degradation while other reactive species made a minor contribution. In light of the generation of HO·, SO₄²⁻, and ¹O₂ during PMS activation by the MoS2/vortex system, their contributions to the catalytic degradation of BTR were quantified (see Text S5 and Fig. S9) [37,38]. From Fig. 2a, the contributions of HO· and SO₄²⁻, O₂⁻ and ¹O₂ were calculated to be 44.9%, 22.9%, and 97.5%, respectively.

Furthermore, piezoelectricity is known to be dependent on the applied stress employed. From Fig. 2b, the BTR removal efficiency gradually increased with the increase in magnetic stirring speed. This is because faster stirring can provide a stronger vortex to drive MoS2 to produce a stronger piezo-potential. It is beneficial for the harvest of hydromechanical vibration to induce catalytic redox reactions, thereby facilitating PMS activation to degrade BTR.

Moreover, a comparison of PMS utilization efficiency was carried out among as-prepared MoS2, BaTiO3, and commercial MoS2. BaTiO3 and non-piezoelectric commercial MoS2 showed negligible BTR degradation efficiencies and limited PMS utilization efficiencies (Fig. 2c and Fig. S10). This is because the weak external hydromechanical energy from the vortex was not able to motivate the piezoelectric response of BaTiO3, and the non-piezoelectric commercial MoS2 had no piezoelectric property, therefore both of these cannot generate a piezoelectric effect to activate the PMS under a water vortex.
In addition to BTR, other various organic pollutants, including ciprofloxacin (CIP), ornidazole (ORZ), sulfamethoxazole (SMX), metronidazole (MTZ), and benzothiazole (BTH) were also investigated. As shown in Fig. 2d, the removal efficiency reached, respectively, 99.2%, 97.1%, 88.3%, 85.6%, and 78.9% in the water vortex-driven piezo-activation of the PMS system. These results solidly confirm the excellent organic contaminants treatment performance in the present MoS2/vortex/PMS system.

As common factors widely existing in actual wastewater, Cl⁻, NO₃⁻, CO₃²⁻, SO₄²⁻, and PO₄³⁻ were selected to investigate their influence on BTR degradation (Fig. 2e). The BTR removal efficiency slightly decreased from 91.1% to 78.4%, 75.3%, 76.1%, and 81.5% in the presence of Cl⁻, NO₃⁻, CO₃²⁻, and SO₄²⁻ anions, respectively, but dropped to 62.2% in the presence of PO₄³⁻. This may be because the PO₄³⁻ competed with BTR to consume the ROS [39].

In complexed water matrices, the BTR removal ratio was slightly decreased, being 70.4%, 81.1%, and 61.6% in rain, river, and sewage, respectively (Fig. 2f). This is because some amounts of organic substances existed in these practical water media (Table S1), and these may form a competitive relationship with BTR for ROS. In addition, the used MoS₂ was employed to further study the recycling performance for BTR degradation in the MoS₂/vortex/PMS reaction system (Fig. S11). A comparative BTR degradation efficiency was observed after 4 cycles (85.3%), demonstrating the excellent stability and reusability. Moreover, the morphology of the used MoS₂ was explored by TEM (Fig. S12a) and HRTEM (Fig. S12b), showing a similar structure to the fresh MoS₂ and indicating the stability of the MoS₂.

Generally, the average concentration of BTR is detected with 10 μg L⁻¹ in a natural water body, and the concentration can reach up to 1.1 mg L⁻¹ in the groundwater below a deicing pad, at a regeneration plant and snow disposal site [40]. Moreover, in wastewater treatment plants, the concentrations of pollutants are generally high, where the unit of concentrations of pollutants can reach from μg L⁻¹ to mg L⁻¹. Accordingly, different initial concentrations of BTR were further studied. From Fig. S13, the degradation efficiency of BTR was almost 100% at the initial concentration of 1 mg L⁻¹, 96.9% at 5 mg L⁻¹, 85.3% at 10 mg L⁻¹, and 76.2% at 20 mg L⁻¹ within 30 min. However, the BTR degradation efficiency was only 54.1% at an initial concentration of 50 mg L⁻¹ within 30 min in the MoS₂/vortex/PMS system. The higher initial BTR concentration resulted in a decrease in the BTR degradation efficiency, which was due to the increase in BTR molecules competing with each other for the active species to lower the whole removal efficiency.

In addition, the pH of the point of zero charge (pHpzc) of the MoS₂ was measured to be approximately 2.8 using a Malvern Zetasizer Nano ZSE (Fig. S14a). The influence of the initial pH on the BTR degradation was studied. From Fig. S14b, the BTR degradation was significantly inhibited under alkaline conditions, and slightly enhanced in acidic solution. A detailed discussion on this topic is provided in the supplementary materials.

3.4. BTR degradation pathways and toxicity assessment

The Fukui function distribution was calculated to predict the active sites for ROS during the attack of the BTR molecules. An optimized BTR model is presented in Fig. 3a and the Fukui function values of ω are shown in Fig. 3b and Table S2. The 3C and 6C atoms, with higher ω values, were more susceptible to be nucleophilically attacked, while the 7N and 9N atoms, with lower ω values, were more easily electrophilically attacked [43]. Based on the theoretical calculation of the Fukui function and the liquid chromatography quadrupole time-of-flight tandem mass spectrometry (LC-TOF-MS) data, possible pathways for the BTR degradation were proposed (Fig. 3c). In terms of pathway I, the hydrogenation of the aromatic ring first occurred at 3C and 6C after being attacked by a hydroxide radical, and the aromatic ring was further opened to generate product of E (m/z = 127), F (m/z = 127), and G (m/z = 127), then the product O (m/z = 74) and P (m/z = 157) was produced via further oxidation and ring opening of J, K, L, and M [44]. In pathway II, the triazole ring was opened by the ROS, hydroxylated, then had the
C–C bond cleaved to successively form D, H, I, and N [45–47]. Moreover, the acute toxicity of BTR and its intermediate degradation products were evaluated using the Toxicity Estimation Software Tool (T.E.S.T.) with a quantitative structure-activity relationship (QSAR) mathematical model including acute toxicity, developmental toxicity, mutagenicity, and bioaccumulation toxicity (Text S6). From Fig. 3d and Table S3, the bioaccumulation toxicity of the degradation intermediate products was lower than that of BTR (except for G). From the results of the developmental toxicity and mutagenicity, seven kinds of intermediate in the BTR degradation would be mutagenic negative and developmentally nontoxic, while the parent compound was mutagenic positive and developmentally toxic. Moreover, compared with the LD50 of BTR (326.3 mg kg\(^{-1}\)), the toxicity of its products are clearly reduced. These results indicated that the bioaccumulation toxicity, mutagenicity, developmental toxicity, and acute toxicity of BTR can be effectively decreased and weakened.

Based on the above discussion, a mechanism diagram and the corresponding ROS chain reactions for BTR degradation by MoS\(_2\) piezoelectric-activated PMS were proposed (Eq. (3)–(12) and Scheme 2). In this process, MoS\(_2\) was triggered by the external hydromechanical energy from the water vortex to produce a piezoelectric potential to drive the transfer of free charges. These free electrons activated PMS to produce various reactive species, including \(\text{HO}^*\), \(\text{SO}_2\text{O}^*\), \(\text{O}_2^*\), and \(\text{O}_2^+\). Then, these powerful reactive species fragmented the BTR molecules into different intermediates, small molecule compounds, and CO\(_2\), H\(_2\)O and NO\(_3\) [21,48–52].

\[
\text{MoS}_2 + \text{vortex} \rightarrow \text{Piezo-potential (e}^- + \text{h}^+) \quad (3)
\]

\[
\text{HSO}_5^- + e^- \rightarrow \text{SO}_3^{2-} + \text{HO}^* \quad (5)
\]

\[
\text{SO}_4^{2-} + \text{H}_2\text{O} \rightarrow \text{HO}^* + \text{SO}_3^{2-} + \text{H}^+ \quad (6)
\]

\[
\text{O}_2 + e^- \rightarrow \text{O}_2^* \quad (7)
\]

\[
\text{H}_2\text{O} + \text{h}^+ \rightarrow \text{H}^+ + \text{HO}^* \quad (8)
\]

\[
2\text{HSO}_5^- + 2\text{h}^+ \rightarrow 2\text{SO}_3^- + 2\text{H}^+ + \text{O}_2 \quad (9)
\]

\[
\text{O}_2^* + \text{HO}^* \rightarrow \text{OH}^- + \text{O}_2 \quad (10)
\]
2O2− + 2H2O → O2 + H2O2 + 2OH− (11)
1O2/O2•−/HO•/SO4•− + BTR → intermediates + CO2 + H2O + NO3− (12)

4. Conclusions

The low frequency of hydromechanical energy from a water vortex works on piezoelectric MoS2 to generate piezoelectric potential-induced charges, which further effectively activate PMS for organic contaminants degradation. Upon the assistance of MoS2 in this water-vortex-driven piezo-activation PMS process, significant enhancements to organic contaminants degradation and PMS utilization were achieved which were greater than the sum of the contributions from individual MoS2, water vortex-, and piezocatalysis-PMS activation. The enhancements were attributed to the piezo-potential from MoS2 promoting the transfer of charges, thus more electrons participated in the activation reaction of PMS to generate powerful reactive oxygen species for organic contaminants degradation. Different reactive oxygen species, e.g., HO•, SO4•−, O2•−, and 1O2, were detected in the system while 1O2 was the dominant ROS. Finally, 15 less-harmful intermediates from BTR were identified and produced through two possible reaction pathways. This investigation provides a promising way to enhance the utilization efficiency of PMS as well as the decontamination of organic pollutants by using an environmentally friendly water vortex in environmental remediation.

Declaration of competing interest

The authors declare no competing financial interest.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.eese.2022.100165.

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