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Photocatalytic degradation of COVID-19 related drug arbidol hydrochloride by Ti$_3$C$_2$ MXene/supramolecular g-C$_3$N$_4$ Schottky junction photocatalyst

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**HIGHLIGHTS**

- A novel TiC/SCN Schottky junction photocatalyst was prepared by a simple method.
- 99.2% of ABLH was removed by the 0.5TiC/SCN under real sunlight, exceeding P25 TiO$_2$.
- The toxicities of the by-products decreased with the photocatalytic removal process.
- The superoxide radicals played a major role in the degradation of ABLH by 0.5TiC/SCN.

**GRAPHICAL ABSTRACT**

**ABSTRACT**

Because of the current COVID-19 outbreak all over the world, the problem of antiviral drugs entering water has become increasingly serious. Arbidol hydrochloride (ABLH) is one of the most widely used drugs against COVID-19, which has been detected in sewage treatment plant sediments after the COVID-19 outbreak. However, there has been no report on the degradation of ABLH. In order to remove ABLH we prepared a novel photocatalyst composed of Ti$_3$C$_2$ MXene and supramolecular g-C$_3$N$_4$ (TiC/SCN) via a simple method. The properties of the material were studied by a series of characterizations (SEM, TEM, EDS, XRD, FTIR, UV-vis, DRS, XPS, TPC, PL, EIS and UPS), indicating the successful preparation of TiC/SCN. Results show that 99% of ABLH was removed within 150 min under visible light illumination by the 0.5TiC/SCN (containing 0.5% of TiC). The performance of 0.5TiC/SCN was about 2.66 times that of SCN resulting from the formation of Schottky junction. Furthermore, under real sunlight illumination, 99.2% of ABLH could be removed by 0.5TiC/SCN within 120 min, which was better than that of commercial P25 TiO$_2$. The pH, anions (NO$_3^-$ and SO$_4^{2-}$) and dissolved organic matter (fulvic acid) could significantly affect the ABLH degradation. Moreover, three possible degradation pathways of ABLH were proposed, and the toxicities of the corresponding by-products were less toxic than ABLH. Meanwhile, findings showed that the superoxide radicals played a major role in the photocatalytic degradation of ABLH by 0.5TiC/SCN. This study provides a well understanding of the mechanism of ABLH degradation and provides a valuable reference for the treatment of ABLH in water.

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1. Introduction

The COVID-19 outbreak has caused severe harm to human society and further affected the world in various ways (Ahmed et al., 2021). During the epidemic period, the residual drugs used to control and prevent the COVID-19 are directly discharged into sewers, causing an increase of these drugs in the natural environment (Chen et al., 2021; Reinstadler et al., 2021). Arbidol hydrochloride (ABLH) is one of the effective pharmaceuticals for COVID-19 treatment and has been widely used in the clinic (Deng et al., 2020; Zhu et al., 2020). However, the traditional water treatment technology can not altogether remove these drugs. A study has shown a high ABLH content in the sewage treatment plants samples collected in Arkhangelsk (Ulyanovskii et al., 2022). Moreover, many other COVID-19-related antiviral drugs have also been detected in the natural waters (Reinstadler et al., 2021). These drugs are assessed as high ecotoxicological risk because they may cause potential harm to the environment even at low levels (Kuroda et al., 2021). To our knowledge, ABLH has never been detected in sewage treatment plant sediments until the COVID-19 outbreak; in particular, there has been no report on its degradation. Therefore, deep investigations on the ABLH removal and degradation are needed.

As an appealing method, photocatalysis is considered as an appropriate way to settle this issue (Yang et al., 2020). Previous research has shown that graphic carbon nitride (CN) was a photocatalyst with potential because of its high stability and nontoxicity (Mamba and Mishra, 2016; Sun et al., 2021; Xing et al., 2021). However, fewer surface active sites and higher carrier recombination rate impede the performance of bare CN (Wang et al., 2019; Raziq et al., 2020). Accordingly, various methods have been developed to improve the catalytic ability of CN (Zheng et al., 2014; Ma et al., 2015; Zhao et al., 2020; Oseghie et al., 2021). Among many techniques, supramolecular self-assembly is a convenient method to enhance the properties of CN and has been extensively applied to prepare CN (Li et al., 2018a). Moreover, doping is a widely used and effective method to strengthen photocatalyst activity (Jiang et al., 2017). However, high-performance cocatalysts are usually precious metals whose high-cost limits their wide application (Cheng et al., 2013).

As novel two-dimensional (2D) materials, transition-metal carbides and nitrides (MXenes) are regarded as ideal materials in various fields owing to their outstanding characteristics (Ghidiu et al., 2014; Ran et al., 2017; Pang et al., 2019). As a typical MXene, Ti$_3$C$_2$ MXene is an excellent cocatalyst to improve photocatalyst performance and is an appropriate way to construct a Schottky junction to inhibit the photogenerated carriers’ recombination (Yang et al., 2019). Thus, TiC has been used as an excellent cocatalyst to improve photocatalyst performance and is expected to have a bright future in photocatalysis (Cheng et al., 2020; Xiao et al., 2020; Zhou et al., 2021).

The intention of this paper is to explore the method for the removal of ABLH in water; in particular to investigate the degradation mechanism of ABLH. In this study, we prepared a novel Ti$_3$C$_2$ MXene/supramolecular g-C$_3$N$_4$ (TiC/SCN) photocatalyst with Schottky junction. The photocatalytic degradation of ABLH by TiC/SCN was evaluated under visible light (VL) illumination. The influences of pH, ion type, dissolved organic matter and concentration on the degradation of ABLH were studied. Meanwhile, the possible degradation pathway of ABLH was explored, and the corresponding photocatalytic mechanism was proposed. It is hoped that the findings presented here can provide some reference for the solution to the reduction of the environmental pollution caused by drugs used in large quantities during the COVID-19 epidemic.

2. Experimental section

2.1. Preparation of materials

2.1.1. Preparation of CN

Bulk CN was obtained using a previously developed method (Jin et al., 2022). In brief, the melamine was pyrolyzed at 550 °C for 4 h (heating rate: 5 °C min$^{-1}$).

2.1.2. Preparation of SCN

Firstly, 4.9 g of melamine and 5.0 g of cyanoacid were mixed with 50 mL of ethanol. After being stirred for 4 h, the mixture was dried at 60 °C for 2 h, and then annealed at 550 °C for 4 h with a heating rate of 5 °C min$^{-1}$. Finally, the product was cooled and SCN was obtained.

2.1.3. Preparation of ultrathin TiC

Ultrathin TiC was prepared as described by the previous study (Cao et al., 2018). Firstly, 1 g of Ti$_3$AlC$_2$ was dispersed in 20 mL of HF solution ($\geq$40.0%). After being stirred for 48 h, the suspension was then centrifuged at 8000 rpm, followed by washing with deionized water and ethanol. After drying at 60 °C for 2 h, 0.5 g of the sample was mixed with 20 mL of ethanol, followed by sonicating for 2 h. After centrifugation at 3500 rpm for 10 min, the ultrathin TiC supernatant was finally obtained with a concentration of 0.837 mg/mL.

2.1.4. Preparation of TiC/SCN

Firstly, 100 mg of SCN was added in 20 mL of ethanol, followed by sonicating for 1 h. Afterwards, a portion of TiC solution was added, and then sonicated for 2 h and stirred for 12 h. The mass ratios of TiC to SCN were set as 0.25, 0.5, and 1 wt% by changing the added TiC solution volume, named 0.25TiC/SCN, 0.5TiC/SCN, and 1TiC/SCN, respectively. After centrifugation at 10,000 rpm and drying, the TiC/SCN samples were obtained.

Fig. 1. (a) XRD patterns, (b) FTIR spectra, and (c) UV–vis DRS spectra of the as-prepared materials.
The prepared materials were fully characterized, please see supporting information for details.

2.3. Photocatalytic ABLH degradation

During each test, 50 mL of ABLH solution with a concentration of 10 mg/mL was added to a vial, and 5 mg of TiC/SCN was added. Then the suspension was stirred in the dark for 30 min to achieve the sorption-desorption equilibrium. Subsequently, the suspension was irradiated with VL (A 300 W Xe lamp (420 nm < λ < 780 nm)), and 2 mL of suspension was taken out and filtered at a specific time interval. The pH (5, 7 and 9), concentration was measured by high-performance liquid chromatography (HPLC), as detailed in supporting material. The pH (5, 7 and 9), changes in ABLH concentration was measured by high-performance liquid chromatography (HPLC), as detailed in supporting material. The pH (5, 7 and 9), concentration was measured by high-performance liquid chromatography (HPLC), as detailed in supporting material.

The morphologies of the materials were studied by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Fig. 2a showed that TiC presented a stacked multilayer structure. The SEM image of SCN is given in Fig. 2b; bare CN exhibited a typical layer-like structure. The TEM image of 0.5TiC/SCN is presented in Fig. 2c. The SEM image of 0.5TiC/SCN is given in Fig. 2f. C, N and Ti elements were evenly dispersed on 0.5TiC/SCN, confirming the successful formation of TiC/SCN. The N2 adsorption-desorption isotherms of samples are given in Fig. S1. The specific surface area of SCN was calculated to be 23.51 m²g⁻¹, almost twice that of CN (12.48 m²g⁻¹). After adding TiC, the specific surface area of 0.5TiC/SCN slightly increased, and was calculated to be 24.21 m²g⁻¹. The pore volumes about CN, SCN and 0.5TiC/SCN were calculated to be 0.038, 0.069 and 0.074 cm³g⁻¹.
The elemental composition and electronic state of the material were usually investigated by X-ray photoelectron spectroscopy (XPS). As revealed in Fig. 3a, the C, N and O peaks corresponding to SCN could be detected in the curve of 0.5TiC/SCN. However, no peak corresponding to Ti could be detected, due to the low content of TiC. However, Ti element could be observed in the XPS element information of 0.5TiC/SCN (Table S2), indicating the presence of TiC in the 0.5TiC/SCN sample. As presented in Fig. 3b, two peaks could be seen at 288.00 and 284.55 eV in the C1s spectrum of SCN, owing to the vibrations of N–C=N and C–C respectively (Yang et al., 2020). Four peaks in Fig. 3c at 398.50, 399.50, 400.80 and 404.00 eV in the N 1s spectrum of SCN, corresponding to N–C=N, N-(C)=, C–N–H oscillations, and the charging effects in heterocycles, respectively (He et al., 2020). As given in Fig. 3d, in the Ti 2p spectrum of TiC five peaks were detected at 454.55, 455.90, 458.40, 460.40 and 464.00 eV. The peaks at 454.55 and 460.40 eV were related to Ti–C. The peaks at 458.40 and 464.00 eV were due to Ti–O. The peak at 455.90 eV was assigned to TiO (Cao et al., 2018; Yuan et al., 2021). Compared with bare SCN and TiC, the binding energies of C 1s and Ti peaks in 0.5TiC/SCN exhibited positive and negative shifts, respectively, indicating that electrons could transfer from SCN to TiC in

![Figure 3](image1)

**Fig. 3.** XPS spectra of (a) total survey, (b) C 1s, (c) N 1s and (d) Ti for the as-prepared materials.

![Figure 4](image2)

**Fig. 4.** (a) TPC responses, (b) PL spectra, and (c) EIS Nyquist plots of the materials.
0.5TiC/SCN due to the Schottky effect.

Fig. S2 shows the ultraviolet photoelectron spectrometer (UPS) spectra of the materials. The cutoff energies \((E_{\text{cutoff}})\) of TiC, SCN, and 0.5TiC/SCN were calculated to be 17.14, 17.68 and 17.55 eV, while their fermi levels \((E_F)\) were set to be 0 eV in order to calibrate the instrument. The work functions \((\Phi)\) of TiC, SCN, and 0.5TiC/SCN were calculated to be 4.08, 3.54 and 3.67 eV according to \(\Phi = h \nu - E_{\text{cutoff}} - E_F\) (Li et al., 2018b). The \(\Phi\) of TiC was higher than that of SCN, and the \(\Phi\) of 0.5TiC/SCN exhibited an uplift of 0.13 eV compared with SCN. The \(\Phi\) is the difference between the vacuum level and the \(E_F\). According to the conversion formula \(E_{\text{vac}} = - 4.5 - \Phi_{\text{NHE}}\), the actual \(E_F\) of TiC and SCN were calculated to be –0.42 and –0.96 eV, respectively. The \(E_F\) of SCN was more negative than that of TiC, indicating the e⁻ would transfer from SCN to TiC. These results demonstrated that a Schottky junction had been successfully established between TiC and SCN.

Fig. 4a presents transient photo-current (TPC) response curves of the as-prepared materials. Compared with CN, SCN exhibited an improved photo-current response, suggesting the separation efficiency of electrons (e⁻) and holes (h⁺) on SCN was higher than that on CN. The 0.5TiC/SCN displayed a stronger response than SCN, indicating that the successful formation of the Schottky junction between TiC and SCN extended the lifetime of the e⁻-h⁺ pair. Therefore, the migration of e⁻ was studied by electrochemical impedance spectroscopy (EIS) measurements. As given in Fig. 4c, the semicircle diameter of SCN was shorter than that of CN, suggesting that SCN possessed a weaker electron transfer resistance than CN. In addition, 0.5TiC/SCN exhibited a reduced semicircle diameter compared with SCN, suggesting the separation and migration efficiency of e⁻ on 0.5TiC/SCN was better than that on SCN. These results further confirmed that a Schottky junction had been successfully established between TiC and SCN.

The production of reactive oxygen species (ROSs) over the as-prepared materials under VL irradiation was evaluated by the electron spin resonance (ESR) technology. Fig. S3 showed that 0.5TiC/SCN exhibited the strongest signal regarding DMPO-•O₂ and DMPO-•OH adducts because of the enhanced charge separation, which could promote ROSs production.

### 3.2. Photocatalytic removal for ABLH

The photocatalytic degradation of ABLH under VL illumination by the materials was investigated. The ABLH concentration was almost unchanged by VL irradiation for 30 min without any photocatalyst, meaning that ABLH would not spontaneously undergo photolysis (Fig. S4). The photocatalytic ABLH removal efficiencies by different materials are given in Fig. 5a. The bare TiC exhibited the lowest removal efficiency, and only 10% of ABLH was removed. SCN showed an enhanced performance for ABLH degradation, about twice that of CN. The catalytic activity of materials for ABLH degradation was significantly improved by compounding TiC with SCN. The 0.5TiC/SCN presented the best performance, and ABLH could be completely degraded within 150 min. Furthermore, the 0.5TiC/SCN exhibited the highest ABLH degradation rate (0.02959 min⁻¹) (Fig. 5b), which was about 2.66 times that of SCN (0.01111 min⁻¹). The performance of 0.5TiC/SCN for ABLH degradation was further evaluated by total organic carbon (TOC) content as a measure of ABLH mineralization. As presented in Fig. 5c, more than 37.9% of TOC was removed by 0.5TiC/SCN under VL irradiation within 150 min. What is more, even under real sunlight illumination (time: 12/10/2021, 8:30–10:30 a.m.; average light intensity: 79.6 mW/m²), 99.2% of ABLH could be removed by 0.5TiC/SCN within 120 min, which was better than that of commercial P25 TiO₂ (Fig. S5). These results indicated that 0.5TiC/SCN might be used in practical water treatment.

The effects of pH, ions and concentrations on ABLH degradation by 0.5TiC/SCN were also studied. As displayed in Fig. S6a, the ABLH removal efficiency of 0.5TiC/SCN was improved significantly at pH = 5. It is thought that the photocatalytic performance of photocatalysts is related with its sorption capacity for pollutants. As displayed in Fig. S6b, the adsorption capacity of 0.5TiC/SCN for ABLH was pH-dependent, and was strengthened at pH = 5, which might be attributed to the change in the surface charging properties of materials and pollutants affected by pH, thus affecting the adsorption interaction between materials and pollutants. Therefore, the improved photocatalytic performance of 0.5TiC/SCN at pH = 5 could be attributed to ABLH being more easily absorbed by 0.5TiC/SCN in acidic conditions. However, after the pH was adjusted to 9, the content of ABLH decreased to about one third of the original before adsorption and illumination, which might result from the easy hydrolysis of ABLH under alkaline conditions. The effects of different anions on ABLH removal are exhibited in Fig. S7a. The performance of 0.5TiC/SCN was inhibited after adding NaNO₃ or Na₂SO₄, because NO₃⁻ and SO₄²⁻ could react with h⁺ to form NO₂⁻ and SO₄²⁻, respectively (Guo et al., 2020). Results in present study showed that Cl⁻ seemed to have no effect. Although previous studies have shown that Cl⁻ could react with •OH to form Cl• (Zhang and Parker, 2018), the •OH played only a minor role in the ABLH degradation by 0.5TiC/SCN (proved in latter). The influence of dissolved organic matter on photocatalytic degradation of ABLH was also studied, and the fulvic acid was selected as a representative. The result is exhibited in Fig. S7b. The performance of 0.5TiC/SCN for ABLH degradation was inhibited after adding fulvic acid, which might be due to the competition between fulvic acid and ABLH for ROSs. The effect of ABLH concentration on its degradation is exhibited in Fig. S7c. At concentrations of 5, 10 and 20
mg/L, 99% of ABLH could be completely removed by 0.5TiC/SCN within a certain time. The results showed that ABLH in the concentration of 5–20 mg/L could be removed completely by 0.5TiC/SCN photocatalyst. Moreover, cycling tests were conducted to evaluate the stability of 0.5TiC/SCN, and the results are given in Fig. S8. After four consecutive cycles, the removal efficiency of ABLH by 0.5TiC/SCN decreased from 99.1% to 96.3% within 150 min. The XRD, XPS, and FTIR results of the used 0.5TiC/SCN are given in Fig. S9. There was little difference between the as-prepared and recycled 0.5TiC/SCN. These results further indicated that 0.5TiC/SCN was stable and could be used in practice. The performance of the recovered 0.5TiC/SCN was slightly lower than that of the fresh, thereby owing to the inevitable loss in the recovery process and the residue of pollutants on materials.

3.3. Mechanism for photocatalytic degradation of ABLH

The degradation pathway of ABLH was studied by HPLC-MS (Fig. S10) and frontier electron density (FED) calculation (Table S3). In theory, the position with a large $2F_{\text{HOMO}}$ value is prone to react with oxidant, while the position with a large $F_{\text{HOMO}} + F_{\text{LUMO}}$ value is prone to react with ROS (Jiang et al., 2020). Accordingly, N1, C4, C5, C6, C7, C8, C9 and O22 were easy to react with the oxidant, while N1, C2, C3, C5, C6, C8, C9, C10, S14 and O22 were easy to react with ROSs. The degradation pathway of ABLH is given in Fig. 6d. In path I, ABLH underwent bond breaking to generate A. Then, A was transformed to F by hydrolysis and oxidation. In pathway II, ABLH could also be converted to B by hydrolysis and oxidation. Subsequently, B underwent substitution and bond breaking, forming G. In pathway III, ABLH was transformed to C, D and E via bond breaking and hydrolysis. D was further oxidized and converted to E and H, while E underwent deamination and was converted to I. In addition, the acute and chronic toxicities of by-products to fish, daphnia, and green algae were predicted by the QSAR model of ECOSAR program. As shown in Table S4, the acute and chronic half-lethal concentrations of all by-products were higher than those of ABLH, indicating that all by-products’ acute and chronic toxicities were lower than those of ABLH. The result indicated that photocatalysis was an environmentally friendly and effective method to remove ABLH.

The results of trapping tests are displayed in Fig. S11, $•\text{O}_2^-$, h', $\text{O}_2$, $\text{H}_2\text{O}_2$ and $•\text{OH}$ were captured by TEMPOL, sodium oxalate, l-histidine, catalase and TBA, respectively. In the absence of $•\text{O}_2^-$, the ABLH removal efficiency by 0.5TiC/SCN declined dramatically from 99.2% to 39.0%. Moreover, in the absence of the $\text{O}_2$, h' and $\text{H}_2\text{O}_2$, the ABLH removal efficiency by 0.5TiC/SCN decreased from 99.2% to 80.8%, 88.4% and 92.2%. However, the effect of the absence of $•\text{OH}$ on the degradation of ABLH seemed negligible. These results indicated that $•\text{O}_2$ played an essential role and $•\text{OH}$ played a minimal role in ABLH degradation by 0.5TiC/SCN.

Fig. 6. (a) The structure of ABLH; (b) The HOMO and (c) LUMO of ABLH calculated by Gaussian 09 program at the B3LYP/6-31+g(d, p) level; (d) Possible degradation pathways of 0.5TiC/SCN for ABLH.
3.4. The mechanism for enhanced photocatalytic performance

Based on the above discussion and characterization results, the possible mechanism of TiC/SCN Schottky junction is displayed in Fig. 7. The band gap energy (Eg) of SCN was calculated to be 2.73 eV (Fig. S12a). As given in Fig. S12b, the valence band energy (EVB) of SCN was calculated to be 1.74 V. Therefore, the conduction band energy (ECB) of SCN was calculated to be ~0.99 V, which was more negative than the redox potential of O2/•O2 (−0.33 V versus NHE), suggesting that reduction of O2 could form •O2 and H2O2. The EVB of SCN was more negative than the redox potential of OH−/•OH (1.99 V versus NHE) and H2O2/O2•−OH (2.37 V versus NHE), indicating that the direct generation of •OH was infeasible. Therefore, •OH was produced by the further reduction of H2O2. In addition, the oxidation of •O2 with h+ produced 1O2. According to the previous results of UPS, the eφ of TiC (4.08 eV) was higher than that of SCN (3.54 eV); thus, e− would transfer from SCN to TiC to achieve the balance of Fermi level (Eg) when TiC was in contact with SCN. As a result, the space charge layer was formed on the side of SCN, which resulted in the upward curving of energy band and the formation of a Schottky barrier (φSB). Thereby the production of ROSs was promoted. And then the photocatalytic performance of 0.5TiC/SCN was enhanced (Yang et al., 2019). Therefore the reaction formulas for ABLH degradation are proposed as follows:

\[0.5 \text{TiC} / \text{SCN} + h\nu \rightarrow e^- + h^+\]  
(1)

\[e^- + \text{O}_2 \rightarrow • \text{O}_2\]  
(2)

\[• \text{O}_2 + h^+ \rightarrow 1\text{O}_2\]  
(3)

\[• \text{O}_2 + 2\text{H}^+ + 2e^- \rightarrow \text{H}_2\text{O}_2\]  
(4)

\[\text{O}_2 + 2\text{H}^+ + 2e^- \rightarrow \text{H}_2\text{O}_2\]  
(5)

\[\text{H}_2\text{O}_2 + e^- \rightarrow • \text{OH} + \text{OH}^-\]  
(6)

\[\text{ROSs} + \text{ABLH} \rightarrow \text{CO}_2 + \text{H}_2\text{O}\]  
(7)

4. Conclusion

In this study, in order to remove ABLH from water we have prepared a novel TiC/SCN photocatalyst with a Schottky junction by a simple method. The recombination of e−−h+ was effectively restricted due to the Schottky effect. 99% of ABLH can be removed within 150 min under UV illumination by the 0.5TiC/SCN. Furthermore, even under real sunlight illumination, 99.2% of ABLH could be removed by 0.5TiC/SCN within 120 min, which was better compared with the commercial P25 TiO2. The pH and anions (NO3− and SO42−) could significantly affect the ABLH degradation. In acidic conditions the removal efficiency of ABLH by 0.5TiC/SCN was improved. And the performance of 0.5TiC/SCN was inhibited after adding NaN3 or Na2SO4 because of reacting with h+. Moreover, three possible degradation pathways of ABLH were proposed, and the toxicities of the corresponding by-products were less toxic than ABLH. Meanwhile, results showed the significant contribution of superoxide radicals in the photocatalytic ABLH degradation by 0.5TiC/SCN. This work contributes to understand the photocatalytic ABLH degradation mechanisms and provides new references for the treatment of drugs in water.

Credit author statement

Dexin Jin: Conceptualization, Methodology, Investigation, Writing–original draft. Yihan Lv: Software & Formal analysis. Dongyang He: Data curation. Dongmei Zhang: Supervision, Funding acquisition, Writing–review & editing. Yue Liu: Validation. Tingting Zhang: Investigation. Fangyuan Cheng: Formal analysis. Yanan Zhang: Investigation. Jiaqiong Sun: Validation. Jiao Qu: Funding ac quisition.

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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.chemosphere.2022.136461.
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