

Perspective

Challenges and opportunities
in the selective degradation
of organophosphorus herbicide glyphosateLei Jin,¹ Yingping Huang,¹ Liqun Ye,¹ Di Huang,^{1,*} and Xiang Liu^{1,*}

SUMMARY

The wide and continuous usage of glyphosate in the environment poses a serious threat to biological systems. Besides the accumulation of glyphosate *in vivo*, a growing body of research has revealed that aminomethylphosphonic acid (AMPA), the main degradation intermediate of glyphosate, has significant environmental and biological influences by inducing chromosome aberration of fish and canceration of human erythrocyte. Therefore, the development of new strategies avoiding the generation of the toxic AMPA intermediate during the full degradation of glyphosate is becoming of high importance. Herein, we provide a mini-review that includes the most recent advances in the selective degradation of glyphosate avoiding the generation of AMPA in the last several years from 2018. The developments of the selective degradation of glyphosate, highlighting its synthesis and selective degradation mechanism, are summarized here. This review intends to attract more attention from researchers toward this area and to emphasize the recent developments of selective degradation of glyphosate in highlighting future challenges.

INTRODUCTION

In 1950, N-(phosphonomethyl)glycine was first synthesized by the Swiss chemist Henri Martin, at Cilag pharmaceutical company, but it was not found to be useful at the time.¹ In 1970, Hamm and Franz discovered its excellent herbicidal activity and named it glyphosate.² The herbicidal activity of glyphosate is attributed to its inhibition of shikimic acid pathway, which breaks the photosynthesis of plants, thereby achieving the effect of herbicide.³ Since there is no shikimic acid pathway in animals and humans, glyphosate was originally considered as a completely non-toxic herbicide.⁴

To date, glyphosate has been used as a broad-spectrum herbicide for more than 50 years and is registered for use in more than 130 countries.⁵ However, the widespread use of glyphosate in the environment poses a serious threat to biological systems.⁶ The main degradation intermediate of glyphosate (including the natural degradation and advanced oxidation process), aminomethylphosphonic acid (AMPA), which shows higher stability than glyphosate, is widely found in plant sediments, surface, and groundwater.⁷ In addition, the toxicity of AMPA has also recently been found to be higher than that of glyphosate. A growing body of research has revealed that glyphosate and AMPA may induce chromosome aberration of fish and canceration of human erythrocyte.⁸ For instance, in 2022, Cheng and Zhang's group found that glyphosate could affect the all-round development of zebrafish embryos, including decreased body length, hatching abnormalities, and mortality (Figure 1).⁹ With continued exposure to glyphosate, zebrafish embryos show cardiac malformations, including rhythm disturbances, enlarged chambers, and thinned ventricular walls. In 2015, glyphosate was categorized by the International Agency for Research on Cancer as "carcinogenic to humans."¹⁰ As a new pollutant, the environmental content of glyphosate and its degradation process in wastewater treatment have aroused wide public concern.^{11–15} Degradation strategies, adopted to avoid the formation of AMPA during the full degradation of glyphosate, need to be developed urgently. Herein, we offer a review that includes the most recent advances in the selective elimination of glyphosate by avoiding the formation of toxic AMPA intermediate, summarizing developments in the selective degradation of glyphosate and highlighting its synthesis and selective degradation mechanism in last several years from 2018. The review intends to introduce the fundamental principle and recent developments concerning the selectivity of degradation of glyphosate, to highlight future challenges, and to attract more attention from researchers toward this interesting area.

Synthesis of glyphosate

Twenty years after Martin's discovery,¹⁶ in 1970, the Monsanto company reported its own method for the synthesis of glyphosate.¹⁷ So far, there are four main synthetic methods for glyphosate (Figure 2).¹⁸

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<https://doi.org/10.1016/j.isci.2024.110870>



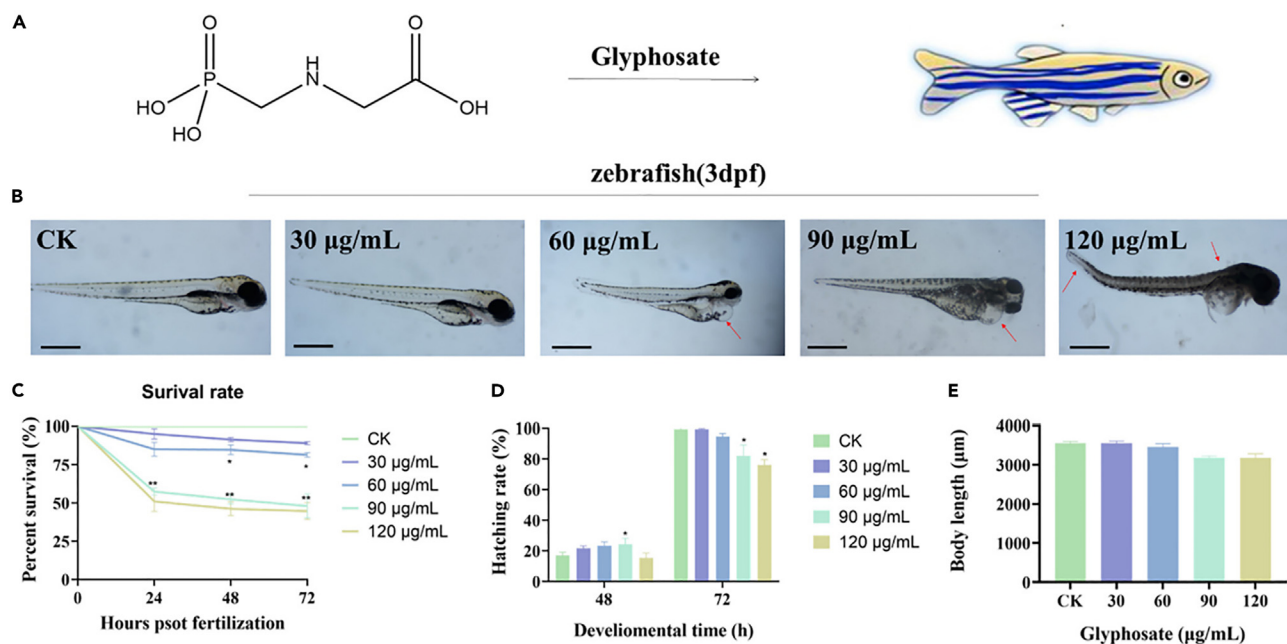


Figure 1. Glyphosate induced developmental delay in zebrafish embryo/larvae

(A–E) Schematic diagram of toxicity test in zebrafish embryo (A). Morphology (B), survival rate (C), hatching rate (D), and body length (E) of zebrafish that were exposed to different concentrations of glyphosate. Error bars represent mean \pm SD for three replicates ($n = 3$). p value: **, $p < 0.01$; and *, $p < 0.05$. Reproduced with permission.⁹ Copyright 2022, Elsevier B.V.

First, aminoacetic acid (glycine) was widely applied as the *N*-containing starting material for glyphosate synthesis at all stages, because its secondary reactions are easily controllable and predictable.¹⁹ Second, glyphosate was synthesized by the oxidation of *N*-(phosphonomethyl)-iminodiacetic acid, which was obtained from ethylene oxide and ammonia (DEA process).^{20–23} Third, glyphosate was also formed by the oxidation of *N*-(phosphonomethyl)-iminodiacetic acid, but it was synthesized from hydrocyanic acid and formaldehyde.^{24–26} Fourth, glyphosate was synthesized, via an atom-efficient method, by dealkylation of *N*-substituted glyphosates.^{27–29}

Despite the advances of synthetic methods for glyphosate, most of them require strong oxidants, anhydrous conditions, and multi-steps. Therefore, it is of high importance to explore simpler, greener, and more efficient methods for the synthesis of glyphosate.

Degradation pathways of glyphosate

Based on the different reactions of bond breaking, degradation pathways of glyphosate (including biodegradation,^{30–42} photocatalysis,^{3,43–48} Fenton,⁴⁸ electrocatalysis,⁴⁹ and advanced oxidation process^{50–57}) are divided into the following three types (Figure 3).

Selective degradation of glyphosate

So far, there are only a few studies on selective elimination of glyphosate avoiding the formation of the toxic AMPA intermediate for the safe and efficient degradation of glyphosate. Among them, adsorption is a very simple and direct treatment technology. For example, in 2018, Jing's group firstly investigated the selective adsorption of glyphosate on goethite's surface at the molecular level by attenuated total reflection fourier transformed infrared spectroscopy (ATR-FTIR), two-dimensional correlation spectroscopy (2D-COS), and density functional theory (DFT) study. They found adsorption of the phosphonate group of glyphosate on goethite's surface via the formation of inner-sphere complexes with numerous configurations under different pH conditions. Specifically, the adsorption of phosphonate group by mononuclear bidentate without H^+ and binuclear bidentate occurs at $pH = 5$, whereas the adsorption mode is binuclear bidentate without H^+ and mononuclear monodentate with H^+ at $pH = 6–8$. The adsorption mode becomes mononuclear monodentate without H^+ at $pH = 9$ (Figure 4).⁶⁷ Later, Martínez's group confirmed that the phosphonate group of glyphosate preferentially interacted with goethite via the formation of inner-sphere monodentate complexes, whereas the phosphonate group of glyphosate preferentially interacted with goethite via the formation of bidentate configurations (Figure 5).⁶⁸ Adsorption is a very simple and direct treatment technology to avoid the formation of toxic intermediate AMPA, but the adsorbent is often difficult to regenerate and has no selectivity. Although adsorption does not fundamentally degrade glyphosate,^{69–72} these studies provide an idea to regulate the surface of catalysts for selective adsorption of glyphosate. As long as the catalyst is designed to preferentially adsorb the phosphate group of glyphosate,^{73–75} it could be activated *in situ* to split the C-P/C-N bond at the terminal phosphate group, take the route of sarcosine and glycine intermediates, and avoid the generation of the toxic intermediate AMPA. In this direction, in 2018, Jaisi's group reported the selective

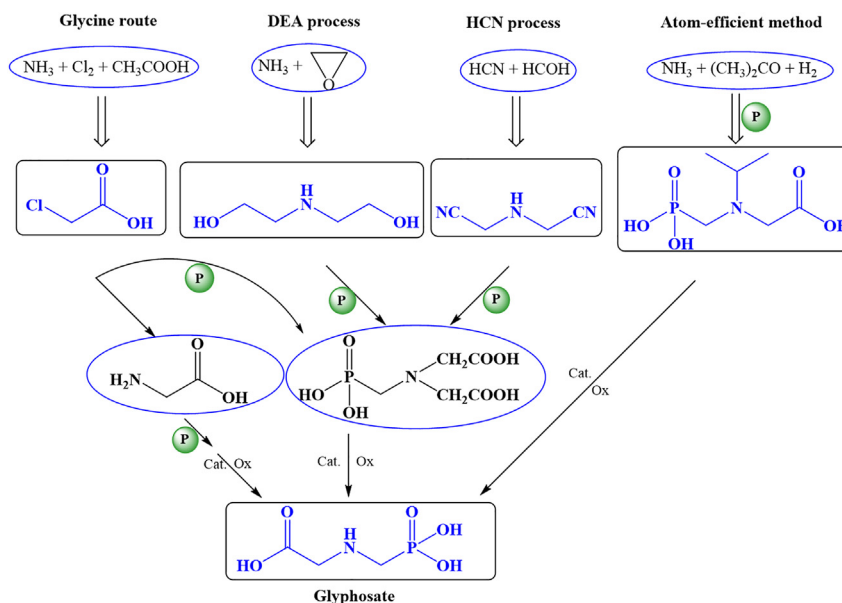


Figure 2. Synthesis methods of glyphosate

elimination of glyphosate firstly, via the cleavage of C-N bond, catalyzed by birnessite to form methylphosphonic acid, sarcosine, and phosphoric acid with the presence of high concentrations of NaCl. Due to the ionization effect of Cl⁻, the C-N bond at the terminal glycine group of glyphosate was found to preferentially interact with the Na-O sites of birnessite by DFT calculations (Figure 6).⁷⁶ This results in the preferential coordination and activation of Mn(IV) and C-P/C-N bond at the terminal phosphate group of glyphosate, thereby avoiding the formation of the toxic intermediate AMPA. In 2021, our group reported the selective photocatalytic degradation of glyphosate catalyzed by fluorinated TiO₂ for avoiding the formation of the toxic intermediate AMPA.⁷⁷ F⁻ weakens the coordination performance and electronegativity of Ti-O bond on the surface and sublayer of TiO₂, thereby resulting in the reduction of its ability to coordinate with the C-N bond at the terminal glycine group of glyphosate. The C-P/C-N bond at the terminal phosphate group of glyphosate could preferentially interact with the Ti-O bond on the surface of TiO₂. Besides the fluorinated TiO₂, our group also reported a new photocatalyst by doping F⁻ on BiVO₄, via the cleavage of (P)-C-N bond, for selective photocatalytic elimination of glyphosate, significantly reducing the toxic emissions of AMPA.⁷⁸ In the F-BiVO₄ system, glyphosate was degraded to glycine and phosphoric acid selectively, avoiding the formation of AMPA. Consequently, the surface regulation of catalysis, which could control the electrostatic attraction between reactants and surface active sites, was proven to be an effective strategy to evade the generation of the toxic intermediate AMPA. According to this principle, our group developed a BiVO₄/BiPO₄ photo-catalyst, the dipole of which can be easily regulated by pH in solution. With the pH-mediated electron cloud distribution of glyphosate simultaneously, the BiVO₄/BiPO₄ system had the ability to control the preponderant adsorption of glyphosate bonds. When the solution adjusted to alkaline, the

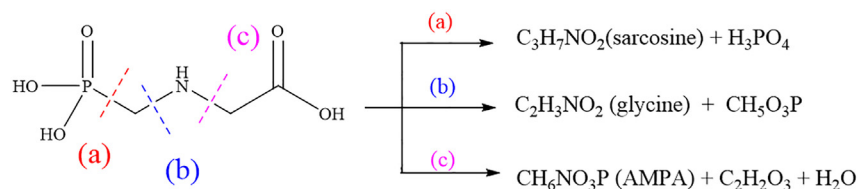


Figure 3. Degradation pathways of glyphosate

(a) Glyphosate was decomposed, via the cleavage of C-P bond at the terminal phosphate group, into non-toxic sarcosine and phosphoric acid.
 (b) Glyphosate was degraded, via the breakage of C-N bond at the terminal phosphate group, into non-toxic methylphosphonic acid and glycine. In fact, the first two glyphosate degradation pathways are the green ones because neither of them involves the formation of AMPA.⁵⁸⁻⁶²
 (c) Glyphosate was decomposed, via the cleavage of C-N bond at the terminal glycine group, into glyoxylic acid and highly toxic and more stable AMPA. Relevant studies showed that AMPA with high stability is the thermodynamically controllable degradation intermediate of glyphosate.⁶³⁻⁶⁵ Whether it is bio-degradation or advanced oxidation methods (such as photocatalysis, Fenton, electrocatalysis, and O₃ oxidation), the degradation of glyphosate is easily controlled by thermodynamics and generates a large number of more stable and highly toxic AMPA intermediate.⁶⁶ For example, the degradation was precisely along the toxic intermediate AMPA pathway under thermodynamic control in the photo-degradation of glyphosate.¹³ Therefore, it is still a challenge to develop the selective elimination of glyphosate by avoiding the formation of toxic AMPA intermediate for the safe and efficient degradation of glyphosate.

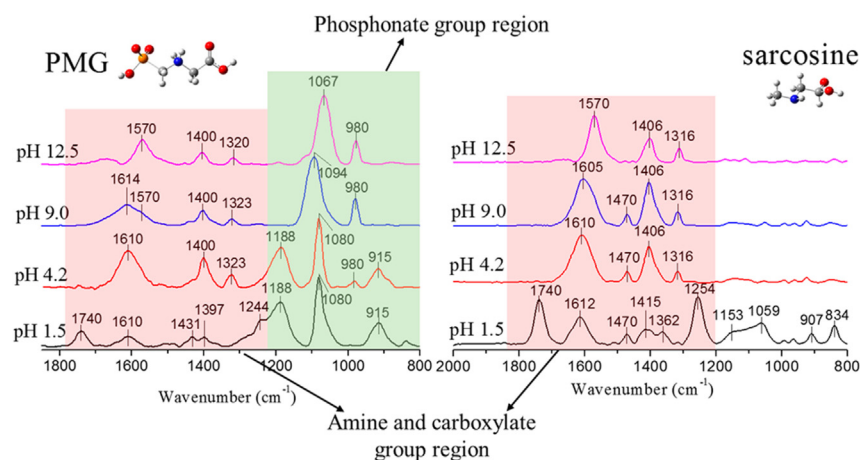


Figure 4. ATR-FTIR spectra

ATR-FTIR spectra of $1,000 \text{ mg L}^{-1}$ dissolved glyphosate and sarcosine in 0.1 M NaCl for pH values 1.5, 4.2, 9.0, and 12.5. Reproduced with permission.⁶⁷ Copyright 2018, American Chemical Society.

phosphonate and -NH- groups of molecular glyphosate was preferentially adsorbed on the $\text{BiVO}_4/\text{BiPO}_4$ surface, leading to the formation of sarcosine and orthophosphate, instead of AMPA, during glyphosate degradation by the selective cleavage of (P)-C-N bond.⁷⁹ In 2023, Planas' group also synthesized a thermal and hydrolytically stable mCB-MOF-2 material, which was synthesized from Zr(IV) and a tetracarboxylate carborane ligand, for selective photocatalytic degradation of glyphosate.⁶ To investigate the reaction mechanism of glyphosate on the surface of catalysis, DFT calculations, electron spin resonance analysis, and reactive oxygen species trapping were employed. They suggested the photocatalytic degradation mechanism of glyphosate following the selective adsorption of glyphosate and activation of C-N bond, subsequently. First, the electron-withdrawing carborane rendered the Zr(IV) clusters of mCB-MOF-2 more Lewis acidic,^{46,80,81} and the Lewis basic phosphonic group of glyphosate was selectively adsorbed and coordinated to Zr(IV) sites, which favored the activation of the C-P bond. Under UV-visible light, the photoexcited electrons were transferred into the conduction band (CB) at the surface of mCB-MOF-2, forming holes in the valence band (VB). The adsorbed or dissolved O_2 was reduced by the photogenerated electrons into $\bullet\text{O}_2^-$, which was further oxidized by photogenerated holes into $^1\text{O}_2$ (step II in Figure 7). Second, glyphosate was oxidized by $^1\text{O}_2$ into non-toxic orthophosphate and sarcosine (step III in Figure 7). Concurrently, $\bullet\text{OH}$ was generated from the oxidation of water by holes in the VB (step II in Figure 7). Then, the photogenerated nucleophilic $\bullet\text{O}_2^-$ and $\bullet\text{OH}$ species attacked the P-atom of glyphosate via an $\text{S}_{\text{N}}2$ -like substitution.⁸² Thus, orthophosphate was formed by the incorporation of the O-atom of the incoming nucleophile into the phosphate, and sarcosine was formed by hydrolysis of the carbon-centered radical.⁸³

In summary, the aim of selective degradation of glyphosate was successfully achieved by interfacially controlled engineering of the catalysts. Indeed, as long as the catalyst is designed to preferentially adsorb the phosphate group of glyphosate, it could be activated *in situ* to break the C-P/C-N bond at the terminal phosphate group. This strategy led glyphosate to take the route of sarcosine and glycine intermediates, which could avoid the generation of toxic intermediate AMPA, although the AMPA pathway is the thermodynamically controlled preferential degradation pathway for glyphosate.^{84–88}

CONCLUSION

As a new pollutant, glyphosate in the environment and its degradation in wastewater treatment plants have aroused wide public concern. In recent years, a growing body of research has revealed that glyphosate and AMPA may induce chromosome aberration of fish and canceration of human erythrocyte. Therefore, it is of high importance to develop selective degradation of glyphosate by avoiding the generation of the toxic AMPA intermediate for the safe and efficient degradation of glyphosate. Herein, we have provided a review that includes the most recent advances from recently published literature and some recent work from our groups on the selective glyphosate degradation by avoiding the formation of toxic AMPA intermediate, summarizing the developments on the selective degradation of glyphosate with a highlight on its synthesis and selective degradation mechanism. It is intended that researchers will be attracted to this promising area and will address future challenges.

Despite the pioneering advances in the design and exploration of selective degradation of glyphosate by avoiding the generation of the toxic AMPA intermediate, almost all the selective degradation of glyphosate were only efficacious in their separate catalytic systems. Hence, the development of novel, efficient, and universal catalytic systems for selective glyphosate degradation by avoiding the formation of toxic AMPA intermediate is still a severe challenge toward the treatment of actual wastewater for academic and industrial developments.

Another remarkable aspect involves how to selectively degrade glyphosate by avoiding the formation of the toxic intermediate AMPA in real sewage treatment. These catalytic systems are often seriously affected by background inorganic anions and natural organic matter in the treatment of actual wastewater.^{89–96}

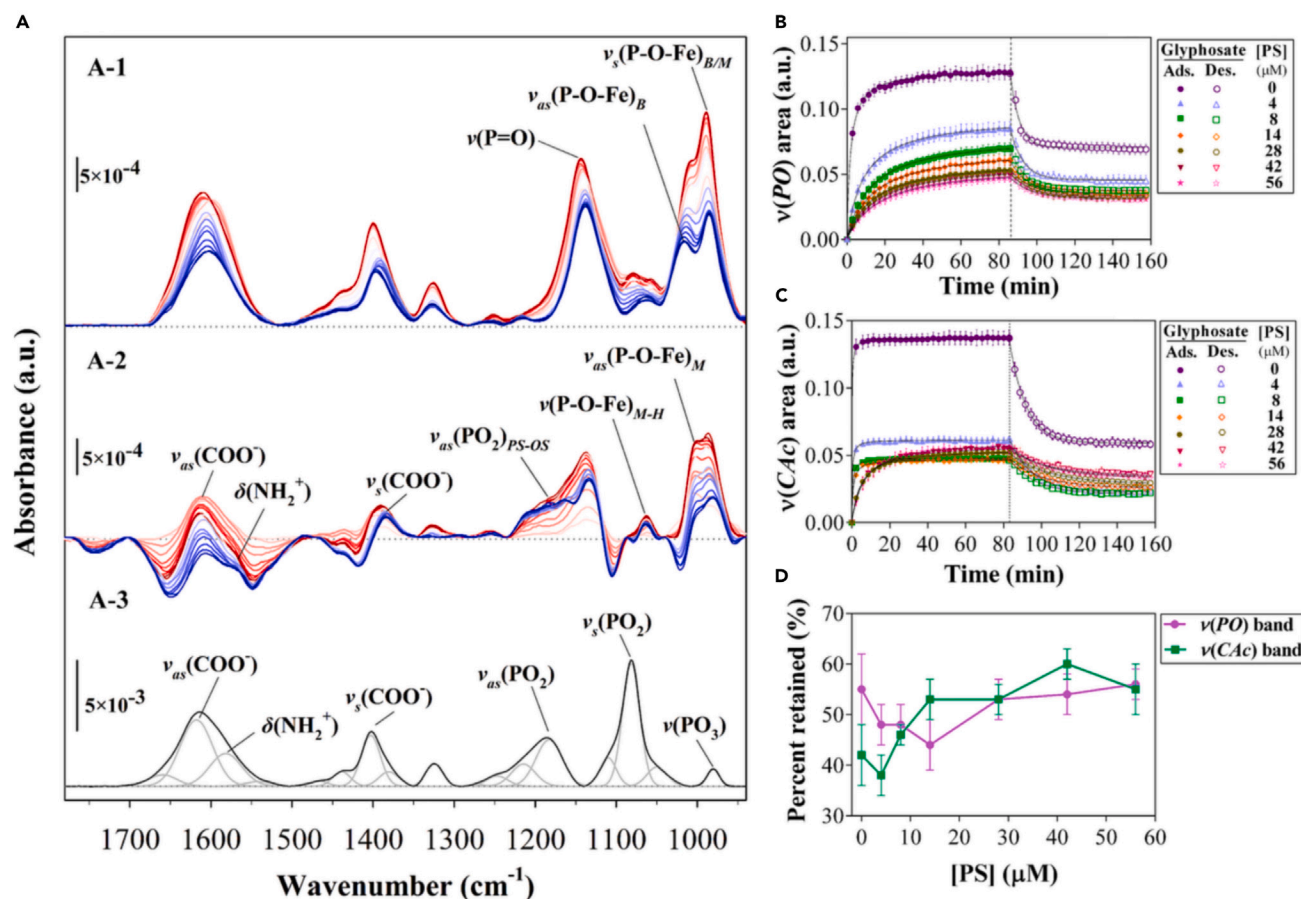


Figure 5. Characterization of glyphosate adsorption

(A–D) Representative time-resolved *in situ* ATR-FTIR spectra collected during glyphosate adsorption and desorption experiments at pH 5 on (A-1) goethite and (A-2) PS-goethite organo-mineral associations (OMAs) (polysaccharide [PS] = 14 μM). Red and blue lines indicate the evolution of interfacial spectra collected every ≈ 8 min (A-3) Solution state ATR-FTIR spectrum of 0.1 M glyphosate in 10 mM KCl at pH 5.0; gray lines show deconvoluted infrared (IR) components. Evolution of integrated (B) phosphonate ($v(\text{PO})$) and (C) carboxylate-amine ($v(\text{CAc})$) bands of glyphosate with increased PS surface loading. (D) shows the percentage of $v(\text{CAc})$ and $v(\text{PO})$ bands retained at the end of the desorption phase (percentage retained = $[A(\tilde{\nu})_{e,des}/A(\tilde{\nu})_{e,ads}] \times 100$). Reproduced with permission.⁶⁸ Copyright 2023 Elsevier Inc.

Limitations of the study

This review intends to attract more attention from researchers toward this area and to emphasize the recent developments of selective degradation of glyphosate in highlighting future challenges. Thus, future research should focus on the potential of trapping and mediated species that can be added to reactions to create direct chemical pathways for selective degradation.

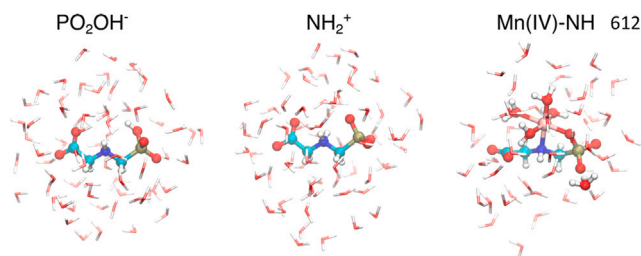


Figure 6. DFT calculations

Minimum energy structures of glyphosate in two of its zwitterionic states (i.e., PO_2OH^- and NH_2^+) and in the presence of manganese (Mn(IV)-NH) as predicted by DFT. Reproduced with permission.⁷⁶ Copyright 2018, American Chemical Society.

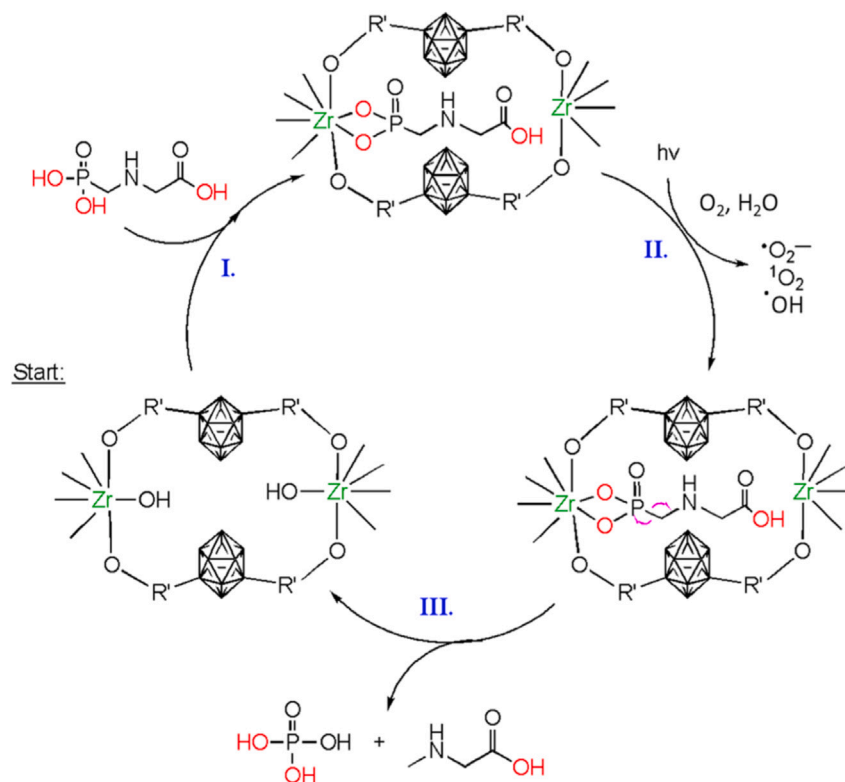


Figure 7. Glyphosate photodegradation cycle

Starting from an accessible pore of mCB-MOF-2: (I) glyphosate adsorption within the pore; (II) UV irradiation and photogeneration of reactive oxidation species; and (III) C-P lyase biomimetic photodegradation of glyphosate into sarcosine and phosphoric acid. Reproduced with permission.⁶ Copyright 2023 The Authors. Published by American Chemical Society.

ACKNOWLEDGMENTS

Financial support from the National Natural Science Foundation of China (Nos: 52470045, 22136003, 21972073, and 22076098) and the 111 Project (D20015) is gratefully acknowledged.

AUTHOR CONTRIBUTIONS

L.J.: investigation, data curation, and formal analysis; Y.H. and L.Y.: data curation; D.H. and X.L.: conceptualization, methodology, supervision, validation, funding acquisition, data curation, writing – original draft, writing – review and editing, and resources.

DECLARATION OF INTERESTS

The authors declare no competing interests.

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