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Synthetic effect of zero-valent iron and granular activated carbon on methane production from pentachlorophenol wastewater

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ABSTRACT

The main aim of this study was to investigate the effects and mechanisms of zero-valent iron (ZVI) and granular activated carbon (GAC) on biological degradation of pentachlorophenol (PCP) for methane production. In the bath tests, the optimal dosage of ZVI was determined to be 200 mg/L under the combined action of GAC. Combination addition of ZVI and GAC (ZVI/GAC) was more conducive to PCP degradation and chemical oxygen demand removal than that of single ZVI addition, corresponding removal efficiencies were 79.58% and 31.38%, respectively. The kinetic study was showed that ZVI/GAC addition could enhance PCP degradation rate in anaerobic reactor. Furthermore, continuous flow experiments were also conducted in three two-phase anaerobic reactors. The results indicated that the operating performances including operational stability and methane production of PCP wastewater were enhanced by ZVI or ZVI/GAC, and the average methane production reached 1241.5 mL and 1482.3 mL, respectively. Degradation pathway analysis demonstrated that the PCP was successively reduced to be lower chlorinated organic substances, and the total CPs decreased under the action of ZVI/GAC.

Keywords: Anaerobic digestion; Granular activated carbon; Pentachlorophenol wastewater; Zero-valent iron

1. Introduction

Pentachlorophenol (PCP), an industrial chlorinated aromatic compound, is listed as a priority pollutant by U.S. Environmental Protection Agency (EPA) due to its toxic, carcinogenic and persistent nature [1]. It has been prohibited production and use in China as pesticide, fungicide, and herbicide [2]. However, PCP and other chlorophenol congeners (CPs) are still major by-products of some processes such as bleaching effluent of pulp and textile industries [3–5]. The discharge of CPs wastewater not only leads to accumulation and biological amplification, but also causes liver and kidney damage and infertility problems in humans [3,6]. Therefore, various physical, chemical and biological treatment methods have been used to treat wastewater containing CPs [7–9].

Anaerobic digestion of PCP has been considered as a promising technology due to its potential in recovering energy, making compounds less toxic and more readily biodegradable, and decreasing treatment costs [10,11]. Under the anaerobic conditions, the chlorines in the PCP can be removed from the aromatic ring via successive reductive dechlorination of microbial metabolism [12]. It is reported that the PCP removal efficiencies in the up-flow anaerobic sludge bed (UASB) or immobilized biomass reactors could reach 90%–99% [11,13]. However, phenolic substances

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exhibit great adverse effects on the anaerobic digestion process because of their biological toxicity [14]. Coupled with the special environmental conditions of methanogens and the long adaptation and retention time (above 20 d), the application of anaerobic digestion in wastewater treatment of phenolics was restricted [13,15]. The restrictive factors of PCP wastewater treatment were mainly focused on the use of chemicals and the stability of microbial community.

Therefore, some chemical material such as Fenton oxidation [16], persulfate [17] and zero-valent iron (ZVI) [18,19] have been added into the anaerobic digestion process to decrease biological toxicity of PCP, shorten the hydraulic retention time, and improve methane production [20,21]. As reported in the literatures, Fenton oxidation and persulfate activation could effectively remove PCP and CPs pollutants, but they were harmful to microorganisms or introduced pollutants and other equipment [16,17]. ZVI as a low-cost, non-toxic and potential oxidation properties was applied for dichlorination reaction [22]. Meanwhile, ZVI as highly reductive substance has showed its low oxidation-reduction potential in providing electrons to methanogens and then increasing the activity of methanogens [18]. It is reported that the methane production could increase by 40.4% by adding 0.1% ZVI into anaerobic reactors. In addition, granular activated carbon (GAC) was used as electron carriers to promote electron transfer between syntrophic partners and methanogens in the anaerobic processes, and the methane production was improved by about 34% [23]. The good pore structure, large specific surface area of GAC was also benefit for the adsorption of organic pollutants. In particular, the combined application of nano ZVI and activated carbon (AC) increased methane production by buffering the pH and enriching the Methanobacterium [24]. Nevertheless, the researches on the potential mechanism of ZVI and AC addition in anaerobic processes mainly focused on easily biodegradable organic matters rather than refractory organic matters.

Therefore, the effect and mechanism of simultaneous addition of ZVI and GAC (ZVI/GAC) on anaerobic biodegradation of PCP wastewater need to be studied. In this study, the effects of critical parameters on PCP degradation rate and kinetics, chemical oxygen demand (COD) removal were determined by batch tests. Continuous flow experiment was conducted in two-phase anerobic reactor to treat PCP wastewater under the optimal operational conditions, the relationship between PCP degradation, methane production and ZVI/GAC addition was investigated, and the PCP degradation pathways were also analyzed with ZVI or ZVI/GAC addition.

2. Materials and methods

2.1. PCP wastewater

The PCP wastewater used in this experiment was synthetic wastewater. It was composed of glucose, ammonium chloride, potassium dihydrogen phosphate, magnesium sulfate, calcium chloride, sodium bicarbonate and PCP. The characteristics were summarized in Table 1. In addition, each liter of feedwater needed to be added 1 mL of trace element solution, and the composition of trace element solution was described in previous study [25].

Table 1
Characteristics of pentachlorophenol wastewater

Item	Value
Chemical oxygen demand	5,000 ± 200 mg/L
PCP	50 ± 2 mg/L
Phosphate (PO_4^{3-}–P)	25 ± 0.5 mg/L
Ammonia nitrogen (NH_4^+–N)	125 ± 2 mg/L
pH	8.03–8.1

2.2. Inoculum

The inoculated sludge was collected from an UASB reactor in a brewery (Nanjing, China), and the VSS was 18,000 ± 200 mg/L. The inoculated sludge was domesticated with synthetic PCP wastewater, and the influent PCP concentration was controlled at 50 mg/L. The acclimation time of each stage was determined according to the removal stability of PCP and COD.

2.3. Characteristics of ZVI and GAC

The average particle size of ZVI was 80 nm, the purity was 99.9%, specific surface area was 27 m^2/g (Ningbo Jinlei Technology Co., Ltd., China). GAC produced from fruit shell was provided by a manufacturer (Changzhou Like Environmental Technology Co., Ltd., China). The shape of GAC was columnar, and the average particle size was 3 mm. The ZVI and GAC was washed in order to remove the surface oxides or other impurities with 2% hydrochloric acid solution and distilled water in turn, and finally dried under N_2 gas.

2.4. Experimental setup

2.4.1. Batch tests

Batch tests were conducted to study the effect of ZVI or ZVI/GAC addition on PCP degradation. In this experiment, the ZVI dosages were controlled at 0, 50, 100, 200 and 300 mg/L, respectively. The ZVI/GAC was formed by mixing ZVI and GAC, and the GAC dosage in ZVI/GAC group was fixed at 100 mg/L. The pH values in all reactors were maintained at 7 ± 0.5 by automatically adding 1 M sodium hydroxide or 1 M hydrochloric acid. The reaction temperature and time were 25°C ± 1°C and 5 h, respectively. Samples were taken and analyzed every 1 h.

2.4.2. Batch data analysis

The degradation efficiency of PCP by the treatment methods is closely related to the reaction kinetics. To understand and explain the effect of ZVI and ZVI/GAC on the degradation process of PCP in anaerobic reactor, the degradation kinetics of PCP was further explored. The degradation kinetics data have been compared with the pseudo-first-order rate equations.

$$v = -\frac{d[\mathrm{PCP}]}{dt} \quad (1)$$

$$k_{obs} = -\ln\left[\frac{PCP}{PCP_0}\right] \quad (2)$$

where v is overall reaction rate, mg/(L min); t is reaction time, min; k_{obs} is apparent rate constant, PCP_0 is initial concentration of PCP, mg/L; PCP is the concentration at time, mg/L.

2.4.3. Continuous flow experiment

Two-phase anaerobic reactors were used to simulate continuous flow experiment for PCP wastewater treatment. The experimental device is shown in Fig. 1. The working volume was 2 L, and the size parameters were 12 cm × 10 cm × 25 cm. The hydraulic retention time of hydrolysis stage and methanogenesis stage was 4 and 20 h, respectively. The operational temperature was 25°C ± 1°C. Samples were taken and analyzed every day.

In this experiment, the ZVI and GAC dosages were determined according to the result of batch tests of PCP degradation. Three parallel experiments were R1 (0 mg/L), R2 (200 mg/L ZVI) and R3 (200 mg/L ZVI, 100 mg/L GAC), respectively.

2.5. Analytical methods

The COD was determined according to the standard methods [26]. pH and oxidation reduction potential (ORP) were monitored using WTW Handheld Multiparameter Instruments (pH/Oxi 340i, WTW, Germany). The PCP was analyzed by high performance liquid chromatography (Alliance e2695, waters, America) as described in the standard methods [26]. The metabolites of PCP were qualitatively analyzed using gas chromatography-mass spectrometry (Agilent 6850 series II GC-MS) according to the literature [10].

The samples were performed in triplicate, and the average value was calculated.

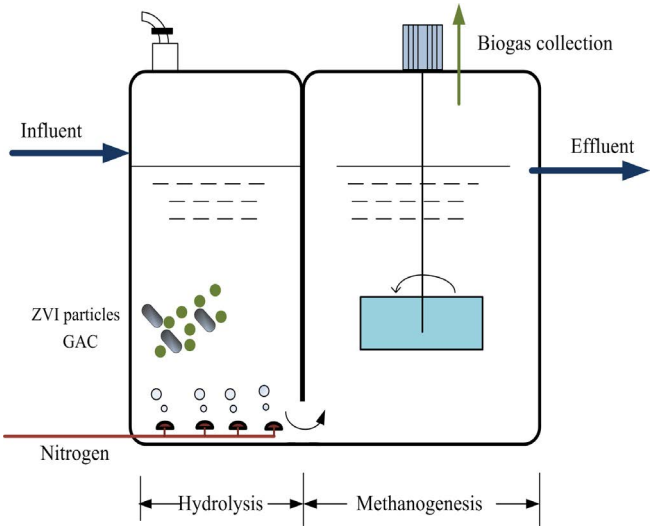

Fig. 1. Schematic of the two-phase anaerobic reactor.

3. Result and discussion

3.1. Effect of ZVI on PCP degradation

The variation of PCP concentrations at different ZVI dosages is shown in Fig. 2a. An obvious decrease of PCP concentration was observed with the increase of ZVI addition. For instance, on the 300 min, the PCP concentrations decreased from 36.63 to 15.64 mg/L when the ZVI increased from 0 to 300 mg/L, corresponding removal efficiencies increased from 26.7% to 68.72%. Simultaneously, it can be seen that the PCP concentrations decreased firstly and then remained relatively stable with the extension of time. Take ZVI of 300 mg/L for example, the PCP concentration decreased significantly from 50 to 17.9 mg/L in the first 120 min. In addition, the data in Fig. 2b show the effects of ZVI/GAC on the PCP concentrations. The change trend of PCP concentrations with ZVI/GAC addition was similar to that with ZVI addition. The PCP concentrations decreased from 35.63 to 10.21 mg/L with ZVI/GAC increasing from 0 to 300 mg/L, corresponding removal efficiency also increased from 28.74% to 79.58%. Simultaneously, it was observed that the PCP concentration at ZVI/GAC of 300 mg/L decreased from 50 to 12.09 mg/L in the first 120 min, and then stabilize at around 10 mg/L. These results indicated that ZVI/GAC was benefit for the degradation of PCP.

The kinetic study was carried out to analyze the influence of ZVI and ZVI/GAC on the performance of PCP degradation in the two-phase anaerobic reactor. The degradation rate of PCP within 60 min fitted well with the pseudo-first-order reaction model [Eqs. (1) and (2)] at each given ZVI concentration. The plotting ln(kobs) yielded a linear curve in ZVI and ZVI/GAC group with a slope approximating 2.333×10^{-5} and 4.809×10^{-5}. It was clear that the dosages of ZVI/GAC had a significant influence on the PCP degradation.

It is proposed that theoretically there were three reactions in the degradation of chlorinated organic compounds by ZVI: (1) Direct electron transfer on the surface of anaduring corrosion of ZVI. The corresponding chemical equations are shown in Eqs. (3)–(5).

$$Fe^0 + RCl + H^+ \rightarrow RH + Fe^{2+} + Cl^- \quad (3)$$

$$2Fe^{2+} + RCl + H^+ \rightarrow RH + 2Fe^{3+} + Cl^- \quad (4)$$

$$H_2 + RCl \rightarrow RH + H^+ + Cl^- \quad (5)$$

In this study, the data of pH (shown in Fig. 1s) was used to reflect the change of redox potential. ZVI oxidation was happened rapidly in low pH environment since a large number of volatile acids were produced in hydrolysis-acidification process ($Fe^0 - 2e- \rightarrow Fe^{2+}$; $Fe + 2H^+ \rightarrow Fe^{2+} + H_2$). As the reaction proceeded, H^+ is consumed and pH value increased. In ZVI addition group, a large number of H^+ was consumed with the increase of ZVI addition amount, pH value increased to 8.7 from 5.5. So, the main reason for the increase in PCP removal rate with ZVI addition might be due to the oxidation-reduction reaction between ZVIand

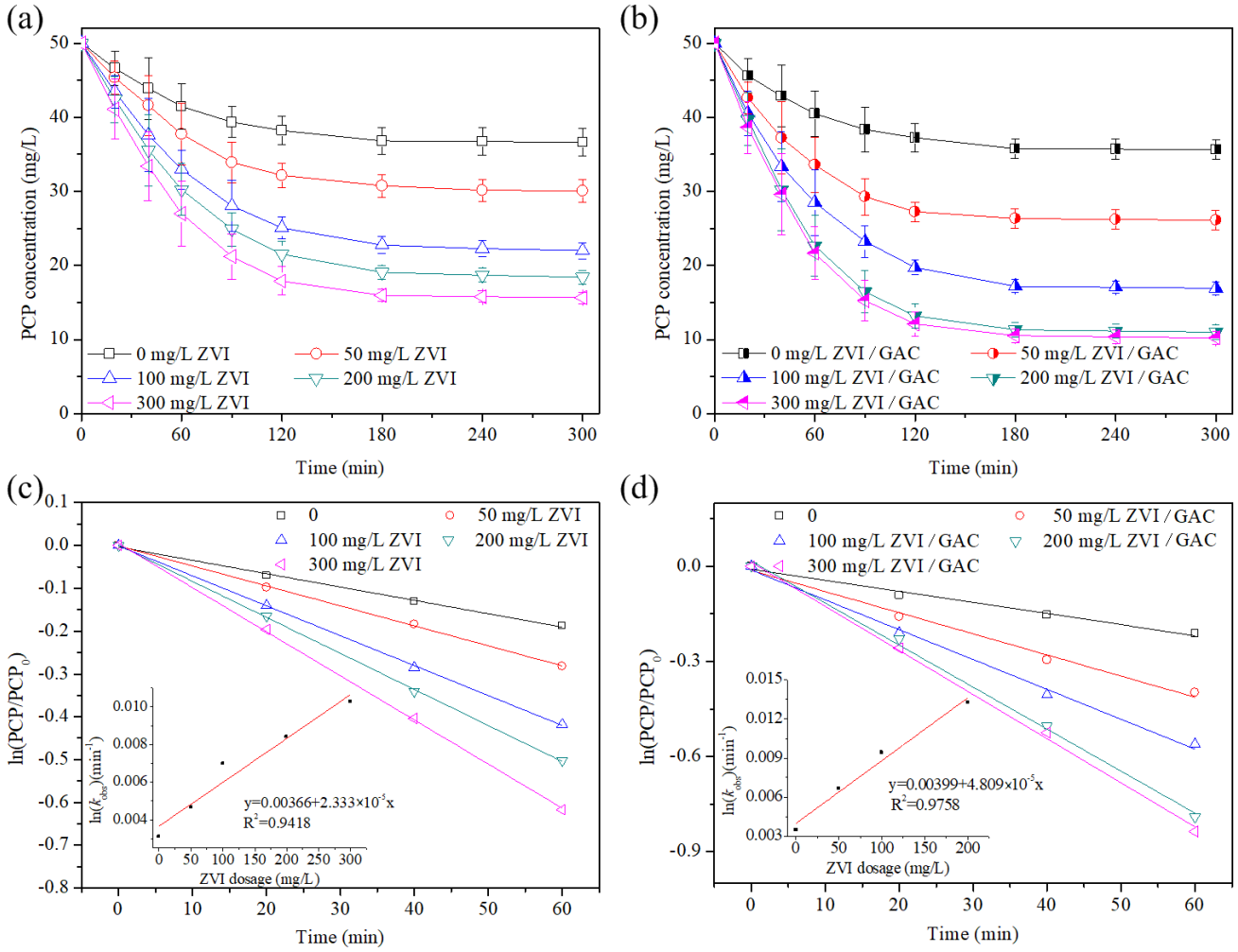

Fig. 2. Effects of (a) ZVI dosage, (b) ZVI/GAC on PCP degradation, (c) kinetic analysis of PCP degradation with ZVI addition, and (d) kinetic analysis of PCP degradation with ZVI/GAC addition.

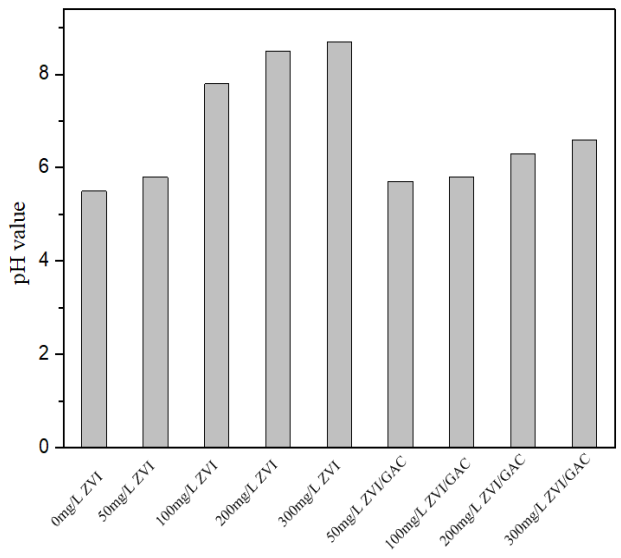

Fig. 1s. pH values in different addition groups.

PCP. It is generally believed that reducing agent ZVI acted as an electron donor during the reaction, and the PCP got electrons to be reduced and degraded. The reduction and degradation of PCP and chlorophenol compounds by ZVI might be mainly through the direct electron transfer between iron and PCP (described in Eqs. (6) and (7)).

$$Fe^0 - 2e^- \rightarrow Fe^{2+} \quad (6)$$

$$RCl_x + 2e^- + H^+ \rightarrow RHCl_{x-1} + Cl^- \quad (7)$$

In addition, the pH value maintained in range of 5.7–6.6 in the group of ZVI/GAC addition (Fig. 1s). Combined with the degradation efficiency of PCP (Fig. 2), this result indicated that the primary battery formed by ZVI/GAC has a better degradation rate of PCP than the ZVI single oxidation process. The previous research also demonstrated the fact that the GAC has been used to form primary battery with ZVI, and the internal electrolysis played an important role [21]. At a certain dosage of GAC, a larger number of primary

batteries produced with the increase of ZVI dosage, thereby improving the removal efficiency of PCP. Notably, a slight change of PCP concentration was observed when ZVI further increased from 200 to 300 mg/L. Because the number of primary batteries were limited by the dosage of GAC, simultaneously, the excessive ZVI directly reacted with H^+, resulting in a reduction in the redox reaction [21,27].

3.2. Effect of ZVI on COD removal

As shown in Fig. 3, the COD removal performances were enhanced after adding ZVI and ZVI/GAC, and the change trends was similar to the PCP concentrations. For example, after reaction time of 300 min, the COD concentrations decreased from 4,482 to 4,124 mg/L with ZVI dosages increasing from 0 to 300 mg/L, and corresponding values in ZVI/GAC group decreased from 4,468 to 3,431 mg/L. Simultaneously, the removal efficiency of COD was positively correlated with the dosages of ZVI and ZVI/GAC. The above results indicated that the synchronous enhancement of ZVI and GAC was more conducive to promote COD removal. The internal electrolysis of primary battery produced by ZVI and GAC also played a significant role in oxidizing other biodegradable organics.

3.3. Continuous operation performances

The effluent concentrations and removal efficiencies of PCP during anaerobic digestion process were investigated by long-term continuous experiments. From Fig. 4, when the influent PCP concentration was 50 mg/L, the effluent PCP concentrations of hydrolysis phase followed the order: R3 (10.2–12.4 mg/L) < R2 (14.9–17.7 mg/L) < R1 (34.3–40.5 mg/L), and corresponding removal efficiencies of PCP were 75.2%–79.6%, 64.6%–70.2%, and 19.0%–31.4%, respectively. Simultaneously, a significant decrease in the effluent PCP concentrations was observed during the methanogens phase. The effluent PCP concentrations in the R1, R2 and R3 were in the range of 8.65–20.51 mg/L, 4.3–8.7 mg/L, and 0.21–3.48 mg/L, respectively, and corresponding removal efficiencies reached 60.0%–82.7%, 82.6%–91.4%, and 93.0%–99.6%. The results indicated that both PCP removal performance and the operational stability were significantly enhanced by the addition of ZVI/GAC during 30-d running period.

3.4. Methane production

The methane production during anaerobic digestion at different ZVI and ZVI/GAC dosages is shown in Fig. 5. The average methane productions were 552.1, 1,241.5 and 1,482.3 mL in the R1, R2 and R3 reactors, respectively. This result demonstrated that the activity of methanogens and the stability of anaerobic digestion was enhanced with by ZVI or ZVI/GAC addition. It is reported that ZVI not only improved the yield of acetic acid and provided sufficient substrate for methanogens, but also was directly used as an electronic donor to reduce CO_2 to produce methane [28]. In addition, the maximum methane production was observed with 200 mg/L ZVI/GAC addition, which might be partially due to the internal electrolysis of ZVI and GAC. The stability of methane production might be that ZVI/GAC addition enhanced the biodegradability of PCP, which in turn reduced the substrate toxicity for microorganisms involved in hydrolysis, acidification and methanogenesis.

3.5. Degradation pathway analysis

It is well known that the metabolites of PCP include monochlorophenol (MCP), dichlorophenol (DCP), trichlorophenol (TCP), tetrachlorophenol (TeCP) and so on [10,29]. As shown in Fig. 6, the PCP was successively reduced to be lower chlorinated organic substances, and the total CPs in the R3 reactor were significantly decreased. For example, the PCP concentration was decreased to 10.21 mg/L, and the 2,4-DCP and 2-CP concentrations were enhanced to 7.13 mg/L and 8.64 mg/L by ZVI/GAC. It can be seen that the 2,3,4,6-TeCP increased and 2,3,5,6-TeCP decreased after adding ZVI or ZVI/GAC, indicating the degradation pathway were

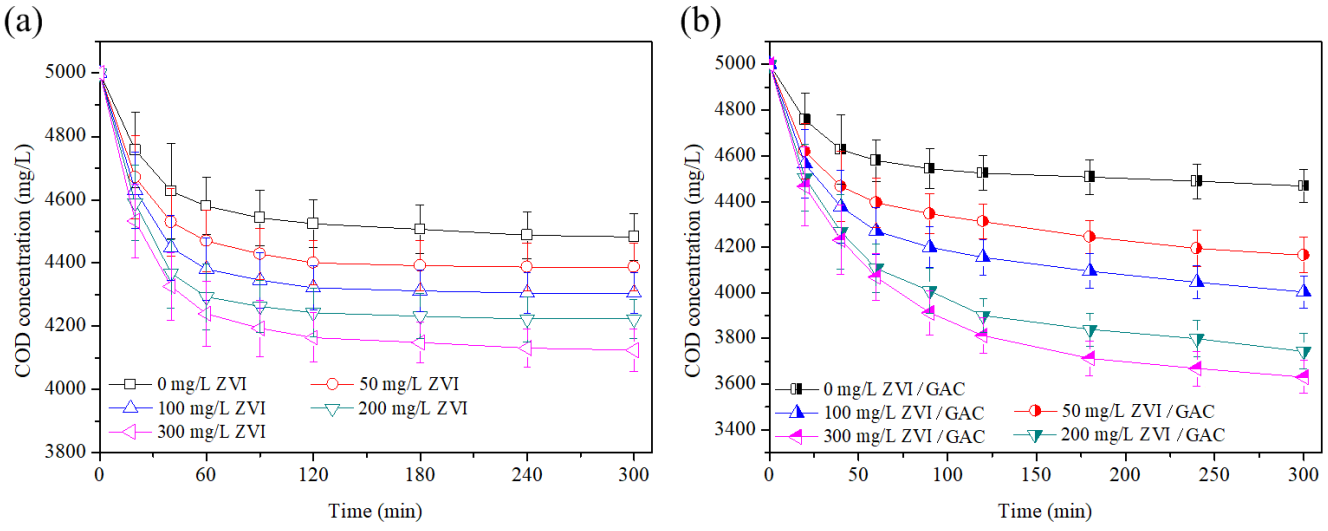

Fig. 3. Effects of (a) ZVI dosage and (b) ZVI/GAC on COD degradation.

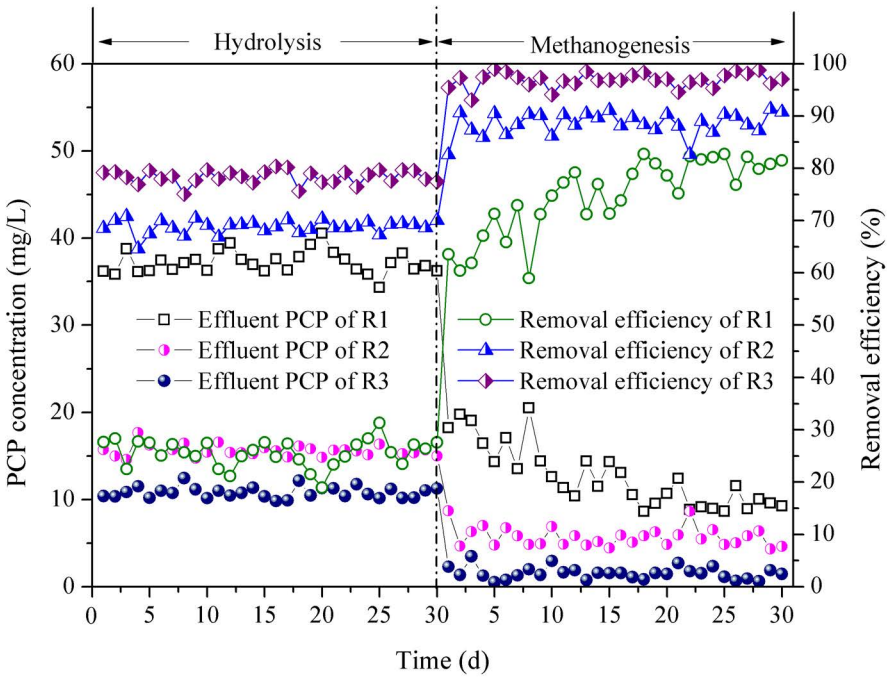

Fig. 4. Effect of ZVI and ZVI/GAC on PCP and COD degradation in a continuous flow reactor.

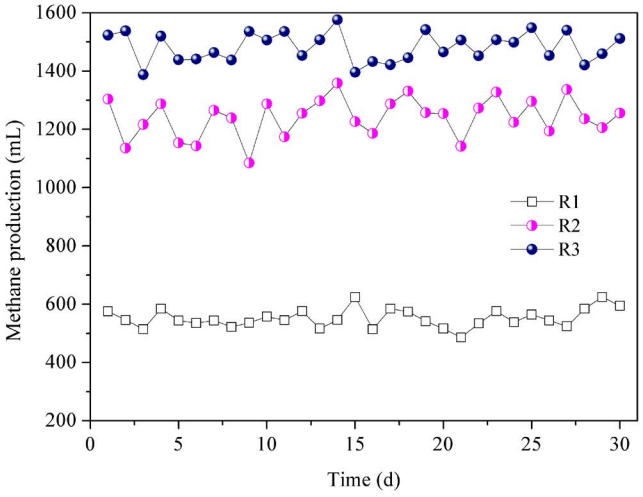

Fig. 5. Effect of ZVI and ZVI/GAC on methane production in the methanogenic stage.

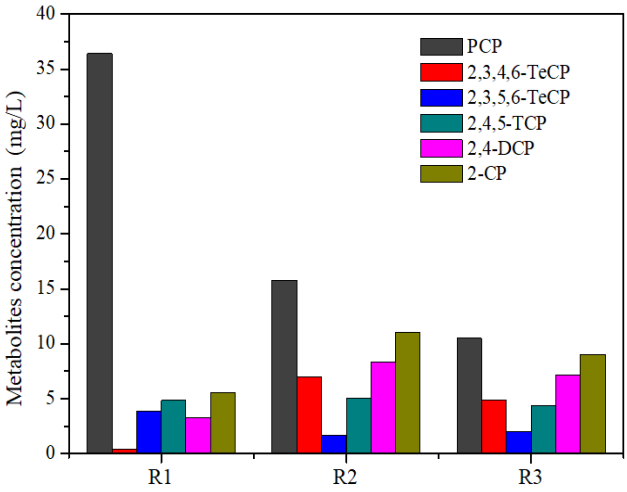

Fig. 6. Effects of ZVI and ZVI/GAC on PCP metabolites.

changed significantly. Combining with Figs. 4 and 5, ZVI/GAC had a better effect than ZVI in terms of PCP removal, methane production, and PCP conversion. Because ZVI/GAC could degrade organics in the wastewater by thousands of tiny microbatteries produced by ZVI and GAC [28].

4. Conclusions

This study demonstrated the addition of ZVI or ZVI/GAC showed an effective role in PCP removal and methane production. The optimal dosage of ZVI for PCP and COD removal were determined by batch tests, and the removal efficiency of PCP by ZVI can be further enhanced with the GAC addition. Simultaneously, the comparative results of continuous flow experiments also demonstrated that ZVI/GAC had more conductive effect in removing PCP, promoting methane production, and improving operational stability. In addition, the analysis of degradation pathways also showed that PCP and its metabolites are oxidized effectively by thousands of microbatteries formed by ZVI and GAC, thereby reducing the toxic effects of PCP on methanogens.

Author contributions

Conceptualization, P.C.; validation, P.C. and Y.Z.; investigation, P.C.; data curation, P.C.; writing-original draft preparation, Y.L.; writing-review and editing, Y.L.

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