Characteristics of ball-milled PET plastic char for the adsorption of different types of aromatic organic pollutants

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Abstract
Ball-milled plastic char (BMPC) was manufactured by ball-milling of native plastic char (PC) that was synthesized via slow pyrolysis of polyethylene terephthalate (PET) water bottle waste, and its adsorption characteristics of aqueous phenanthrene (PHE), phenol, and 2,4,6-trichlorophenol (2,4,6-TCP) and its possible mechanisms were investigated. With the increase of PC pyrolysis temperature, the specific surface area of BMPC increased obviously, forming larger functional groups compared to PC. Boehm titration showed that total acidic groups of BMPC decreased significantly with the increase of pyrolysis temperature. The sorption kinetics of three adsorbates was adequately simulated by pseudo-second-order model \( R^2 > 0.99 \). Langmuir model fitted well the adsorption isotherms of PHE and phenol, while Freundlich model simulated the adsorption isotherm of 2,4,6-TCP better. The adsorption amount of PHE, phenol, and 2,4,6-TCP increased significantly as the pyrolysis temperature increased. The maximum BMPC adsorption capacity reached 21.9 mg·g\(^{-1}\) (for PHE), 106 mg·g\(^{-1}\) (for phenol), and 303 mg·g\(^{-1}\) (for 2,4,6-TCP) at 25 °C in aqueous solution. FTIR analysis suggested that surface sorption-based π–π interaction was a dominant mechanism of PHE adsorption; meanwhile, H-bonding between O-containing groups on BMPC and hydroxyl groups of adsorbates was responsible for phenol and 2,4,6-TCP removal. This paper shows that BMPC can be used as adsorbent for treating aromatic compounds in aqueous environment and has an economic worth of application.

Keywords  Ball-milled plastic char · PET · Adsorption · Phenanthrene · Phenol · 2,4,6-Trichlorophenol

Introduction
Due to economic and population growth, the contemporary world is facing the challenge of increasing plastic waste in the environment. The bulk of municipal solid waste is plastic packaging, which accounts for about 40% (polyvinyl chloride (PVC) 0.8%, polystyrene (PS) 2.05%, PET 9.02%, polypropylene (PP) 7.32%, high-density polyethylene (HDPE) 8.3%, and low-density polyethylene (LDPE) 12%) of global plastic production (Tejaswini et al. 2022). More than 90% of plastic feedstock comes from fossil fuels, so recycling is the best solution to guide the reduction of fossil fuel extraction in circular economy activity.

Of existing plastics, PET, as most important engineering polymer, is harmless to the human body and widely used in all fields of life and industry, such as water bottle, packaging foods, liquid soap, and cooking oil, manufacturing films, and fibers. Enormous consumption of PET plastic inevitably led to abundant accumulation of PET wastes (Ansari et al. 2021), that is, PET waste generation increased annually from 2015 (13 million tons) to 2019 at a rate of 5.2% (Kim et al. 2020). According to official statistics, nearly 20,000 waste plastic bottles have been thrown away every second, and the recycling rate for these PET bottles is 72.1% in Japan and only 48.3% in Europe and 31% in the USA. Therefore, any new methodologies for the recycling of these plastics...
have been proposed even though their applicability is not so plausible at larger scale (Fuks et al. 2021). Generally, the chemical composition of these plastics is mostly composed of volatile; however, PET also contains about 12% fixed carbon in addition to volatile components (Chattopadhyay et al. 2016; Hasanzadeh et al. 2022), which is higher than that of other plastics. Therefore, higher content of fixed carbon in PET would be superior to that of other plastics from the aspect of using as substrate for carbonaceous adsorbents.

Pyrolysis-based thermochemical principle is an effective technique to convert solid wastes into usable gas, liquid, and solid products in the condition of oxygen-free, and the technology has been mature and widely used to treat large amounts of municipal solid wastes (Al-Salem et al. 2017; Dhahak et al. 2020; Muhammad et al. 2015). Recently, it has been proved that the solid by-product (plastic coke) pyrolyzed at temperatures typically between 500 and 780 °C (Muhammad et al. 2015) could be efficiently used for the removal of heavy metals (Fuks et al. 2021; Singh et al. 2020a, 2021). It has been improved that the pyrolysis under the inert atmosphere could change PET to a highly porous material (Bratek et al. 2013) with pores in the micro-/meso-sizes, which is applicable for different adsorption processes (Parra et al. 2006). Ester bonds of PET chain were randomly decomposed at the initial stage of pyrolysis to produce carboxyl and vinyl esters and through further thermal process, which resulted in the product with high content of aldehydes at low-temperature treatments and one with a high content of aromatics at high-temperature treatments (Martin-Gullon et al. 2001). The specific surface area also has a significant effect on the adsorption of various organic pollutants (Guo et al. 2017). Ball-milling method, as one of the methods to improve the absorbent properties, is a cost-effective way to increase the specific surface area of carbon materials; therefore, ball-milled biochar has a good adsorption effect on heavy metals, dyes, and organic pollutants in water and wastewater (Amusat et al. 2021; Lyu et al. 2018b; Sun et al. 2022; Xiang et al. 2020).

The organic pollutants studied in here, such as PHE, phenol, and 2,4,6-TCP, are aromatic hydrocarbons, which are all toxic and recalcitrant with different physicochemical properties. PHE is a typical aromatic (3-ring) carbides of polycyclic aromatic hydrocarbons, and due to its low solubility to water (1.2 mg·L⁻¹ at 25 °C) (Zindler et al. 2016), it has high eco-toxicity and long-term persistence; it is considered as an environmental pollutant that can cause acute and chronic diseases to the human health (Kumar et al. 2021). On the other hand, phenol as the basic raw materials of industry is weak acid and has high toxicity and high solubility in water (85 g·L⁻¹ at 25 °C) (Guo et al. 2021). It was seriously polluted in many kinds of industry wastewater, e.g., 6–500 mg·L⁻¹ in refinery wastewater, 28–3900 mg/L in coking wastewater, and 2.8–1220 mg·L⁻¹ in petrochemical industry wastewater (Mei et al. 2020), and undoubtedly its direct discharge to river could bring serious environmental problems (Yan et al. 2018). 2,4,6-TCP has been reported to cause many health diseases, such as respiration, cardiovascular effects, gastrointestinal effects, and cancer except for nervous toxicity to human (Fan et al. 2011). In addition, the relatively high solubility of 2,4,6-TCP at room temperature (434 mg·L⁻¹ in water) (Pei et al. 2013) as well as the stable C–Cl bonds and the positions of chlorine atoms in phenol ring led to higher toxicity and endurance in the environment compared to phenol (Tan et al. 2009).

Many methods have been proposed to remove organic pollutants, of which, adsorption method has been considered to be one of the effective and economic approaches to remove different organic pollutants from aqueous environments. For example, adsorption capacities of PHE and phenol were 15.8 mg·g⁻¹ (for PHE), 166 mg·g⁻¹ (for phenol) with biochar based on bamboo (Mohammed et al. 2018; Tang et al. 2015), and 367 mg·g⁻¹ (for 2,4,6-TCP) with Loosestrife-based activated carbon at 25 °C, respectively (Fan et al. 2011). In the previous study, the plastic char mainly consisted of mesoporous and macroporous material with adsorption capacities being 3.59–22.2 mg·g⁻¹ for methylene blue dye. This indicates that the plastic char can also have good adsorption effect on organic molecules (Bernardo et al. 2012; Sharuddin et al. 2016).

In the study of PET pyrolysis, it is mainly concentrated on the gas and liquid production with the change of thermal decomposition temperature (Dimitrov et al. 2013); however, there are few studies on the characteristics of solid products (Fuks et al. 2021; Parra et al. 2006; Sogancioglu et al. 2017), especially there has been no study on the adsorption characteristics of organic pollutants by PET plastic char produced from different pyrolysis temperatures. This paper aimed (1) to analyze the physicochemical characteristics of BMPCs (BMPC-500, BMPC-600, BMPC-700, and BMPC-800) produced at different pyrolysis temperatures (500–800 °C) through the adsorption capacities of PHE, phenol, and 2,4,6-TCP in aqueous environment and (2) to investigate the adsorption mechanism of BMPCs and influencing factors.

Materials and methods
Sample preparation and treatment
In this study, Wahaha water bottle was used as raw material (PET plastics) for the plastic char. PHE, phenol, and 2,4,6-TCP (MERYER 99%) as pollutants, calcium chloride anhydrous (CaCl₂) as regulator of ionic strength, and sodium azide (NaN₃) as an inhibitor of microbial growth were purchased from Tianjin Huaxun Medical Technology Co., Ltd (China). Wahaha water bottle was washed and air-dried and
crushed into pieces below 0.5 cm × 0.5 cm of size. Firstly, 6 g PET pieces was pyrolyzed under various pyrolysis temperatures in N2 gas condition using a tubular furnace (TDRG, Tengda Thermal Technology Co., Ltd., Yixing, China). The pyrolysis experiment was carried out at different temperature (500, 600, 700, and 800 °C) for 30 min (see detail in Text S1). Secondly, the four kinds of plastic char samples were ball-milled (F-P2000, Focucy, Hunan, China) for 24 h (see detail in Text S2).

Characterization of BMPCs

A multiple-point Brunauer–Emmett–Teller (BET) method was employed to measure the surface area, pore size, and pore volume of BMPC (ASAP2460, Micromeritics, Atlanta, USA), and element analysis was determined using the CHN/O Analyzer (EA3000, Italy). The pH value was measured by a pH meter (PB-10, Sartorius, Goettingen, Germany). SEM analysis (JSM-7800F, JEOL, Japan) was performed to monitor the shapes and surface morphologies of the samples. Raman spectroscopy (SR-5001-A, TEO, USA) was used to characterize the aromatic structure. The functional groups of BMPCs were investigated by Boehm titration method (see detail in Text S3) and Fourier transform infrared spectroscopy (FTIR) measurements (TENSOR 37, BRUKER, Germany). Zeta potential was determined with a Zetasizer Nano ZS90 (Malvern Instruments, Malvern, UK).

Adsorption experiment

PHE, phenol, and 2,4,6-TCP were adopted as experimental organic pollutants, respectively. The adsorption characteristics of BMPCs and PCs prepared at different pyrolysis temperatures were studied in batch experiment. For PHE adsorption experiment, test solution of PHE at various concentration was prepared with the PHE stock solution diluted with HPLC-grade methanol containing 0.01 M CaCl2 (ionic strength adjuster) and 200 mg·L−1 NaN3 (biocide) solutions. The concentration of methanol in the final solutions was always controlled below 0.1% (v/v) (Hundal et al. 2001). Two mg of BMPCs were, respectively, put into 50-mL brown vials, and then a 40 mL aliquot of PHE solution (0.6 mg·L−1) was added into the vials. The vials were then tightly capped with Teflon-lined screw caps and vibrated at 150 r·min−1 on a reciprocating shaker (HNY-2102C, Honour Instrument Co., Ltd., Tianjin, China) under the condition of the dark at 25 °C for 72 h (Jin et al. 2018; Zhao et al. 2014). As for phenol and 2,4,6-TCP adsorption, samples (10 mg) of BMPCs were put into 50-mL brown vials, and then a 40 mL aliquot of phenol and 2,4,6-TCP solution (the initial phenol and 2,4,6-TCP concentration was 10 mg·L−1 and 50 mg·L−1, respectively) were added into every vial and carried out for 24 h. Other operational condition was the same as above. The adsorption characteristics of PCs were also carried out in the same way as that of BMPCs.

In order to determine the effective dosage for PHE, phenol, and 2,4,6-TCP removal, experiments were carried out by changing BMPC dosages (0.025–0.15 g·L−1 for 0.6 mg·L−1 PHE, 0.125–0.375 g·L−1 for 10 mg·L−1 phenol, and 0.125–0.5 g·L−1 for 50 mg·L−1 2,4,6-TCP).

Initial solution concentration of 0.6 mg·L−1 PHE, 10 mg·L−1 phenol, and 50 mg·L−1 2,4,6-TCP was individually used to describe sorption kinetic of PHE, phenol, and 2,4,6-TCP, while BMPC dosage was 0.05 g·L−1 for PHE solution and 0.25 g·L−1 for phenol and 2,4,6-TCP solution. Adsorption kinetics models such as the pseudo-first-order model and pseudo-second-order model were usually employed to analyze their kinetics behavior and mechanisms in their adsorption process. The integrated adsorption kinetics equations are separately written as.

Pseudo-first-order model:

\[ q_t = q_e \left(1 - e^{-K_1t}\right) \]  (1)

Pseudo-second order model:

\[ \frac{t}{q_t} = \frac{1}{q_e} + \frac{1}{K_2q_e^2} \]  (2)

where \( q_t \) (mg·g−1 solid material) and \( q_e \) (mg·g−1 solid material) are the amount of adsorbed PHE, phenol, and 2,4,6-TCP at equilibrium and time \( t \) (h), respectively, and \( K_1 \) (h−1) and \( K_2 \) [g·(mg·h)−1] are rate constants for the pseudo-first-order and pseudo-second-order, respectively.

For the sorption isotherm study, experiments were carried out according to initial concentration change of PHE, phenol, and 2,4,6-TCP (0.6–1 mg·L−1 for PHE, 10–50 mg·L−1 for phenol, and 50–100 mg·L−1 for 2,4,6-TCP); in here, dosage in these experiments were 0.05 g·L−1 BMPCs per 40 mL of PHE solution and 0.25 g·L−1 BMPCs per 40 mL of phenol and 2,4,6-TCP solution, and experiment was carried out with 72 h for PHE adsorption and with 24 h for phenol and 2,4,6-TCP adsorption test. In order to confirm the maximum adsorption capacity of adsorbents and clarify the adsorption mechanisms, the Langmuir and Freundlich equations are used to describe the adsorption isotherm. The Langmuir isotherm model posits that adsorption is monolayer and takes place at specific homogeneous adsorption sites within the adsorbent which have equal solute affinity, and there is no interaction between the adsorbed molecules. Following homogeneous adsorption, the adsorbent molecules have constant enthalpies or surface activation energy.

The Langmuir isotherm model is represented as

\[ q_e = \frac{q_m \cdot K_L \cdot C_e}{1 + K_L \cdot C_e} \]  (3)
where $q_m$ (mg·g$^{-1}$) is the maximum adsorption capacity of the adsorbent and $K_L$ (L·mg$^{-1}$) is the Langmuir adsorption constant related to the adsorption energy. The Freundlich isotherm model describes a multilayer adsorption process, and the surface of adsorbent is heterogeneous with non-uniform distribution of adsorption heat and affinity. The Freundlich isotherm model is described as

$$ q_e = K_F \cdot C^\frac{1}{n} $$

where $K_F$ [(mg·g$^{-1}$) ·(mg·L$^{-1}$)$^{-n}$] and $n$ are Freundlich adsorption isotherm constants related to the saturation capacity and intensity of adsorption, respectively. The experiment was carried out in triplicate, and the adsorption amount was calculated according to the initial and final concentration of pollutants and the dosage of adsorbents (see detail in Text S5). Adsorption data were fitted to the adsorption kinetic and isotherm models using software Origin 9.0, and nonlinear regression analyses were conducted by the least-squares method.

**Data analysis**

The determination of PHE was as follows. A 0.5 mL of each supernatant that was centrifuged at 4000 r·min$^{-1}$ for 10 min was diluted with 0.5 mL of HPLC-grade methanol and then followed by filtration using a 0.22 nm PTEF filter membrane. Agilent HPLC 1260 (USA) equipped with a Thermo Scientific C18 column (25 cm × 4.6 mm) was used for PHE quantification (Shi et al. 2018). A mixture of acetonitrile and Millipore water (v/v = 80/20) was eluted at a flow rate of 1 mL·min$^{-1}$ and system temperature of 30 °C, and detection was conducted at wavelength of 254 nm with a UV detector for PHE determination.

The following manipulation was performed for phenol and 2,4,6-TCP. A 1.5 mL of supernatants that were centrifuged at 4000 r·min$^{-1}$ for 5 min was filtered using a 0.22 nm PTEF filter membrane and then measured by HPLC. The method is the same as that of PHE, but the wavelength (280 nm) and the system temperature (25 °C) were different to that of PHE.

**Results and discussion**

**Physicochemical characterization of BMPCs**

The pyrolysis product of PET (C$_{12}$H$_8$O$_4$)$_n$ consisted of gas, waxy, and carbonaceous residue (Dhahak et al. 2019); as pyrolysis temperature increased (500–800 °C), production yields of solid residues (carbonaceous residue) decreased gradually from 18.9 ± 0.2 wt% at 500 °C to 13.6 ± 0.2 wt% at 800 °C (Table 1). Solid residues from PET pyrolysis were initially lumped like slag, of which, the specific surface area of PCs (PC-500, PC-600, PC-700, and PC-800) was relatively small, and the size of them was arranged from hundreds to thousands of microns (see detail in Table S1). In general, specific surface area is considered as a crucial factor to the adsorbent sorption; therefore, produced carbonaceous residues were ball-milled for 24 h to increase specific surface area. PCs were initially looked like black flakes to be indistinguishable from each other; however, after ball-milling, BMPCs could be seen differently with different pyrolysis temperature. Color of BMPCs was changed from black-brown to strong black as pyrolysis temperature raised (see

| Sample       | BMPC-500 | BMPC-600 | BMPC-700 | BMPC-800 |
|--------------|----------|----------|----------|----------|
| Product yield (%) | 18.9     | 17.3     | 15.2     | 13.6     |
| BET surface area (m$^2$·g$^{-1}$) | 361      | 439      | 539      | 583      |
| Pore volume (cm$^3$·g$^{-1}$) | 0.213    | 0.258    | 0.308    | 0.318    |
| Average pore size (nm) | 2.361    | 2.347    | 2.288    | 2.177    |
| Average particle size (μm) | 0.934    | 0.828    | 0.756    | 0.524    |
| pH | 6.41     | 6.59     | 6.61     | 6.75     |
| C (%) | 86.432   | 89.115   | 89.225   | 91.236   |
| H (%) | 4.589    | 4.318    | 3.601    | 2.481    |
| O (%) | 8.979    | 6.567    | 7.175    | 6.284    |
| O/C | 0.0779   | 0.0804   | 0.0804   | 0.0689   |
| H/C | 0.637    | 0.581    | 0.484    | 0.326    |
| AI [mol DBE$_{AI}$/(mol C$_A$)]$^{-1}$ | 0.805   | 0.834    | 0.885    | 0.965    |
| I$_y$/I$_x$ | 0.920    | 0.934    | 1.007    | 1.004    |
| Zeta potential (mV) | −33.8   | −34.3    | −34.5    | −35.2    |

$^a$O/C, atomic mole ratio of oxygen to carbon; $^b$H/C, atomic mole ratio of hydrogen to carbon; $^c$AI, aromaticity index
Specific surface area of BMPCs increased from 361 to 583 m²·g⁻¹ as the pyrolysis temperature raised. The pH value is another index to explain adsorbent sorption (Mohammed et al. 2018; Ri et al. 2022). The pH of BMPCs and PCs were weak acid with values of 6.41–6.75 and 6.83–6.91, respectively, with the increase of pyrolysis temperature from 500 to 800 °C (see detail in Text S1). Ball-milling reduced pH values of BMPCs compared to PCs. The carbon contents of BMPCs increased from 86.4 to 91.2% when pyrolysis temperature increased from 500 to 800 °C (Table 1), whereas hydrogen and oxygen contents decreased from 4.59% to 2.48% and from 8.98% to 6.28%, respectively. According to previous reports, the gas produced during the pyrolysis of plastics such as HDPE, LDPE, PP, PS, PET, and PVC contained hydrogen, methane, ethane, ethene, propane, propen, butane, and butane (Martin-Gullon et al. 2001; Sharuddin et al. 2016; Singh et al. 2020b). In the range of 500–700 °C, the content of H₂ gradually increased accompanied by the decrease of CO content with temperature increase, and the contents of CH₄ and CO₂ kept constant. On contrary, the increase of H₂ and CH₄ contents while the decrease of CO and CO₂ contents occurred at the stage of 700–800 °C. It is worthwhile that the gas fraction of CH₄ increased at higher temperatures during PET pyrolysis (Bai et al. 2020); therefore, BMPC-700 had a high oxygen content more than the others.

On the other hand, the ratios of H/C varied from 0.637 to 0.326 concurrently with the aromaticity index (see detail in Text S4) from 0.805 to 0.965, as the charring temperature raised, from which, evidently implying the evolution of conjugated structure. The ratio of O/C can reflect the changes in O-containing functional groups of BMPCs with different pyrolysis temperatures (Xiao et al. 2016). The ratios of O/C showed a decreasing trend at 500–600 °C (from 0.0779 to 0.0553) but at 600–700 °C increased again from 0.0553 to 0.0804 and at 800 °C decreased again to 0.0689 (Table 1). BMPC sorption can be interpreted by the structure–activity relationships between the pyrolysis temperature, H/C atomic ratio, aromatic cluster formation, and sorption capacity. With decrease of the ratio of H/C atomic, the sorption capacity of organic pollutant by char increased linearly with different pyrolysis temperatures. As shown in Table 1, not only specific surface area and surface hydrophobicity of BMPCs but also the aromatization degree was gradually increased with increase in pyrolysis temperature. Zeta potential is an important indicator to characterize the behavior of nanoparticles in colloidal solutions (Biriukov et al. 2020). Zeta potential values of all BMPCs were getting more and more negatively charged as charring temperature raised.

Raman measurement showed that BMPCs represent two significant peaks at 1350 cm⁻¹ and 1595 cm⁻¹, and the I_D/I_G ratio increased with the increase of pyrolysis temperature, explaining more defects with higher pyrolysis temperature, which may be related to the condensation of small amorphous defects, the removal of some O-containing groups, and the succession of aromatic clusters (Guizani et al. 2017). However, at 800 °C, I_D/I_G ratio was a little bit less than that at 700 °C (Fig. 1a). Raman spectroscopy can be used to analyze the structural similarity between the produced materials and the perfect graphite. This analysis is usually used to evaluate the aromatic cluster structure of carbonaceous materials. The D-peak (1350–1400 cm⁻¹) represents the in-plane vibrations of sp³–bonded carbon atoms that reflects structural defects of graphene structure, while the G-peak (1550–1600 cm⁻¹) corresponds to the in-plane stretching vibration of SP² graphic carbon structures. I_D/I_G indicates the total number and/or size of graphitic micro-domains and the proportion of the sp³ to sp² bonding, which can be used to evaluate the degree of disorder/ordering structure (Tang et al. 2015; Xiong et al. 2021).

**Fig. 1** Raman (a) and FTIR (b) spectra of BMPCs derived from PET soft bottle at different pyrolysis temperature.
Previous study demonstrated that there existed a well-linear relationship between the intensity ratio of Raman band and atom ratio of O, H to C (Veiga et al. 2021; Xu et al. 2020). The Boehm titration result were summarized in Table S2. For all BMPCs and PCs, surface acidity was dominated by phenolic hydroxyl groups and lactonic and carboxyl groups on the all BMPCs which were hold in less 50% of total acidic functional groups. The total acidic functional group content of BMPCs decreased significantly from 0.0651 to 0.0501 mol·kg⁻¹, while that of PCs changed between 0.0252 and 0.0285 mol·kg⁻¹, respectively, as pyrolysis temperature raised. Boehm titration experiment showed that these findings were rather consistent with Raman measurement, i.e., ball-milling enhanced the surface acidity of adsorbents, and the change of O/C ratio and the pH value of BMPCs were dependent on total acidic functional groups (Tang et al. 2015; Xu et al. 2020; Yang et al. 2018).

The morphology of all BMPCs was also illuminated by SEM analysis (see detail in Fig. S2). All BMPC particles were mostly less than 2 μm of diameter in size, which caused the surface to enlarge and form many pores, and especially, BMPC-800 particles were mostly less than 1 μm of diameter in size.

FTIR helped to measure the characteristic stretching frequencies of all BMPCs and elucidate the adsorption mechanism of PHE, phenol, and 2,4,6-TCP. FTIR spectra of different BMPCs were compared with previous studies (Fig. 1b). BMPCs had peaks of many functional groups, including adsorption band positions at wavelengths of ~3600 cm⁻¹ (free OH groups representing alcoholic and phenolic), ~3020 cm⁻¹ (aromatic hydrogen), ~1710 cm⁻¹ (-COOH), ~1601 cm⁻¹ (aromatic C=C/C=O), ~1443 cm⁻¹ (=CH₂ bend; aromatic ring), ~1245 cm⁻¹ (C-O stretching vibration representing aromatic ethers), and 600–900 cm⁻¹ (aromatic C=H in plane bending) (Meng et al. 2014; Ri et al. 2015; Singh et al. 2020b; Tang et al. 2015; Zuo et al. 2021). As shown in Fig. 1, with increasing of thermal decomposition temperature, the richness of functional groups decreased, especially, the intensity of ~OH (3649 cm⁻¹), C-H bond (3057 cm⁻¹), and =CH₂ bend (~1443 cm⁻¹) were decreased until negligible at 800 °C, respectively (Meng et al. 2014). The peak positions of most functional groups shifted from 3637 to 3649 cm⁻¹ and 1443 to 1454 cm⁻¹ for =CH₂ stretching band, from 3028 to 3057 cm⁻¹ for C-H stretching band, from 1708 to 1716 cm⁻¹ for –COOH stretching band, and from 1245 to 1265 cm⁻¹ for C-O stretching band whereas negligible for C = C/C = O stretching band. The test of FTIR spectra and Boehm titration explained that BMPCs contained the C atoms bonded to different oxygen-containing groups, such as aromatic C = C/C = O and carboxylic –COOH groups (Amusat et al. 2021; Hu et al. 2014; Parra et al. 2006), and these hydrogen bonds were looser than the tightly bound cyclic OH isomer and OH-ether with increasing of charring temperature.

**Effects of pyrolysis temperature on PHE, phenol, and 2,4,6-TCP sorption capacity**

Different adsorbents of BMPCs were compared to investigate the effect on removal of three adsorbates (Fig. 2). With the increase of pyrolysis temperature, the removal efficiency by using PCs (from 35 to 68% for PHE, from 28 to 53% for phenol, and from 13 to 35% for 2,4,6-TCP) and BMPCs (from 72 to 99% for PHE, from 61 to 80% for phenol, and from 79 to 90% for 2,4,6-TCP) increased significantly; in particular, 2,4,6-TCP adsorption rate of BMPCs was more than twice as high as that of PCs.

However, 2,4,6-TCP removal rate increased from 79 to 90% at 500–700 °C of pyrolysis temperature, and removal rate of BMPC-800 reduced by 82%. The result explained

![Fig. 2](image-url)

**Fig. 2** Comparing with the removal rate of PHE (a), phenol (b), and 2,4,6-TCP (c) by using PCs and BMPCs. a PHE removal rate at 0.6 mg·L⁻¹ of PHE; solution volume = 40 mL, the adsorbent dosage of PCs and BMPCs = 0.05 g·L⁻¹ respectively, pH = 6.4–6.9, equilibrium time = 72 h, and temperature = 25 °C. b Phenol removal rate at 10 mg·L⁻¹ of phenol; solution volume = 40 mL, the adsorbent dosage of PCs and BMPCs = 0.25 g·L⁻¹ respectively, pH = 6.4–6.9, equilibrium time = 24 h, and temperature = 25 °C. c 2,4,6-TCP removal rate at 50 mg·L⁻¹ of 2,4,6-TCP; solution volume = 40 mL, the adsorbent dosage of PCs and BMPCs = 0.25 g·L⁻¹ respectively, pH = 6.4–6.9, equilibrium time = 24 h, and temperature = 25 °C.
that with the increase of pyrolysis temperature, the removal rate of three adsorbates on BMPCs and PCs improved except for the removal rate of on phenol and 2,4,6-TCP on BMPC-800 (Inyang et al. 2014; Lyu et al. 2018a). PHE, as a nonionic organic compound, can be hardly ionized; thus, its morphological structure may not be so respective to the change of the solution pH (Hu et al. 2014). The adsorption efficiency of phenol is best in the solution pH 6.5 (Lawal et al. 2021), and the TCP removal decreases significantly with initial pH of the solution changed from pH 2 to 12 (Pei et al. 2013; Tan et al. 2009).

Effects of adsorbent dosage on PHE, phenol, and 2,4,6-TCP removal

Effects of BMPC dosage on PHE, phenol, and 2,4,6-TCP removal efficiency were shown in Fig. 3. PHE removal rate enhanced from 53 to 99% when four kinds of BMPC dosage was increased from 0.025 to 0.15 g·L⁻¹ at 0.6 mg·L⁻¹ PHE, phenol removal efficiency enhanced about from 35 to 99% with increasing BMPC dosage from 0.05 to 0.375 g·L⁻¹ at 10 mg·L⁻¹ phenol, and 2,4,6-TCP removal efficiency increased from about 40 to 99% with BMPC dosage from 0.125 to 0.5 g·L⁻¹ at 50 mg·L⁻¹ 2,4,6-TCP.

PHE and phenol removal efficiency of BMPCs significantly increased with higher pyrolysis temperature under equal dosage condition; especially, the removal effect of BMPC-800 was better than BMPC-500 and BMPC-600, while the adsorption effect showed almost no difference compared with BMPC-700, and there was rather a slight tendency to shrink. For 2,4,6-TCP removal efficiency, BMPC-600 and BMPC-700 had a good removal rate; however, the removal rate of BMPC-800 was slightly higher than that of BMPC-500. From the test, in order to demonstrate the characteristics of each BMPCs, the optimal dosage of BMPCs used in subsequent trials was determined to be 0.05 g·L⁻¹ for PHE adsorption study and 0.25 g·L⁻¹ for phenol and 2,4,6-TCP adsorption study.

Adsortion kinetics and isotherms

The adsorption kinetics of PHE, phenol, and 2,4,6-TCP on BMPCs were simulated via pseudo-first-order kinetic model and pseudo-second-order model in this work (Mohammed et al. 2018; Wang et al. 2020; Zaghouane-Boudiaf and Bouitahala 2011). Sorption kinetic of different pollutants on BMPCs was shown in Fig. 4.

Most of PHE, phenol, and 2,4,6-TCP were rapidly adsorbed to BMPCs in less than 3 h (over 95%), and the apparent adsorption equilibrium was achieved within 72 h for PHE adsorption (8.6, 9.5, 11.5, and 11.7 mg·g⁻¹), 24 h for phenol adsorption (25, 27.5, 30, and 31 mg·g⁻¹), and 24 h for 2,4,6-TCP adsorption (153.8, 178.6, 181.8, and 156.3 mg·g⁻¹). Adsorption capacity of BMPCs increased significantly with increasing pyrolysis temperature in the both PHE and phenol cases (Fig. 4a and d), whereas adsorption capacity of BMPC-800 was not significantly different comparing to BMPC-700. In addition, 2,4,6-TCP adsorption capacity of BMPC-800 decreased significantly comparing to others (Fig. 4g). Pseudo-first-order kinetic model (Fig. 4b, e, and h) is the well-known and reliable model for the initial rapid response, and as for the pseudo-second-order model (Fig. 4c, f, and i), it is assumed that the adsorption rate relies on the number of active sites on the adsorbent surface as a rate constraint step (Hu et al. 2014; Tang et al. 2015). As expected, pseudo-second-order kinetic model fits the best for three kinds of organic pollutants sorption. In sorption kinetics, the correlation coefficients ($R^2$ value) of BMPCs for the pseudo-first-order model (0.79-0.821 for PHE, 0.72-0.8 for phenol, and 0.73-0.77 for 2,4,6-TCP) were smaller than that of pseudo-second-order model ($R^2$>0.99), which showed that
pseudo-second-order models described the kinetic adsorption process well. Table S3 shows the adsorption kinetic parameters, in which the adsorption process of PHE, phenol, and 2,4,6-TCP on BMPCs accords with pseudo-second-order kinetics, and reasonably, the adsorption rate relates to the number of active sites on the adsorbent surface, and the sorption of these pollutants predominantly occurred by chemisorption mechanism (Inyang et al. 2014).

By establishing the adsorption isotherm model, the adsorption capacity and equilibrium constant of PHE, phenol, and 2,4,6-TCP on BMPCs were derived, and Langmuir and Freundlich model equations were applied to describe the adsorption isotherm in detail (Fan et al. 2011; Godlewksa et al. 2019; Hu et al. 2014; Lv and Li 2020; Mohammed et al. 2018).

Figure 5 showed adsorption isotherm of PHE, phenol, and 2,4,6-TCP on BMPCs, and for three adsorbates, the sorption data was fitted using Langmuir and Freundlich models (see detail in Table S4). The result showed that two models illustrated the sorption isotherm data of three adsorbates well ($R^2 > 0.92$); with the pyrolysis temperature increase, for PHE sorption isotherm, $R^2$ values of two models decreased from 0.969 to 0.951 (Langmuir model) and from 0.951 to 0.921 (Freundlich model), and for phenol sorption isotherm, $R^2$ values of Langmuir model decreased from 0.998 to 0.994, and $R^2$ values of Freundlich model increased from 0.991 to 0.995; meanwhile, for 2,4,6-TCP sorption isotherm, $R^2$...
values of two models increased from 0.993 to 0.998 (Langmuir model) and from 0.996 to 0.998 (Freundlich model). Adsorption isotherm data explained that for PHE and phenol adsorption on BMPCs, Langmuir model gave better fit than Freundlich model; however, for 2,4,6-TCP adsorption on BMPCs, Freundlich model fits better than Langmuir model, and Langmuir model fits well in 2,4,6-TCP adsorption on BMPC-800. KL, Langmuir constant as a measure index of affinity between adsorbent and adsorbate, increased significantly with pyrolysis temperature increase for PHE and phenol sorption isotherm, suggesting that the adsorption capacities increased from 16.69 to 21.91 mg·g⁻¹ (for PHE) and from 90.9 to 106.4 mg·g⁻¹ (for phenol); however, for 2,4,6-TCP adsorption, KL values were nonlinear with pyrolysis temperature, resulting in the increased the adsorption capacities from 263 to 303 mg·g⁻¹ (at 500–700 °C of pyrolysis temperature), and adsorption capacity decreased to 270 mg·g⁻¹ at 800 °C. KF and 1/n, the Freundlich constants corresponding to adsorption capacity and adsorption intensity, represented that adsorption was favorable; in here, all the 1/n values were below 1 and decreased nonlinearly, and KF values changed linearly with pyrolysis temperature. Based on the hypothesis of Langmuir isotherm, adsorption occurs at a specific uniform surface position within the adsorbents and is considered as single-layer adsorption; meanwhile, Freundlich isotherm is an empirical equation, describing the adsorption on heterogeneous surfaces (Lawal et al. 2021; Wang et al. 2020). In previous studies, surface adsorption seems likely the dominant mechanism at low concentrations (especially, below 1% of its solubility) (Kong et al. 2011).

### Adsorption mechanism

In order to clarify the adsorption mechanism of three adsorbates on BMPCs, FTIR spectra before and after sorption were performed. As shown in Fig. 6, the position and intensity of FTIR peaks shifted significantly after adsorption. The peaks at 1443 cm⁻¹ and 1245 cm⁻¹ assigned to =CH₂ band (aromatic ring) and C-O stretching band were shifted to 1467 cm⁻¹ (for 2,4,6-TCP), 1469 cm⁻¹ (for phenol), 1226 cm⁻¹ (for phenol and 2,4,6-TCP), and 1243 cm⁻¹ (for PHE), respectively; in here, not only the position of the peaks was shifted, but also was increased significantly in the intensity of the vibration (Pei et al. 2013; Wo et al. 2018; Zhao et al. 2018). This study suggested that three adsorbates attended on the adsorption of BMPC-500 through π–π interaction and O-containing functional groups played in sorption. With the increase of pyrolysis temperature, even though the positions of the BMPC functional groups shifted in a little range, FTIR study of BMPCs was similar to that of BMPC-500 after adsorption of three adsorbates (Fig. 6b, c, and d). This explained that specific surface area was the main factor affecting the adsorbent adsorption in this study, and π–π interaction and functional groups such as carboxylic –COOH, aromatic C=C, and so on enhanced the adsorption characteristics of adsorbents, possibly due to O-containing groups’ exposure after ball-milling (Lyu et al. 2018b), and hydroxyl groups of phenol and 2,4,6-TCP could interact with O-containing groups on BMPCs through H-bonding (Pei et al. 2013). Furthermore, the peak intensity between 1000 and 1800 cm⁻¹ got stronger after 2,4,6-TCP and phenol adsorption than after PHE adsorption, which indirectly explained a large difference in adsorption of three aromatic organic pollutants (Pei et al. 2013).

The feasible reason why there appeared the difference of adsorption capacity for three pollutants could be explained as follow. Generally, the adsorption of the aromatic organic pollutants is not only related to the adsorbent properties (surface area, pore size distribution, and surface functional groups) and the solution chemistry (pH,
ion strength, dosage of absorbents and contact time, and reaction temperature) but also related to the physicochemical properties of the adsorbates (molecular structure and weight, hydrophobicity, polarity, and substituent groups) (Luo et al. 2022). Unlike phenol and 2,4,6-TCP, PHE is hydrophobic nonpolar substance with three benzene rings. Previous studies have suggested that the adsorption of hydrophobic organic pollutants on adsorbents is predominantly a surface action, which is related to the surface properties (Peng et al. 2016; Shen et al. 2017), and the equilibrium adsorption capacity of polycyclic aromatic hydrocarbons decreased with the increase of benzene ring number (Zhou et al. 2022). In addition, the adsorption capacities for phenol and 2,4,6-TCP are function of molecular weight and cross-sectional area (Laszlo and Szucs 2001) and are in direct proportion to the polarity of adsorbate. With the increase of chlorination degree, the adsorption capacity also increases. Due to chlorine group as an electron-withdrawing group, the increase of chlorine base resulted in lower the electron density in the aromatic ring (Denizli et al. 2004; Hamdaoui and Naffrechoux 2007), which led to enhanced adsorption.

Conclusions

BMPCs prepared by pyrolysis of PET plastics at 500–800 °C could be used to adsorb aromatic compounds such as PHE, phenol, and 2,4,6-TCP. With the increase of pyrolysis temperature, the specific surface area of PCs increased significantly; however, the adsorption performance of PCs was inferior to that of BMPCs. The ball-milling method enhanced the adsorption effect of PHE, phenol, and 2,4,6-TCP in aqueous solutions due to increase the specific surface area of PCs and exposure of its surface functional groups. With the increase of pyrolysis temperature, the adsorption capacity of BMPCs for PHE, phenol, and 2,4,6-TCP generally increased; however, the adsorption capacity of phenol and 2,4,6-TCP onto BMPC-800 was lower than that of BMPC-700. And there were significant differences in the adsorption capacity of BMPCs for the three substances mostly due to the difference of physicochemical properties. Recently, the amount of plastic waste in the world has been explosively increasing, and how to treat plastic waste controllably, efficiently, and future-oriented is the great challenge facing to human community. The method proposed
in this paper has certain guiding significance for the pyrolysis treatment of PET plastic wastes and its application and for the adsorption removal of organic pollutants in water environment.

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Declarations

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