



Removal of Pesticides from Water by Adsorption on Activated Carbon Prepared from Invasive Plants

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Abstract Pesticide pollution of surface water is a serious global problem. This research was focused on the monitoring of pesticides in surface waters and their subsequent removal using adsorption on activated carbon (AC). Based on the monitoring, four pesticides—acetamiprid, diethyltoluamide, thiacloprid and thiamethoxam—occurred in higher concentrations in all sampling points. Invasive plants occurring near monitored water bodies, *Reynoutria japonica* (RJ) and *Impatiens glandulifera* (IG) were used for the preparation of activated carbon with an activating agent (H_3PO_4 and NaOH) using microwave pyrolysis. The prepared AC was subsequently used for adsorption of the above-mentioned pesticides. The prepared AC was characterized by Brunauer–Emmett–Teller analysis, elemental analysis, Fourier transform infrared spectroscopy and scanning electron microscopy. Individual AC types showed different effects for different pesticides.

The maximum adsorption capacity calculated from the Langmuir model was 18.30 mg g^{-1} for thiacloprid on H_3PO_4 -activated AC from *I. glandulifera*.

Keywords *Reynoutria japonica* · *Impatiens glandulifera* · Activated carbon · Pesticides · Adsorption

1 Introduction

I. glandulifera and *R. japonica* currently belong to problematic invasive plant species in Europe. Both plants were originally imported to Europe as ornamental plants. *I. glandulifera* was imported from the western Himalayas, whereas *R. japonica* is native to Asia (Helsen et al., 2021; Stefanowicz et al., 2021).

Invasive plants have become a global problem. These are mainly fast-growing plants that affect the composition of natural species when they spread into nature. Their negative impact on the ecosystem has been proven. They are able to change the properties of the soil and the processes that take place in it. Invasive plants often alter the ion and water content of the soil, thus influencing the hydrological conditions and salinity of the habitats and preventing the growth of naturally occurring plants. In addition, some species are at risk of hybridisation with closely related wild species and the creation of new invasive organisms (Li et al., 2004). *I. glandulifera* contains an allelopathic substance in its leaves, which has strong inhibitory effects on the

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growth of mycorrhizal fungi, as well as on the germination of native herbs. This substance is released into the soil (Gaggini et al., 2018). *R. japonica* is one of the 100 worst invasive species in the world. Its occurrence also leads to changes in the physicochemical properties of the soil, while it can increase or decrease the amount of nutrients in the soil (Zubek et al., 2022).

Efforts to eliminate invasive species are still growing, but this is often laborious and expensive. Disposal methods are primarily of a physical or chemical nature. Physical processes include, for example, cutting, and pulling, and the chemical process uses pesticides, which are already problematic (Wei et al., 2023). However, pesticides in the environment pose a risk to nontarget organisms, especially in the aquatic environment. The main threats are microorganisms, invertebrates, plants, and fish (Le et al., 2017). Pesticides also pose a risk to amphibians due to their permeability through their skin and eggs, which are shell-less. Amphibians lose their ability to swim, and their ability to escape predators also decreases. Exposure to pesticides does not directly cause their mortality but threatens their existence (Guo et al., 2022). Therefore, new methods and procedures are being sought for the use of these plants.

Based on their large use, the priority insecticides selected in this work are acetamiprid, diethyltoluamide (DEET), thiacloprid, and thiamethoxam. Acetamiprid is a broad-spectrum neonicotinoid pesticide that is used to control pests in rice, fruits, and vegetables. Acetamiprid has been found in surface waters around the world at concentrations of ng L^{-1} to $\mu\text{g L}^{-1}$ (Guo et al., 2022). DEET is one of the most detected pesticides in waters. It is mainly used as an insect repellent, and is part of sprays, lotions, and liquids. Its concentration in products is up to 100% (Marques Dos Santos et al., 2019). Thiacloprid is a neonicotinoid and neuroactive pesticide. It is used against sucking and chewing pests in pome fruits, vegetables, cotton, oilseed rape, potatoes, rice, cereals, rice, and ornamental plants. Thiamethoxam is the second best-selling neonicotinoid insecticide, widely used against sucking, chewing, and soil-dwelling insects. It is used for seed treatment. The main crops are vegetables, fruits, cotton, potatoes, rice, tobacco, and cereals (Elbert et al., 2008).

Adsorption is currently one of the most widely used methods for water purification. Adsorption brings many advantages, mainly low cost, high

efficiency, easy implementation and operation, and the possibility of reusing both the adsorbent and the adsorbate (Dotto & McKay, 2020). A large number of activated carbon-based materials (AC) are used for pesticide adsorption. A study dealing with the adsorption of acetamiprid used AC prepared from pistachio shells (Dolatabadi et al., 2021), a study dealing with a wider spectrum of pesticides used rice husk, date pit, and sugar cane bagasse for AC preparation (Eissa et al., 2023). AC prepared from sewage sludge has already been used for pesticide adsorption (Sanz-Santos et al., 2022).

The aim of the work is to use the biomass of invasive plants for the microwave-assisted production of a carbonaceous sorbent and to test its use for the removal of pesticides found in surface water in the Czech Republic, i.e. acetamiprid, diethyltoluamide (DEET), thiacloprid, and thiamethoxam.

2 Materials and Methods

2.1 Monitoring of Pesticides in Surface Water

Monitoring of pesticides in surface water was carried out once a month for 12 months. Experimental sites included rivers, small watercourses, and ponds around the city of Ostrava, Czech Republic. 1 L of water was taken from each site, which was further subjected to LC–MS/MS analysis (see Section 2.5). In total, 95 different pesticides (Tab. S1) were analysed in each sample. Pesticides acetamiprid, DEET, thiacloprid, and thiamethoxam were found in most samples and therefore were selected for the needs of this work.

2.1.1 Analysis of Water Samples

Water samples were collected in screw-on glass containers. They were kept in a refrigerator at a temperature of 4 °C until analysis. The water samples were freed of unwanted impurities using a vacuum filtration apparatus with microfilters of 47 mm size, for a particle size of 1.2 μm . Subsequently, solid phase extraction was performed using EnviroElut Pesticides SPE extraction columns (Agilent, USA), which were placed in a manifold with an attached vacuum pump.

Solid-phase extraction (SPE) is a powerful technique for the targeted isolation of an analyte from a

sample and its subsequent concentration, with a small sample volume being sufficient for analysis. Extraction columns are formed by a sorbent on which the given analyte is captured due to its physical and chemical properties. Sorbents are chosen according to the properties of the analyte (Cremlyn, 1985).

The extraction was carried out in several steps. Firstly, conditioning of SPE columns was performed with 2 mL MeOH and 2 mL H₂O. Then, 100 mL of water sample was applied to the column with the addition of 25 µL of an internal pesticide standard of $c = 1 \mu\text{g mL}^{-1}$. A flow rate of approximately 5 drops per minute was used to ensure a sufficiently long sample retention time. Subsequently, a 10-min vacuum was applied to dry the column. In the last step, elution was carried out using 2 mL of MeOH in plastic disposable tubes.

The solution was evaporated to dryness using a stream of nitrogen in a sample concentrator (Stuart, Cole-Parmer, USA). The residue was dissolved in 100 µL H₂O. An HPLC gradient liquid chromatograph (Shimadzu, Japan) with a QTRAP 6500+ mass detector (Sciex, Canada) was used for analysis of pesticides (see Sect. 2.5).

2.2 Selected Pesticides

Acetamiprid (Fig. 1) is a broad-spectrum insecticide from the chlorpyridinyl subclass of neonicotinoid insecticides that acts on nicotine receptors in the central nervous system of insects. The main crops to which acetamiprid is applied are rice, fruits and vegetables. This pesticide is very often detected in water sources, where it shows potential toxicity for the environment, as well as for humans. Research

dealing with the effect of acetamiprid on non-target organisms has demonstrated the induction of oxidative stress, disruption of amino acid metabolism and neurotransmitter homeostasis in zebrafish (Elango et al., 2023; Guo et al., 2022).

Diethyltoluamide (DEET) (Fig. 2) represents one of the most and most frequently detected pesticides, the detected concentration ranges from ng L^{-1} to $\mu\text{g L}^{-1}$. It is mainly used as an insect repellent, which is part of sprays, lotions and liquids. The concentration of DEET in products reaches up to 100%. DEET has potential carcinogenic properties in the cells of the nasal mucosa. Even in small doses, it can cause coma and seizures in children. It is only slightly toxic to aquatic invertebrates and freshwater fish (Cai et al., 2022; Marques Dos Santos et al., 2019).

Thiacloprid (Fig. 3) also belongs to the group of neonicotinoids. It was already banned in the EU for outdoor use because it poses a risk to bees. It produces sub-lethal effects along with behavioral changes that affect foraging, navigation and homing ability in bees. It is also harmful to bumblebees, specifically to their immune system and colony development. It is applied to boulders, stone fruits, vegetables, and potatoes for their protection. Through runoff from the fields, it reaches water sources, where it is accessible to aquatic organisms due to its solubility. It causes pathological changes in aquatic organisms, as well as behavioral changes, e.g. in crayfish it leads to aggressive behavior, in carp it reduces their weight and length (Fent et al., 2020; Stara et al., 2021).

Thiamethoxam (Fig. 4) also belongs to the group of neonicotinoid insecticides of the second generation. It is one of the most widespread and used insecticides in agriculture. It is mainly used against pests that have sucking or biting mouthparts, such as aphids, whiteflies, potato beetles, also against flies, etc. Like thiacloprid, it has been banned in the EU for outdoor applications. It is relatively stable

Fig. 1 Chemical structure of acetamiprid

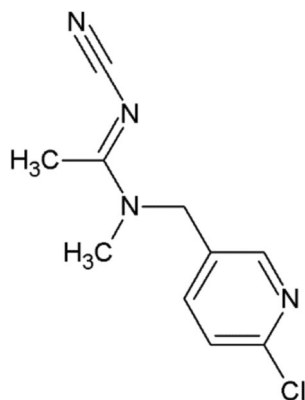
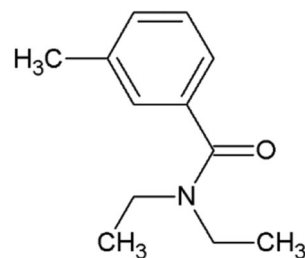


Fig. 2 Chemical structure of DEET



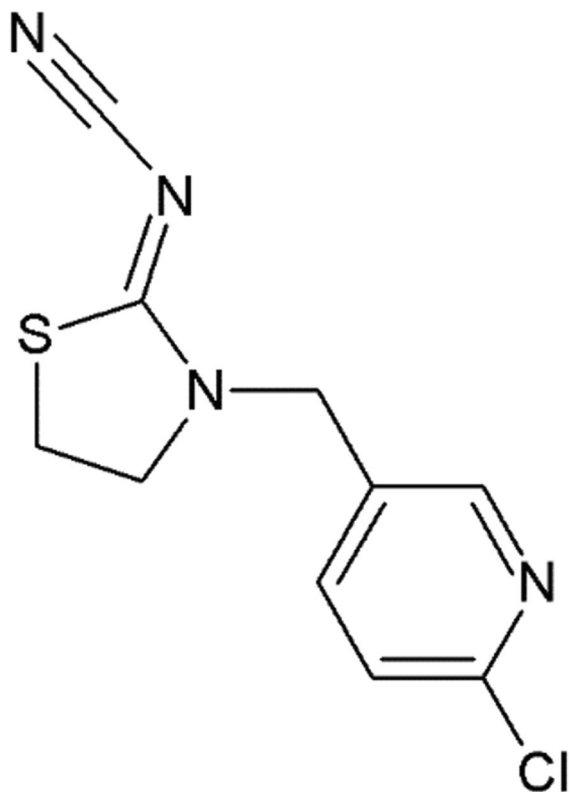


Fig. 3 Chemical structure of thiacloprid

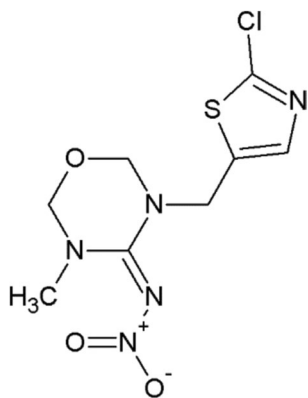


Fig. 4 Chemical structure of thiamethoxam

in the aquatic environment, up to 600 days at pH 7. Long-term exposure to it can lead to oxidative stress in silver catfish (Fent et al., 2020; Yang et al., 2023).

2.3 Chemicals and Materials

Sodium hydroxide (purity $\geq 98\%$) and hydrochloric acid (purity 35%) were purchased from PENTA (Czech Republic), phosphoric acid (purity 85%) was purchased from Lach-ner (purity 85%), methanol for LC–MS (purity 99.95%) purchased from Biosolve (Netherlands), ammonium formate for LC–MS (purity 98 – 100%), acetamiprid (purity $\geq 98\%$), DEET (purity $\geq 95\%$), thiacloprid (purity $\geq 98\%$), thiamethoxam (purity $\geq 98\%$) were purchased from Sigma-Aldrich (USA).

2.4 Preparation of AC from *Reynoutria japonica* and *Impatiens glandulifera*

R. japonica was harvested in the vicinity of the Kateřinský stream (49.9503281N, 17.9302511E) and around the banks of the Odra River (49.9484169N, 18.3331581E). *I. glandulifera* was harvested near the banks of the Opava River (49.8444589N, 18.1966439E). All locations are located in the Moravian-Silesian Region of the Czech Republic, in the basin of the Odra River, which is an important water-course originating in the Czech Republic and flowing through Poland to the Baltic Sea.

After harvest, both plants were dried to constant weight at 105 °C, and then crushed into sawdust. First, carbon was prepared by conventional pyrolysis at 800 °C with heating of 10 °C min⁻¹ for 1 h. The carbon thus prepared was added to the flask with the impregnated material. Borosilicate glass flasks were used for microwave pyrolysis. Each plant was impregnated with NaOH or H₃PO₄ in a biomass to activator ratio of 1:2. Impregnation occurred for 24 h. The samples were further dried prior to microwave pyrolysis due to the high water content. Microwave pyrolysis was performed under nitrogen flow for 20 min. The power of the microwave reactor was set to 400 W for *R. japonica* and 600 W for *I. glandulifera*. After the samples were cooled to room temperature, they were washed on a glass fibre filter using deionised water and HCl to neutral pH. The obtained material was dried to a constant weight at 105 °C and then sieved through a set of sieves to a size below 0.09 mm.

The different powers of the microwave reactor were chosen based on the previous experiment. Each material was first subjected to microwave pyrolysis without the presence of an activator. Three

performances were tested, 200; 400, and 600 W, of which the one that provided a larger specific surface area was chosen.

2.5 Methods of Adsorbent Characterisation

2.5.1 Brunauer–emmett–Teller (BET) Analysis

The 3Flex physisorption apparatus (Micromeritics, USA) was used to determine the structural properties of the prepared activated carbon. Based on the physisorption of nitrogen at 77 K, the specific surface area S_{BET} , the surface area of the mesopores S_{meso} , the volume of micropores V_{micro} , the size distribution of micropores, mesopores, and macropores, and the volume of pores V_{net} were determined.

2.5.2 Fourier-transform Infrared Spectroscopy (FTIR)

The functional groups on the activated carbon surface were analysed using Fourier transform attenuated total reflectance infrared spectroscopy (ATR-FTIR, Diamond) with a Nicolet iS10 (ThermoScientific, USA). The spectrum was measured in the wavenumber range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1} . The sample was measured in the form of tablets, which were formed by thoroughly mixing and homogenising the material with KBr, which was in multiple excess.

2.5.3 Elemental Analysis

The elemental analyzer CHN628 (LECO) was used to determine the content of elemental carbon, hydrogen and nitrogen by the combustion in the temperature range of 950 – 1050 °C. Thanks to pure oxygen in the furnace, the material was burned perfectly. The use of infrared cuvettes also made it possible to determine the content of H_2O and CO_2 in the sample.

2.5.4 Scanning Electron Microscopy

The morphology of the activated carbon surface was investigated using a scanning electron microscope (SEM, Vega, Tescan, Czech Republic) with

a Tungsten cathode. Images were obtained using a combination of secondary electrons and backscattered electrons with an accelerating voltage of 15 keV. Before the measurements, the samples were sputtered with gold to ensure sufficient electronic conductivity of the material surface.

2.5.5 Liquid Chromatography—Mass Spectrometry (LC–MS/MS)

An HPLC gradient liquid chromatograph (Shimadzu, Japan) with a QTRAP 6500+ mass detector (Sciex, Canada) was used for the analysis of pesticides. An electrospray was used as an ion source. A Synergy Fusion RP 80Å (50×2 mm, 4 μm) column (Phenomenex, USA) was used for the analysis, which allows the analysis of a mixture of polar and non-polar substances. 5 mM ammonium formate in MeOH (MF A) and 5 mM ammonium formate in H_2O (MF B) were used as mobile phases. Gradient MF B: 0→0.5: 90% B; 0.5→1: 60% B; 1→8: 10% B; 8.2→9: 90% B. The analysis was based on the identification of MRM transitions.

2.6 Adsorption and Evaluation Experiments

Kinetic experiments were carried out by adding 5 mg of prepared AC to 50 mL of each adsorbate solution (1 mg L^{-1}). The mixture was stirred at 500 rpm and the samples were taken at 5-min intervals. Samples were purified from AC residues using injection filters (Swinnex Filter Holder 13 mm; Sigma-Aldrich, USA) and the concentration was subsequently determined using LC–MS/MS. The data obtained were subsequently evaluated using the selected diffusion model. Based on the calculated regression parameter, it is possible to reveal the mechanism of the adsorption process in the adsorbent/adsorbate system, so the models describe the kinetics of the ongoing process. Each kinetic model consists of several steps that describe the adsorption process, therefore, it is possible to estimate the driving step of the entire process. The experimental data were compared with basic kinetic models of pseudo-first (PFO) and pseudo-second (PSO) order.

The PFO model describes the adsorption rate on the basis of the adsorption capacity, and the adsorption rate is determined by physisorption. The PFO model can be described by Eq. (1).

The PSO model considers chemisorption to be the rate-limiting step of adsorption. The PSO model can be expressed by Eq. (2). The parameter h , which describes the rate of adsorption, can then be calculated using Eq. (3) (Qiu et al., 2009).

$$q_t = q_e(1 - e^{-k_1 t}) \quad (1)$$

$$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t} \quad (2)$$

$$h = k_2 q_e^2 \quad (3)$$

where q_e , q_t (mg g^{-1}) indicate the amount of adsorbate on the surface of the adsorbent when equilibrium is reached during the contact time t (min), k_1 (min^{-1}) and k_2 ($\text{g mg}^{-1} \text{min}^{-1}$) are rate constants, h ($\text{mg g}^{-1} \text{min}^{-1}$) represents the rate of adsorption.

Equilibrium experiments were carried out by adding 5 mg of AC to 50 mL of solution with increasing pesticide concentration (0.01–1 mg L^{-1}). The samples were left on the shaker for 200 min, a time chosen based on kinetic experiments. The equilibrium concentration was then determined by LC–MS/MS. All experiments were carried out at room temperature in an air-conditioned laboratory. The data obtained were processed and fitted with adsorption models. The adsorption isotherms describe the process of pollutant distribution between the liquid and solid phases at a constant temperature and pH, and the dependence of the amount of substance adsorbed on the surface of the adsorbent when the equilibrium concentration of the solution is reached. Interpretation of the resulting curves is an important parameter in understanding the mechanism of the ongoing sorption experiment. The Langmuir and Freundlich models were chosen to evaluate the adsorption isotherms (Foo & Hameed, 2010).

The Langmuir model is based on several assumptions. Adsorption occurs in a monolayer, and since the adsorbed molecules do not interact with each other, adsorption can only occur on specific homogeneous sites, the number of which is finite. The Langmuir isotherm in non-linear form is described by Eq. (4).

Unlike the Langmuir model, the Freundlich model assumes multilayer adsorption on a heterogeneous surface. Its nonlinear shape is described by Eq. (5).

$$Q = q_m \frac{k_L C_e}{1 + k_L C_e} \quad (4)$$

$$Q = k_F C_e^{\frac{1}{n_F}} \quad (5)$$

where C_e (mg L^{-1}) represents the equilibrium concentration, k_L (L mg^{-1}) represents the Langmuir isothermal constant, k_F (mg g^{-1}) represents the Freundlich isothermal constant, n_F represents the Freundlich nonlinearity parameter, q_m (mg g^{-1}) represents maximum sorption capacity (Foo & Hameed, 2010).

3 Results and Discussion

3.1 Monitoring of Pesticides in Surface Water

Figure 5 shows the concentrations of the four selected pesticides at each sampling point in each month. The GPS coordinates of each sampling point are recorded in Table 1. Pesticide monitoring was conducted over 12 months to see the effect of the seasonal agricultural period. The increase in pesticide concentration in surface water occurred mainly in the summer months, especially for DEET. The increase was due to increased consumption of repellents, which are widely used in these months. Conversely, in the winter months, when no substances are applied, the incidence of pesticides was minimal.

The highest concentration of acetamiprid was measured in July 2023 at the sampling point Bohuslavice—Bohuslavický potok (Stream), 17.1 $\mu\text{g L}^{-1}$. For DEET, thiacloprid and thiamethoxam, the highest increase occurred in April 2024, for DEET to 507.5 $\mu\text{g L}^{-1}$ at the sampling point Bohumin—Odra (River), for thiacloprid to 9.3 $\mu\text{g L}^{-1}$ at the sampling point Ostrava—Opava (River) and for thiamethoxam to 164.0 $\mu\text{g L}^{-1}$ at the sampling point Bohumín—Odra (River).

3.2 Physisorption Characterisation

The shape of the adsorption–desorption isotherms of nitrogen on prepared carbon sorbents from different materials activated by different chemical activators corresponds to a mesoporous-macroporous material in which some micropores occur (Figs. 6 and 7).

Sorbents prepared from *I. glandulifera* (IG) showed a higher S_{BET} compared to sorbents from *R. japonica* (RJ) (Table 2), especially in the case of using H_3PO_4 as an activating agent. The sorbents prepared by activation with H_3PO_4 showed a higher content of mesopores and macropores, as well as a higher content of micropores. However, the ratio of the micropore content to the total pore content was not very high; 18% in the case of AC-RJ- H_3PO_4 and 13% in the case of AC-IG- H_3PO_4 . In contrast, sorbents prepared using NaOH as an activating agent showed a smaller content of mesopores and macropores. The micropore content in comparison to the total pore content was higher; in the case of AC-RJ-NaOH 31% and in the case of AC-IG-NaOH 37%. Therefore, it can be assumed that the activation agent used affected the development of the porous structure of the given sorbent. In the case of comparing the effects of H_3PO_4 and NaOH, acid activation had a better effect on the development of the structure.

3.3 Elemental Composition and Surface Functional Groups Characterisation

Elemental composition of the prepared activated carbon was determined (Table 3). All of the prepared AC showed a large amount of carbon (79–87%). The sorbents prepared by activation with NaOH showed the highest carbon content, in *R. japonica* 87.42% and *I. glandulifera* 83.71%. The sorbents prepared by H_3PO_4 activation contained 79.37% and 80.15% of carbon in *I. glandulifera* and *R. japonica*, respectively. The hydrogen content was the opposite, higher amounts were contained in sorbents activated with H_3PO_4 . The results were as follows, AC-RJ- H_3PO_4 3.84%, AC-IG- H_3PO_4 3.57%, AC-RJ-NaOH 1.54% and AC-IG-NaOH 2.16%. The nitrogen content differed only slightly, the sorbents activated with NaOH showed a higher content, AC-RJ-NaOH 1.10%, AC-IG-NaOH 1.02%, AC-RJ- H_3PO_4 0.86% and AC-IG- H_3PO_4 0.83%. Sorbents with a lower carbon content show a higher content of oxygen functional groups, which help the subsequent adsorption because they show a higher sorption capacity (Ang et al., 2020).

The characterisation of the surface functional groups was performed using the FTIR technique (Figs. 8 and 9). The material activated by H_3PO_4 and NaOH showed peaks in similar regions. For

all prepared materials, there were peaks around $3600\text{--}3200\text{ cm}^{-1}$, which can be attributed to O–H vibrations. O–H groups are present here from absorbed water and phenyl groups (Ofgea et al., 2022). Other peaks were located around $2930\text{--}2850\text{ cm}^{-1}$, this is the –CH section, which indicates the presence of methyl and methylene groups (Kumar & Jena, 2016). Peaks in the region around $1650\text{--}1610\text{ cm}^{-1}$ correspond to C=O vibrations of lactone, carboxyl, or anhydride groups (Le Van & Luong Thi, 2014). Bands in the region $1300\text{--}900\text{ cm}^{-1}$ are attributed to C–O stretching in acids, alcohols, phenolic ethers, and esters, which are usually found in oxidized carbons. The peak in this region can also be attributed to phosphorus species, which would correspond to the material prepared by H_3PO_4 activation. It can be P=O, stretching vibration of the O–C group in P–O–C aromatics, and P=OOH. The last peak in the region $610\text{--}550\text{ cm}^{-1}$ corresponds to out-of-plane bending vibrations of –OH (Kumar & Jena, 2016). In the case of the material prepared from *R. japonica*, there is also a peak around the value of 2370 cm^{-1} in the spectrum, which could be attributed to C≡C stretching vibrations in the alkyne groups, which are released when the material is heated in the form of light volatile substances (Yang & Qiu, 2010).

3.4 SEM Characterisation

SEM images of the plant material show the development of a porous structure in the chemically activated samples (Fig. 10), while the surface of the H_3PO_4 -activated samples appears more porous than the NaOH-activated samples, which is also evidenced by the specific surface area values (Table 2). The remaining tissue from the original plant is also visible, especially in specimens of *I. glandulifera* (Fig. 10f). Mechanisms of chemical activation of the material using H_3PO_4 and NaOH have been described in the literature for various biomass-based input materials, while the properties of the resulting carbonaceous materials have been documented, among other methods, by SEM characterisation. (Myglovets et al., 2014) described the preparation of carbonaceous adsorbents from lignosulfonate by H_3PO_4 activation and explained the mechanism of the formation of volatile phosphorus compounds during activation. In the work, SEM images of carbonaceous materials obtained at different temperatures ($400\text{--}800\text{ }^\circ\text{C}$)

were presented, in which the effect of temperature on porosity was visible (the specific surface area decreased with increasing temperature). The effect of increasing the concentration of NaOH on the formation of micropores during the chemical activation of waste lignin was demonstrated using SEM images (Ngamthanacom et al., 2020). (Kra et al., 2019) studied the preparation of wood-based sorbents by activating H_3PO_4 and NaOH. In the case of activation with H_3PO_4 , a larger specific surface area was achieved than with the use of NaOH; however, in both activation methods mainly micropores were formed, and microscopic images revealed a relatively similar structure of the sorbent in the case of using both activators.

3.5 Kinetic Experiments

The adsorption kinetics of acetamiprid, DEET, thiacloprid, and thiamethoxam on the prepared carbon sorbents are shown in Fig. 11. The adsorption proceeded very quickly from the beginning, in all cases. The largest amount of adsorbate was adsorbed during the first 80 min, and then the adsorbed amount increased only very slowly. Steady state occurred after 100 min.

All calculated parameters and values of adjusted R-squared (adj. R^2) for PFO (Fig. S1) and PSO is shown in Table 4. For all adsorbent/adsorbate systems, the PSO model had the best criteria. Individual sorbents showed significant differences for individual pesticides. In the case of acetamiprid, the AC-IG- H_3PO_4 sorbent showed the highest adj. R^2 value (0.984), while the AC-IG-NaOH sorbent showed the lowest value (0.906). For DEET, the highest value was for AC-IG-NaOH (0.981) and the lowest was for AC-RJ-NaOH (0.641). For thiacloprid, it was highest for AC-RJ- H_3PO_4 (0.990) and lowest for AC-IG-NaOH (0.820). For the last pesticide, thiamethoxam, it was highest for AC-IG-NaOH (0.988) and lowest for AC-RJ-NaOH (0.779).

3.6 Adsorption Experiments

In the case of adsorption experiments, the Langmuir model (Fig. 12) showed a higher adj. R^2 value (Table 5) for most of the adsorbent/adsorbate system, only in some cases the Freundlich model (Fig. S2) has a higher value compared to the Langmuir model.

All four substances were significantly better adsorbed on adsorbents prepared using phosphoric acid. These samples exhibited significantly higher porosity and a higher representation of surface oxygen-containing groups. IG- H_3PO_4 had the highest measured adsorption capacities based on the Langmuir model, which corresponds with the highest values of measured physisorption. Besides, RJ- H_3PO_4 had a higher representation of oxygen-containing groups, but this did not compensate for the influence of a narrower pore distribution and a lower content of meso and micropores.

Both samples activated by hydroxide had similar values of porosity measured by physisorption, so the differences in the measured adsorption of pesticides can be attributed to the influence of non-bonding forces caused by the presence of surface functional groups.

IG exhibited a higher content of lignin in its raw state (band at 1606 cm^{-1}), while RJ had a more distinct band at 1633 cm^{-1} indicating a higher presence of cellulose, which may lead to a higher tendency for the formation of turbostratic carbon allowing more functional groups to bind at the edges of graphite planes.

With NaOH activation, IG was about two times more efficient in adsorption of all adsorbates except thiamethoxam, where RJ-NaOH achieved three times higher adsorption capacity than IG-NaOH and its capacity was even close to IG- H_3PO_4 , which had significantly higher porosities measured by physisorption. The reason could be, with high probability, a higher content of COO^- (see band at 1438 cm^{-1}) in RJ-NaOH than in IG-NaOH. Combined with the fact that samples activated by NaOH adsorbed thiamethoxam the best out of all adsorbates, it seems likely that its good adsorption is due to the fact that thiamethoxam represents the most complex molecule of all adsorbates. From its structure, it is evident that it includes the largest number of polar parts readily accessible to interactions. Among the negative ones, there are nitrogen and chlorine of chlorothiazole, both oxygens of the nitro group, and partially the oxygen of the oxadiazine ring. Positive dipoles can be expected on the methylene linking the chlorothiazole ring with the oxadiazine, the carbons of the oxadiazine ring, and the methyl group bound to the nitrogen of the oxadiazine ring. This large number of dipoles will also increase the molecule's ability to interact

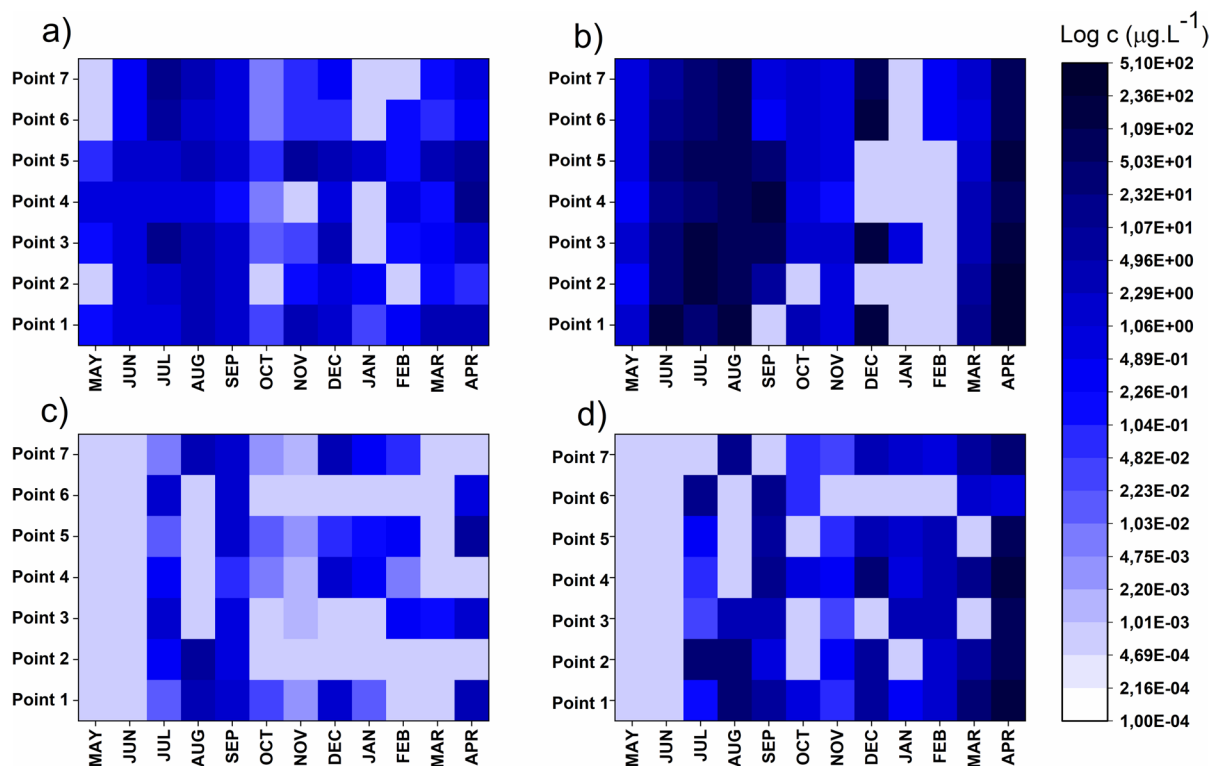


Fig. 5 Concentration of pesticides in surface water in individual locations and months: (a) Acetamidrid, (b) DEET, (c) Thiachlorid, (d) Thiamethoxam

with surfaces occupied by polar groups such as carboxyl and hydroxyl ones. The 2-chlorothiazole subunit exhibits aromatic properties and contains π -electrons delocalized unevenly partly due to the involvement of sulfur and nitrogen in the ring and partly due to the presence of chlorine in position 2. This results in unevenness and a slight reduction in the electron density of the π -electron system compared to benzene. The aromatic ring of 2-chlorothiazole will thus

be able to engage in π - π interactions with delocalized π -electrons or anion- π interactions with deprotonated carboxyl and hydroxyl groups of the adsorbent surface.

Acetamidrid showed the weakest adsorption on hydroxide-activated samples, likely due to the small influence of functional groups except for the RJ, where it is comparable to DEET.

Table 1 Description of collection points

Sampling point	Experimental site (city – name of the water body)	GPS coordinates
Point #1	Bohumín – Odra (River)	49.948417, 18.333158
Point #2	Bohumín—Záblatí (Pond)	49.880949, 18.373840
Point #3	Bohuslavice—Bohuslavický potok (Stream)	49.944131, 18.142446
Point #4	Opava—Kateřinský potok (Stream)	49.949389, 17.931167
Point #5	Ostrava – Opava (River)	49.839465, 18.198504
Point #6	Potštát – Harta (Pond)	49.634665, 17.638398
Point #7	Potštát—Rybník u hřiště (Pond)	49.640628, 17.646015

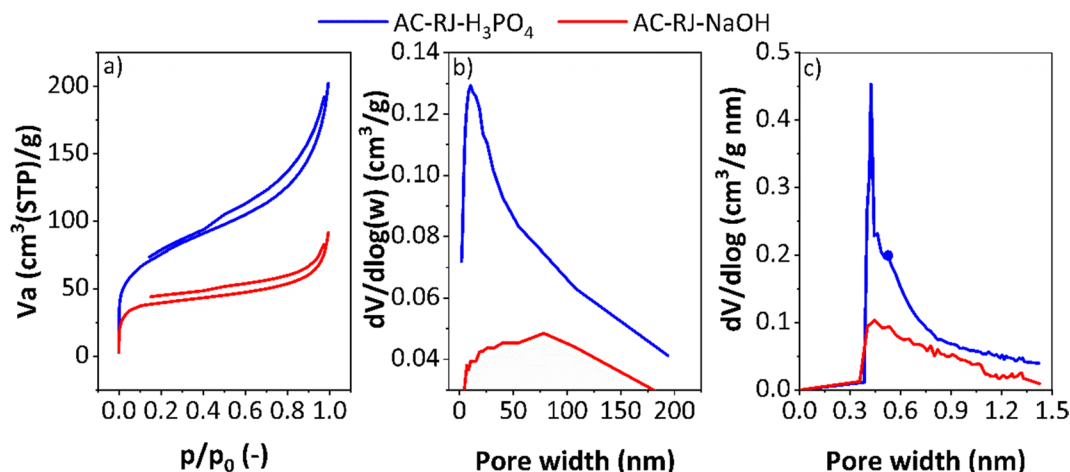


Fig. 6 **a** Measured nitrogen adsorption–desorption isotherms, **(b)** evaluated mesopore-size distributions and **(c)** micropore-size distributions of investigated activated carbons prepared from *Reynoutria japonica* (RJ) activated by H₃PO₄ or NaOH

Fig. 7 **a** Measured nitrogen adsorption–desorption isotherms, **(b)** evaluated mesopore-size distributions and **(c)** micropore-size distributions of investigated activated carbons prepared from *Impatiens glandulifera* (IG) activated by H₃PO₄ or NaOH

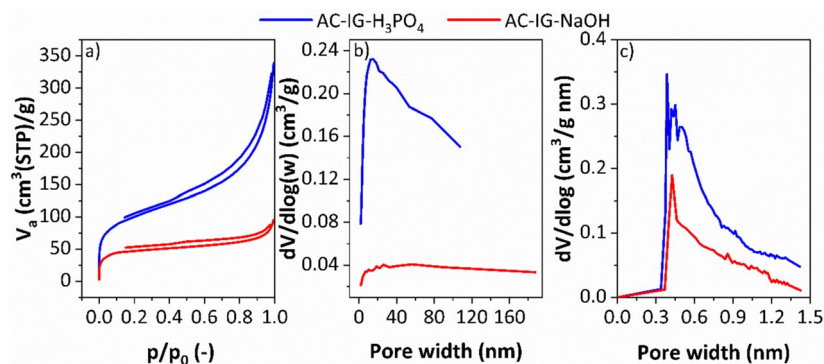


Table 2 Textural properties of investigated activated carbons prepared from *Reynoutria japonica* (RJ) and *Impatiens glandulifera* (IG) activated by H₃PO₄ or NaOH

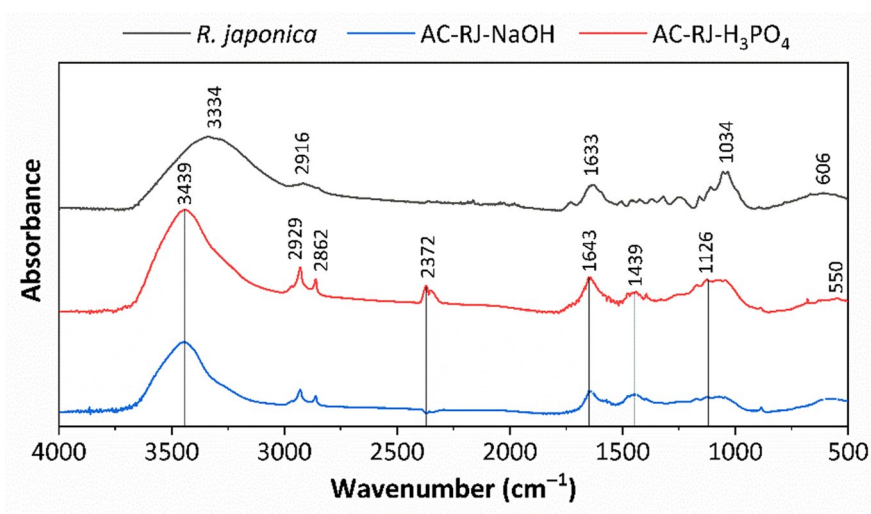
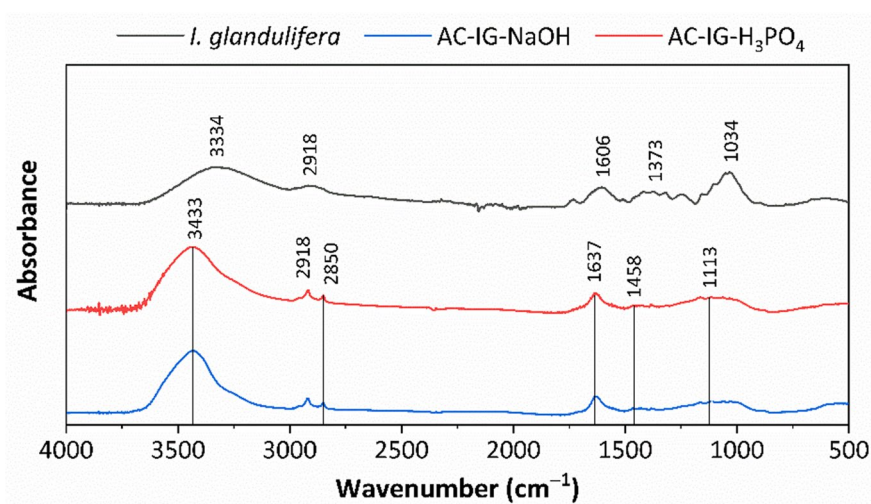
Material	S_{BET} (m ² g ⁻¹)	S_{meso} (m ² g ⁻¹)	V_{micro} (mm ³ liq g ⁻¹)	V_{net} (mm ³ liq g ⁻¹)	V_{micro}/V_{net} (%)
AC-RJ-H ₃ PO ₄	260	151	55	313	18
AC-RJ-NaOH	149	41	44	142	31
AC-IG-H ₃ PO ₄	347	205	68	524	13
AC-IG-NaOH	177	44	54	147	37

Thiacloprid is the second best on hydroxide-activated samples, thus showing better adsorption capabilities than structurally related acetamiprid. The interaction possibilities of thiacloprid, due to a similar molecular structure to acetamiprid, are similar. The difference compared to acetamiprid lies in the presence of a thiazolidine ring, through which a cyano

group is bound to a chloropyridine unit. Nitrogen and sulphur in this ring have a lone electron pair, which, together with their higher electronegativity compared to adjacent atoms, causes an increased electron density and the presence of a negative partial charge. The involvement of nitrogen in non-bonding interactions will be complicated by shielding by surrounding

Table 3 Elemental composition of activated carbons

Sample	C (wt. %)	H (wt. %)	N (wt. %)	W	A	C ^{daf} (wt. %)	H ^{daf} (wt. %)	N ^{daf} (wt. %)
AC-RJ-H ₃ PO ₄	54.19	2.61	0.60	8.70	22.82	79.14	3.81	0.87
	54.36	2.69	0.59	8.70	22.82	79.38	3.94	0.86
	54.51	2.59	0.58	8.70	22.82	79.60	3.79	0.84
AC-RJ-NaOH	61.57	1.11	0.77	4.26	26.02	88.31	1.63	1.13
	60.58	1.01	0.78	4.26	26.02	86.89	1.47	1.14
	60.70	1.03	0.70	4.26	26.02	87.06	1.51	1.03
AC-IG-H ₃ PO ₄	55.89	2.35	0.55	8.37	21.67	79.89	3.43	0.81
	56.26	2.47	0.58	8.37	21.67	80.41	3.60	0.85
	56.08	2.53	0.58	8.37	21.67	80.16	3.69	0.84
AC-IG-NaOH	61.23	1.51	0.73	7.43	19.46	83.75	2.21	1.06
	61.21	1.47	0.70	7.43	19.46	83.72	2.15	1.02
	61.16	1.46	0.67	7.43	19.46	83.65	2.13	0.97

Fig. 8 FTIR analysis of dry biomass (black) and activated carbons (red and blue) prepared from *Reynoutria japonica* (RJ) activated by H₃PO₄ or NaOH**Fig. 9** FTIR analysis of dry biomass (black) and activated carbons (red and blue) prepared from *Impatiens glandulifera* (IG) activated by H₃PO₄ or NaOH

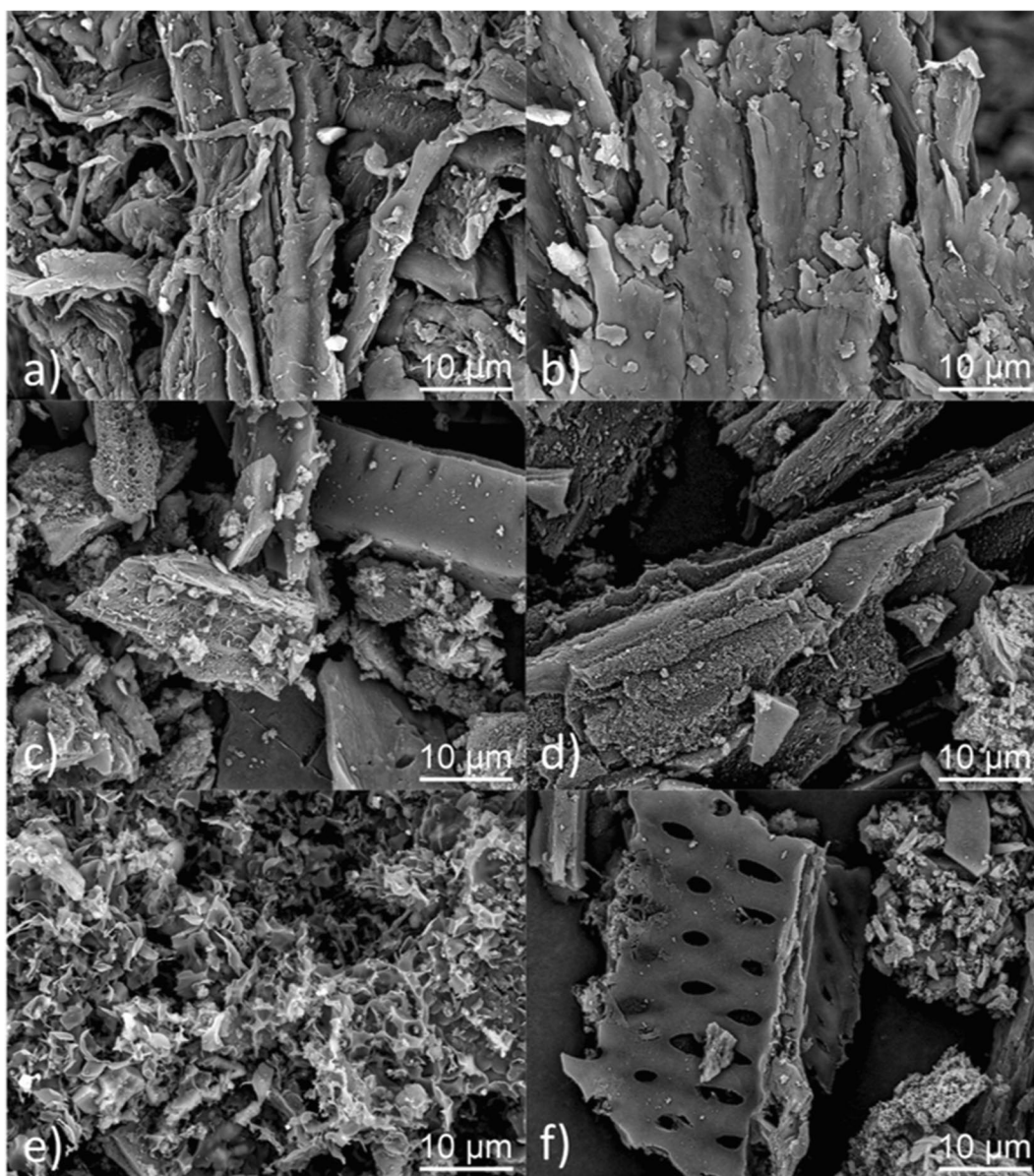


Fig. 10 Surface characterization: (a) *Reynoutria japonica*; (b) AC-RJ-H₃PO₄; (c) AC-RJ-NaOH; (d) *Impatiens glandulifera*; (e) AC-IG-H₃PO₄; (f) AC-IG-NaOH

atoms, but the spatial position of sulphur is more favorable. Moreover, due to the higher electronegativity of sulphur, there is a charge transfer from the adjacent carbon, creating a positively charged dipole easily accessible to bonds with deprotonated carboxyl and hydroxyl groups, or π -electrons of the adsorbent surface.

In samples activated by phosphoric acid, porosity had a fundamental influence on the adsorption

capacity of the selected pesticides, and the order for all adsorbates follows the results of physisorption, i.e., IG-H₃PO₄ adsorbs better than RJ-H₃PO₄, with the values for individual adsorbates within the selected adsorbent being similar to each other. From the above results, it can be concluded that the achieved porosity influenced the sorption capacity more significantly than the presence of polar groups on the surface of the sorbent. Adsorption is an effective method for

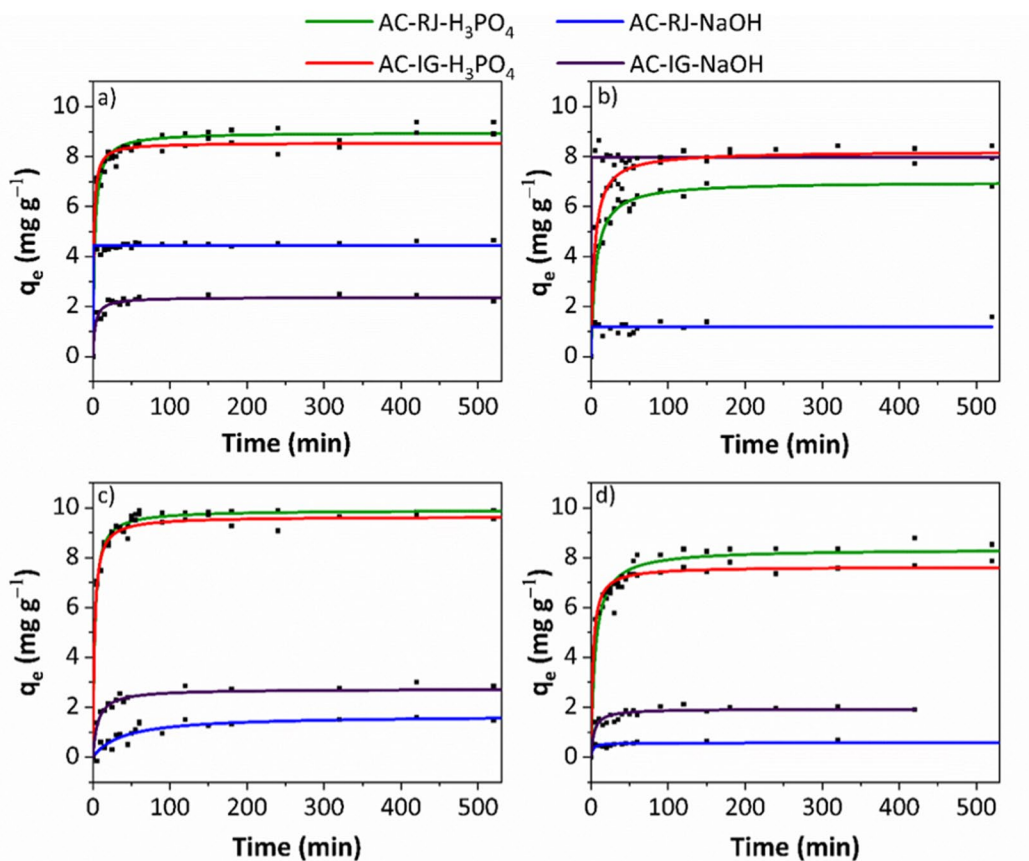


Fig. 11 Adsorption kinetics (pseudo-second order model) of (a) Acetamidrid (b) DEET (c) Thiachloprid (d) Thiamethoxam adsorbed on activated carbons prepared from *Reynoutria japonica* (RJ) and *Impatiens glandulifera* (IG) activated by H_3PO_4 or NaOH

removal of pesticides from water. (Sahraoui et al., 2022) studied the removal of acetamidrid by activated carbon from pomegranate waste in laboratory experiments. The authors achieved the best results with AC activated with H_3PO_4 and found a good correlation of data with both Langmuir and Freundlich models, which is consistent with our results. Unfortunately, their work lacks data for other pesticides. Eucalyptus woodchip-derived biochar was used for adsorption of imidacloprid, acetamidrid, and methomyl from water in (Srikhaow et al., 2022). In their work, the Langmuir model correlated well with the adsorption isotherm data for acetamidrid and methomyl, while the Freundlich model was selected to explain the adsorption of imidacloprid. Similar to our results, the mechanism of adsorption depended on the chemical nature of individual pesticides.

The adsorption of DEET on carbonaceous materials was studied by (Mitra et al., 2021), who used acid-leached carbon black waste (LCBW) for water treatment. According to their results, the adsorption kinetic of DEET followed the pseudofirst-order model, while in our work pseudosecond-order model was more common (Table 4). For 10 ppm DEET, 80% removal was achieved with 0.75 mg mL^{-1} LCBW in 24 h, which was the worst efficiency of all micropollutants studied by (Mitra et al., 2021). Biochar adsorption was used by (Yanala & Pagilla, 2020) for the reduction of trace organic contaminants, including DEET, from treated wastewater. In the experiments with the adsorption of 10 micropollutants on the column filled with biochar activated by NaOH, the adsorption capacity for DEET was found to be 1.160 mg g^{-1} , which is similar to NaOH activator used in this work. Our results show that better

Table 4 Parameters of kinetics models

	<i>Acetamiprid</i>				<i>DEET</i>			
	AC-IG- H ₃ PO ₄	AC-IG- NaOH	AC-RJ- H ₃ PO ₄	AC-RJ- NaOH	AC-IG- H ₃ PO ₄	AC-IG- NaOH	AC-RJ- H ₃ PO ₄	AC-RJ-NaOH
First Order Kinetics								
q_e (mg g ⁻¹)	8.35	2.26	8.56	4.45	7.70	7.96	6.44	1.18
k_1 (min ⁻¹)	0.35	0.17	0.25	12.75	0.14	11.86	0.10	3.73
adj. R^2	0.968	0.860	0.894	0.977	0.848	0.981	0.933	0.641
Second Order Kinetics								
q_e (mg g ⁻¹)	8.55	2.37	8.97	4.45	8.21	7.96	6.99	1.18
k_2 (g mg ⁻¹ min ⁻¹)	0.10	0.14	0.05	1.51	0.03	5.30	0.02	2.40
h (g mg ⁻¹ min ⁻¹)	7.31	0.79	4.02	29.9	2.02	335.82	0.98	3.34
adj. R^2	0.984	0.906	0.959	0.977	0.932	0.981	0.980	0.641
	<i>Thiacloprid</i>				<i>Thiamethoxam</i>			
	AC-IG- H ₃ PO ₄	AC-IG- NaOH	AC-RJ- H ₃ PO ₄	AC-RJ- NaOH	AC-IG- H ₃ PO ₄	AC-IG- NaOH	AC-RJ- H ₃ PO ₄	AC-RJ- NaOH
First Order Kinetics								
q_e (mg g ⁻¹)	9.31	2.52	9.52	1.48	7.32	1.79	7.81	0.53
k_1 (min ⁻¹)	0.21	0.11	0.21	0.02	0.21	0.20	0.11	6.88
adj. R^2	0.961	0.736	0.955	0.845	0.941	0.751	0.840	0.718
Second Order Kinetics								
q_e (mg g ⁻¹)	9.65	2.73	9.9	1.67	7.64	1.93	8.34	0.57
k_2 (g mg ⁻¹ min ⁻¹)	0.05	0.06	0.04	0.02	0.05	0.13	0.02	0.79
h (g mg ⁻¹ min ⁻¹)	4.66	0.45	3.92	0.06	2.92	0.48	1.39	0.26
adj. R^2	0.983	0.820	0.99	0.848	0.988	0.860	0.936	0.779

efficiency would probably be achieved with acid activation (Table 5).

Adsorption of neonicotinoid pesticides including thiacloprid was studied by (Shi et al., 2022) with biochar produced from *Tenebrio molitor* frass and modified by KOH. The isotherm of thiacloprid fitted the Langmuir model and the adsorption was well fitted by the pseudofirst-order kinetic model. The maximum adsorption capacity for thiacloprid was much higher than our results (155.08 mg g⁻¹ vs. 4.15 mg g⁻¹ (AC-IG-NaOH) and 2.17 mg g⁻¹ (AC-RJ-NaOH) (Table 5)). The S_{BET} value of the best adsorbent was of 1858.80 m²g⁻¹, compared to our values up to 350 m²g⁻¹. This may be due to the different input biological material (plants vs. animals) and also the higher pyrolysis temperature (750 °C), the effect of which on the adsorption capacity the authors (Shi et al., 2022) discussed.

Adsorption of both thiacloprid and thiamethoxam using activated (ZnCl₂) biochar was studied by (Matos et al., 2017). The amounts of thiacloprid and thiamethoxam adsorbed per gram of activated biochar adsorbent were 1.02 and 0.97 mg, respectively. Compared to our results, these are lower values, since the maximum adsorption capacity calculated from the Langmuir model was 17.07 mg g⁻¹ for thiamethoxam and a sorbent prepared from *I. glandulifera*, and 18.30 mg g⁻¹ for thiacloprid and a sorbent prepared from *I. glandulifera*, both activated by H₃PO₄.

When comparing our results with the results from the literature, it is clear that it depends on the substrate from which the activated carbon is prepared, the operating conditions, and the activation agent. However, it is also necessary to consider the chemical nature of the substance that is removed from the

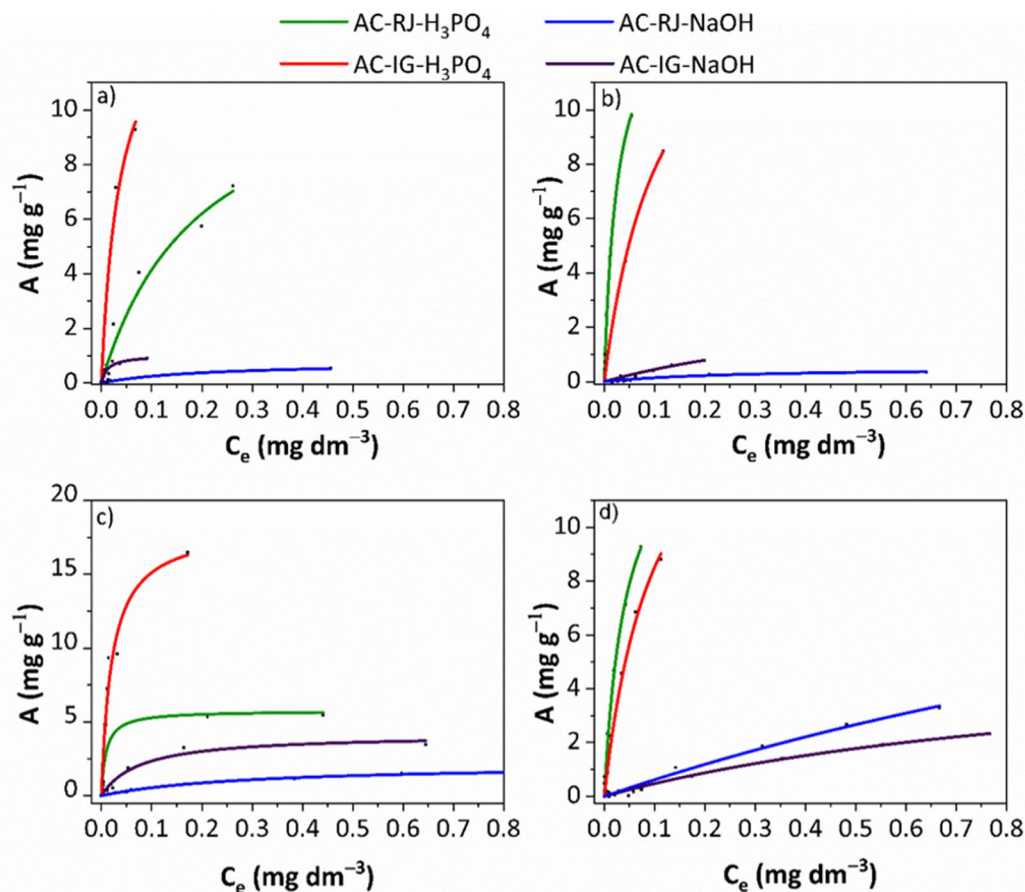


Fig. 12 Adsorption isotherms (Langmuir model) of (a) Acetamiprid (b) DEET (c) Thiachloprid (d) Thiamethoxam adsorbed on activated carbons prepared from *Reynoutria japonica* (RJ) and *Impatiens glandulifera* (IG) activated by H₃PO₄ or NaOH

water, since the adsorption capacity and thus the removal efficiency is not the same for all.

4 Conclusions

Four major pesticide pollutants have been identified in water bodies in Odra river basin—acetamiprid, diethyl-toluamide (DEET), thiachloprid and thiamethoxam. Their occurrence had a seasonal character with the maximum measured concentration 507.5 $\mu\text{g L}^{-1}$ for repellent DEET.

Carbon sorbents prepared from invasive plants were tested for the removal of pesticides from water. The type of activating agent had a significant influence on the textural development and chemical nature

of the surface of the sorbents. For the samples prepared by activation with H₃PO₄, the porosity had a significant effect on the sorption capacity, which was much higher than for the sorbents prepared with NaOH. In the case of similar porosity, the adsorption efficiency was determined by the presence of functional groups on the surface of the sorbent.

Better results were achieved in the case of sorbents from *I. glandulifera*, compared to *R. japonica*, which may be due to the different structure of the plant material. The exception was the adsorption of thiamethoxam, where a high sorption capacity was achieved even in the case of using a sorbent from *R. japonica* activated by NaOH. It can therefore be said that adsorption also depends on the chemical nature

Table 5 Parameters of isotherms models

	<i>Acetamiprid</i>				<i>DEET</i>			
	AC-IG- H ₃ PO ₄	AC-IG- NaOH	AC-RJ- H ₃ PO ₄	AC-RJ- NaOH	AC-IG- H ₃ PO ₄	AC-IG- NaOH	AC-RJ- H ₃ PO ₄	AC-RJ-NaOH
Freundlich								
k_f (mg g ⁻¹)	56.89	2.78	19.44	0.88	32.01	3.34	60.05	0.52
n_f	1.55	2.28	1.37	1.72	1.61	1.13	1.65	1.60
adj. R^2	0.952	0.941	0.935	0.996	0.985	0.988	0.951	0.829
Langmuir								
q_m (mg g ⁻¹)	14.81	1.02	12.33	0.77	16.37	3.24	14.44	0.52
k_L (dm ³ mg ⁻¹)	26.31	81.83	5.07	4.73	9.13	1.62	39.53	3.80
adj. R^2	0.988	0.977	0.951	0.995	0.976	0.990	0.982	0.885
	<i>Thiacloprid</i>				<i>Thiamethoxam</i>			
	AC-IG- H ₃ PO ₄	AC-IG- NaOH	AC-RJ- H ₃ PO ₄	AC-RJ- NaOH	AC-IG- H ₃ PO ₄	AC-IG- NaOH	AC-RJ- H ₃ PO ₄	AC-RJ- NaOH
Freundlich								
k_f (mg g ⁻¹)	38.18	4.68	7.66	1.87	46.34	2.92	55.52	5.02
n_f	2.27	2.44	3.49	1.80	1.37	1.31	1.50	1.07
adj. R^2	0.877	0.840	0.708	0.962	0.946	0.993	0.968	0.976
Langmuir								
q_m (mg g ⁻¹)	18.30	4.15	5.76	2.17	17.07	5.66	14.68	15.42
k_L (dm ³ mg ⁻¹)	47.28	13.02	109.44	3.32	9.94	0.92	23.25	0.42
adj. R^2	0.962	0.961	0.851	0.994	0.968	0.995	0.984	0.981

of the adsorbed substance when molecules with more polarized sites in their structure are better adsorbed.

Our results could be applied in technologies dealing with the extraction and removal of pesticides from wastewater, for which low-cost adsorbents can be produced from invasive plants growing near locations where water treatment takes place.

Authors Contribution Tereza Motúzová: Conceptualization, Investigation, Methodology, Formal analysis, Visualization, Writing – original draft. Ivan Koutník: Investigation, Methodology, Formal analysis, Data curation, Writing – review & editing. Martina Vráblová – Conceptualization, Supervision, Writing – review & editing.

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Data Availability Data will be made available on request.

Declarations

Competing of 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.

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