Article

Physicochemical Characteristics of Biochar and Their Influence on Sorption Mechanisms of Clothianidin Neonicotinoids in Agricultural Soils

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Abstract: This paper aims to two types of biochar were prepared from different sources: the pruning residues of *Pinus roxburghii* and *Cupressus sempervirens* trees at 700°C. Electron dispersive X-ray (EDX) analysis of both types of biochar revealed an abundance of carbon and oxygen with traces of other elements. The vessels of *C. sempervirens* biochar, as observed by scanning electron microscopy (SEM), are wider than those of *P. roxburghii* biochar, which may reflect the efficiency of *C. sempervirens* biochar compared to *P. roxburghii* biochar. Additionally, the fourier transform infrared (FTIR) spectra show that both types differ in their functional group content, which may reflect differences in their effects. These types of biochar were added at a rate of 5% to the soil to study their effect on the absorption capacity of clothianidin neonicotinoids by sandy clay loam soil, sandy loam soil and sandy soil. Adding biochar to tested soils enhanced the adsorption of clothianidin, especially when *C. sempervirens* biochar was added. All soil parameters agree with the Freundlich equation through high value of R^2, and low value of SE.

Keywords: Agricultural, Biochar, Clothianidin, Soil, Sorption

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Introduction

Pesticides are critical chemical compounds essential for crop growth; however, both enzymatic function and soil microbial biodiversity are damaged by pesticides, accompanied by decay of organic matter (OM) in soil. The accompanying environmental problems are mostly the result of pesticides' enduring usage and widespread notion that prejudices biodiversity. As a result, studies aimed at lowering pesticide risks in soil and water has received more attention [1-4].

At present, different techniques are frequently used for removing toxic compounds from soil. These techniques include chemical, and physical remediation (oxidation and reduction, washing, and sorption), phytoremediation and bioremediation. Each technique has advantages and limitations. Both phytoremediation and microbial remediation are practical and cost-effective techniques, but their application depends on availability and strength of insecticide residues and capacity of detoxifying agent to break down insecticide compounds [5, 6]. There is a benefit to the sophisticated oxidation process, but it requires a lot of upkeep and capital [7]. Physical remediation techniques are nevertheless successful, but because of the lack of environmental safety, their use is discouraged. Therefore, it is necessary to create safe, very stable, cost-effective, and environmentally friendly ways for cleaning up pesticide residue from the environment. Even while every remediation technique has some positive effects on soil remediation, it's crucial to note any negative effects as well. The type and concentration of the pesticide, the soil's physico-chemical characteristics (such as OM), the surrounding environment, or the application cost should all be taken into consideration when selecting the best remediation technique [8, 9].

Even while soil OM makes up a small portion of the total dried material in the soil, it is one of the main adsorbents for pesticides [10, 11]. Due to pesticides' increased chemical susceptibility to organic molecules, a variety of interactions between pesticides and OM are permitted, which leads to pesticide adsorption on soil OM [11, 12]. Soil OM is structurally quite diverse [11]. The adsorption capacity of soil is significantly influenced by the chemical composition of OM [11]. It comprises of substances which are non-humic and humic in nature. When it comes to pesticides, humified matter is chemically more reactive than non-humified matter [11, 13]. Various reactive functional groups like amines, amide, alkoxy, carboxylic acids, carbonyl, esters hydroxyl, and phenols are found in humic acids. In addition to humic acids, soil contains water-loving and water-repelling molecules that play a crucial role in how organic pesticides interact with soil organic matter [11, 14].

One of the promising adsorbent materials is biochar, which has exceptional adsorption properties and is highly stable; additionally, biochar is eco-friendly and cost-effective [15, 16]. Biochar contains many adsorption sites due to its high specific surface area and porosity. Biochar improves soil microbial activity, reduces the chance of soil contamination, and aids in the adsorption and degradation of pesticides and other persistent organic pollutants [17-19]. The production process of biochar is logical for feedstocks containing agricultural waste. The comprehensive use of these wastes is consistent with sustainable development concepts [20]. Functional groups distributed on surface are significant in biochar applications as catalysts, electrode materials, and adsorbents [21]. Therefore, biochar is has a large surface area and rich in elements, porous structure, ion exchange, and electrostatic attraction, which allow biochar to be applied directly as a modifier, adsorbent, catalyst, and catalyst carrier. Furthermore, biochar is a multifunctional material with significant application potential and research value due to its capacity to load different metal ions and graft functional groups [22].

The benefits of using biochar to ascertain the destiny and behavior of pesticides have grown in importance recently. The interactions of pesticide with biochar into soil occur through different processes, such as degradation, leaching, and adsorption/desorption to limit their mobility and availability [23]. Because of its great ability to bind pesticides, biochar can lower the concentration of pesticides in soil. It can also alter the fate and behavior of pesticides that may compete with other pollutants [24, 25]. Multiple mechanisms are involved in pesticide adsorption on biochar. Chemical adsorption starts through interactions between charged pesticide molecules and charged functional groups of biochar surface through electrostatic attraction [26]. Furthermore, the porous structure and large surface area of biochar affect the process by which pesticide molecules can be trapped inside pores through size exclusion through physical adsorption [27, 28]. Information about the effects of biochar and how it interacts with different pesticides in soil is usually available. Because of its superior absorption capacity, biochar, for example, lowers the danger of water pollution and ensures the safety and purity of agricultural products by reducing pesticide leaching from the soil and absorption in the plants [27, 29, 30].

One of the largest groups of insecticides increasingly used in the world is neonicotinoids [31-33]. Recently, neonicotinoids have attracted increasing interest due to their harmful effects on different nontarget organisms [1, 34]. Neonicotinoids are highly soluble in water, which makes them highly soluble in groundwater or surface runoff. This poses serious concerns about the possibility of neonicotinoids being applied widely on agricultural soils [5]. One of the neonicotinoid insecticides that was introduced recently to our market is clothianidin, which has different characteristics, such as mobility into environment and its persistence, stability toward hydrolysis to groundwater and transport through runoff to surface water. The major factor that affects the leaching pattern of clothianidin is solubility [7].

Here, we report a study that examines the effects of biochar application on the adsorption behavior of neonicotinoid clothia-

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nidin on soils. Two contrasting biochars, *Pinus roxburghii* and *Cupressus sempervirens*, at 5% (w/w) were mixed with three contrasting soils (thus yielding 9 soil–biochar treatments). Using these soil–biochar mixtures and biochar-free soils, we studied the adsorption of clothianidin in sandy clay loam soil, sandy loam soil and sandy soil. The objectives were to determine effect of soil type and biochar type on the adsorption of clothianidin into tested soils.

Materials and Methods

Clothianidin

IUPAC name: 1-(2-chloro-1,3-thiazol-5-ylmethyl)-3-methyl-2-nitroguanidine. The technical grade (99.9%); was obtained from Egyptchem Pesticides and Chemicals Co. (Second Industrial Zone, New Nubaria City, Beheira Governorate, Egypt). The chemical formula of clothianidin is $C_6H_8ClN_5O_2S$, and its structure is presented in Fig. 1.

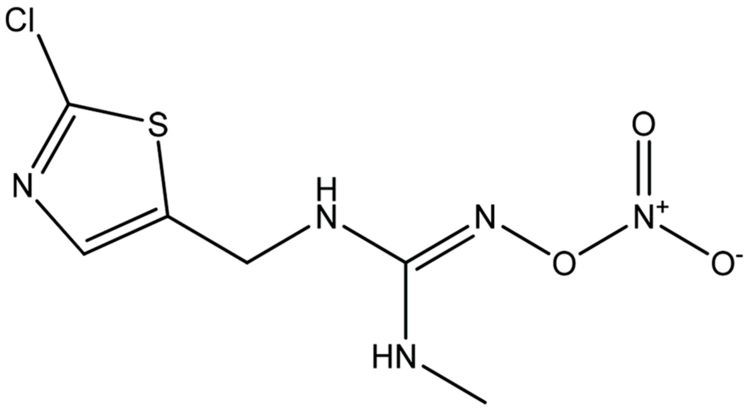

Fig. 1. Chemical structure of tested clothianidin.

Soil preparation

Three types of the soils were tested in present study: alluvial soil (sandy clay loam soil), calcareous soil (sandy loam soil), and sandy soil (sandy soil) from the Alexandria region in Egypt. Samples of the plow layer soil were gathered from several sites. Following air drying, stubble, stone removal, and weed removal, the soil samples were crushed, powdered, and run through a 10-mesh screen. The soil's texture was ascertained using the hydrometer method. The pH of the soil was measured using a 1:2 w/w soil: solution slurry that contained 0.01 M calcium chloride ($CaCl_2$). Dichromate oxidation was utilized to ascertain the OM-content [28, 29]. Table 1 lists the fundamental physicochemical characteristics of soils that were investigated.

Table 1. Physicochemical characteristics of soil

Particle Size (%)			Texture class	pH	EC (ds/m)	Total soluble cations (meq/L)	Total soluble anions (meq/L)	Total carbonate (%)
Clay	Silt	Sand						
41	18	41	Sandy clay loam	8.25	1.32	18.17	13.30	7.87
13	12	75	Sandy loam	8.19	2.32	33.49	23.29	40.10
10	3	87	Sandy	8.51	1.18	17.42	15.51	4.01

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Biochar preparation

P. roxburghii and *C. sempervirens* pruning residue from the forestry research division of the Antoniades Botanical Garden in Alexandria, Egypt, was used to make biochar. The pruning branches were allowed to air dry for approximately three months before being stored at room temperature in the laboratory. The branches were debarked and then sawed into the proper parts. Wood samples were placed in crucibles, covered tightly with a lid, and pyrolyzed in a muffle furnace with minimal oxygen. The pyrolysis temperature was maintained for 60 minutes after reaching 700°C at a rate of roughly 15°C per minute. The biochar was then ground, left to cool to room temperature, and stored in airtight bags until it was needed [30].

Biochar characterization

A scanning electron microscope (JEOL JSM-5300 SEM) was used to evaluate the surface microstructure of the biochar, and elemental composition was examined using SEM combined with electron dispersive X-ray analysis (EDX) at an acceleration voltage of 15-20 KeV [31].

Determination of clothianidin content

Stock solution (1000 μg/mL) was dissolved in acetonitrile (HPLC grade) and then progressively diluted to create standard solutions of clothianidin (0.05, 0.1, 0.5, 1, 5, and 10 μg/mL). The ideal wavelength (λ_{max} = 298 nm) was found using a UV-Vis spectrophotometer with a scanning range of 200–400 nm. Plotting the amounts against their corresponding absorbance at optimal λ_{max} produced the standard calibration curve, as presented in Fig. 2 [32].

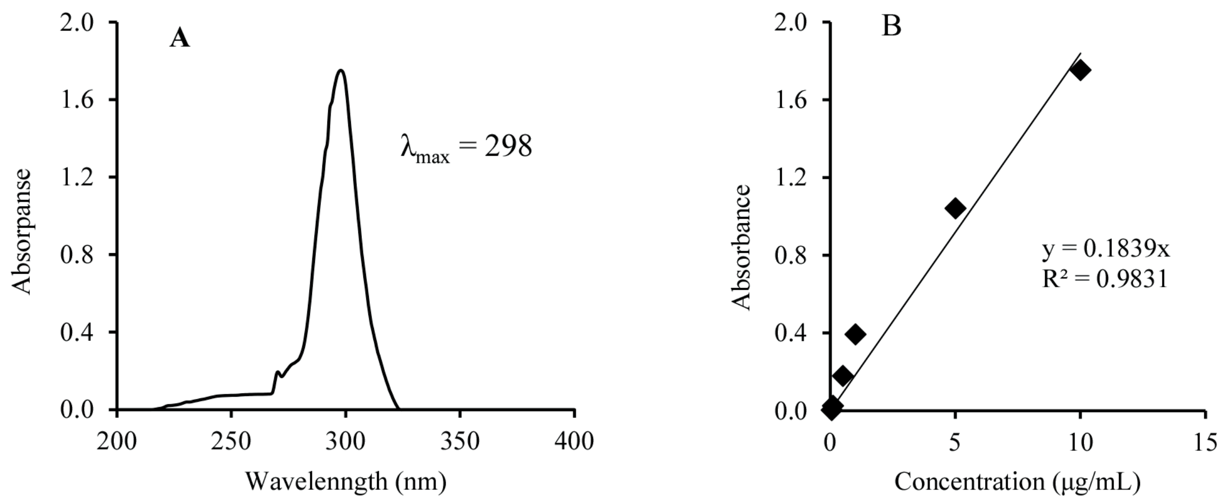

Fig. 2. Spectral-density curve (A) and calibration density curve (B) of clothianidin by UV-spectrophotometry.

Adsorption kinetics test

The equilibration time for clothianidin's adsorption to the studied soils was ascertained by a kinetic investigation. The experiment on batch adsorption kinetics was carried out twice. Known weight of soil (1 g) was placed in a flask containing 5 ml of $CaCl_2$ solution (0.01M) in which the pesticide was dissolved at a concentration of 5 μg/mL. The soil and the pesticide solution were placed in 25 mL polypropylene centrifuge tubes, which were physically shaken at 200 rpm at room temperature in the dark. The tubes were centrifuged at 3000 rpm for 10 minutes after intervals of 1, 5, 15, 20, 25, and 30 hours, and the supernatant was examined for the tested pesticide.

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Adsorption isotherm test

The air-dried soil samples were run through a sieve with 2 mm holes for the adsorption experiments. Triplicates of 1 g of dried soil, 1 g of dried soil-5% *P. roxburghii* biochar, and 1 g of dried soil-5% *C. sempervirens* biochar were added to 50 mL centrifuge tubes and mixed with 5 mL of 0.1 M $CaCl_2$ solution containing 0.05, 0.1, 0.5, 1, 5, and 10 μg/mL clothianidin. The slurries were shaken for twenty-four hours at a vibration rate of 200 rpm at room temperature (23±2°C) [33]. Following agitation, samples were centrifuged for 10 minutes at 3000 rpm. A spectrophotometer was used to measure the amount of insecticide in the supernatants at the appropriate λmax. Included were control samples (devoid of clothianidin) that contained just adsorbent materials and 0.1 M $CaCl_2$. The blanks containing pesticide solution without adsorbents demonstrated minimal pesticide degradation during the experiment and modest sorption on the tube [34, 35].

Freundlich model

It can be expressed as $q_e = K_F C^{1/n}$, where *n* is correction factor and K_F is distribution coefficient. By plotting linear form of equation: $\log q_e = \frac{1}{n}\log C + \log K_F$, the intercept is equal to log K_F, and the slope is value of 1/*n*.

Results and Discussion

Electron dispersive X-ray (EDX)

As stated by Budai et al., the elemental compositions of the biochar series were previously ascertained [36]. Both forms of biochar's elemental analyses showed that carbon was the primary element, followed by oxygen, with traces of other elements, including potassium and copper, found in *P. roxburghii*. The biochar of *C. sempervirens* contained traces of potassium and calcium, as shown in Table 2 and Fig. 3. EDX analysis revealed that carbon content of biochar prepared from *C. sempervirens* was greater than that of the biochar prepared from *P. roxburghii*. Feedstock is a key factor that affects carbon content of biochar. In general, carbon-rich biochar is more effective as a pesticide adsorbent owing to its high content of carbonaceous surfaces [37]. A high content of lignocellulose in feed stocks may increase the porosity of biochar, consequently enhancing its ability to adsorb pesticides [38, 39]. Furthermore, the remediation of organic contaminants with hydrogen and oxygen functional groups is favored by the high carbonized matter concentration of biochar [40].

Scanning electron microscopy (SEM)

SEM is useful for identifying biochar macropores and is mostly used to characterize biochar [41]. SEM pictures can provide information on surface shape, which is important for adsorbent/adsorbate interactions. These results imply that a considerable amount of the original wood cell shape is retained by porous surfaces with a well-organized structural structure (Fig. 4). According to SEM, wood porosity and residual wood cell morphologies are significant components of the final biochar preparation [42].

Table 2. EDX elemental analysis of biochar

Element	*Pinus roxburghii*	*Cupressus sempervirens*
	Mass % ±SD	Mass % ±SD
C	84.57±0.26	89.85±0.25
O	12.36±0.37	8.84±0.30
K	2.49±0.08	0.19±0.03
Cu	0.59±0.08	------------
Ca	-----------	1.12±0.05

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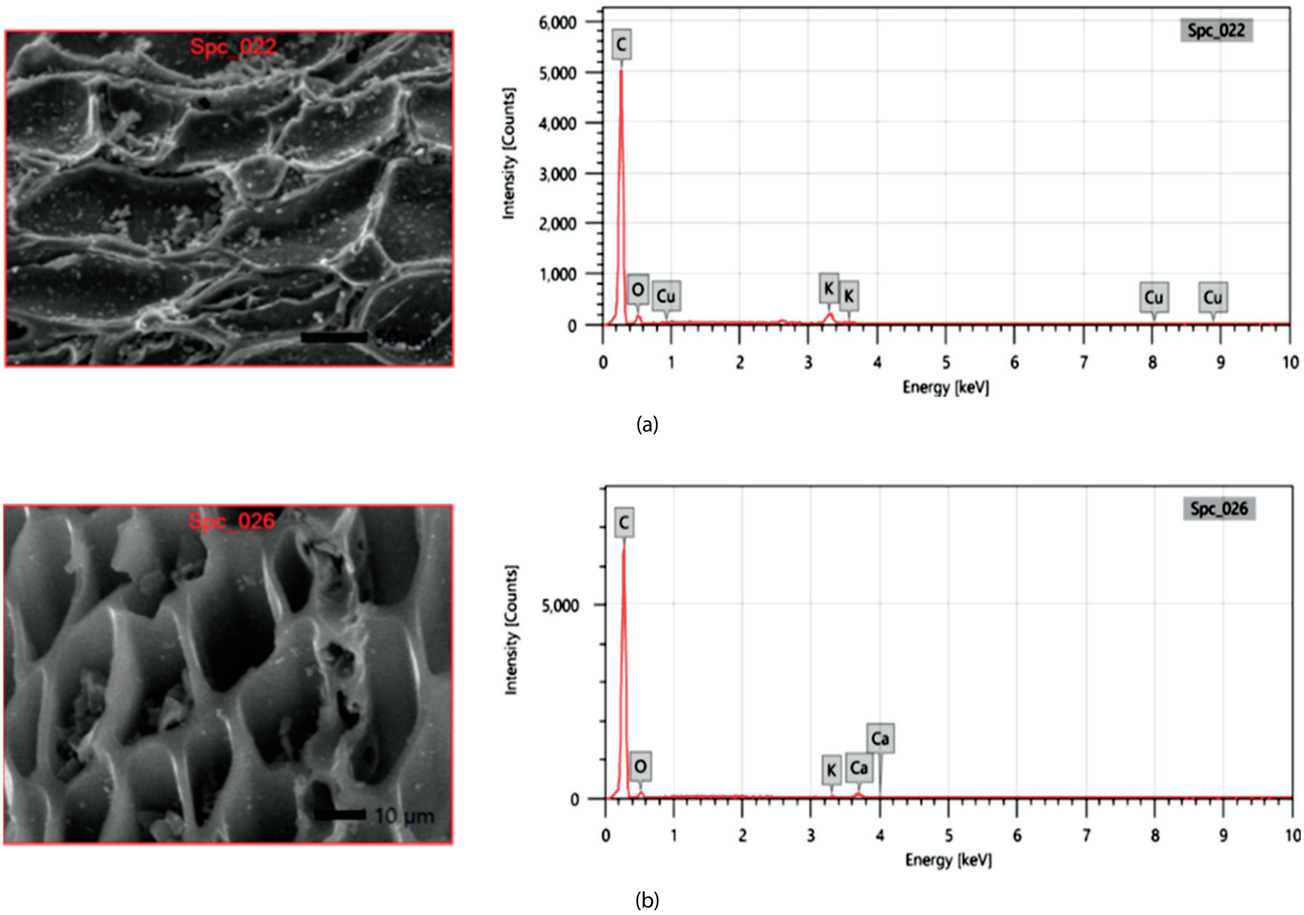

Fig. 3. Electron dispersive X-ray (EDX) image of biochar prepared from *Pinus roxburghii* (a) and *Cupressus sempervirens* (b).

Fourier transform infrared (FT-IR) analysis

The FTIR spectra show the relative differences between the two types in their functional groups, which may reflect the differences in their effects (Table 3 and Fig. 5). FTIR-analysis revealed presence of O-H stretching vibrations in *C. sempervirens* biochar at 3616 cm^{-1}, which are connected to carboxylic and phenolic compounds' hydrogen-bonded O-H groups [43-45]. Both types of biochar contained methyl groups substituted on aromatic rings, which were observed at 2984.4865 cm^{-1} - 2875.2252 cm^{-1} and 2969.4828 cm^{-1} - 2875.2491 cm^{-1} for *P. roxburghii* and *C. sempervirens*, respectively [45]. The bands observed for both biochar types in range of 2000–1500 cm^{-1} may refer to presence of functional groups with double bonds such as C=C and C=O [46-48], which were recorded at 1900, 1707 and 1548 cm^{-1} for the *P. roxburghii* biochar but recorded at 1923 and 1519 cm^{-1} for the *C. sempervirens*, which also recorded presence of C-H bending in-plane at 1360 cm^{-1} that may have resulted from cellulose or hemicellulose [49]. The bands found for both kinds in the 1000–1240 cm^{-1} range are linked to phenolic, alcoholic, and carboxylic acid C–O stretching [50, 51], as observed at 1136 and 1083 cm^{-1} for the *P. roxburghii* and *C. sempervirens* biochars, respectively. Both types are common in the presence of aromatic rings with more substituents, which was confirmed by band at 877 cm^{-1}, this was attributed to the C-H group's out-of-plane binding [52, 53]. Our results showed that biochar prepared from *C. sempervirens* is more efficient than that prepared from *P. roxburghii* and thus may result from presence of more functional groups, which increase chemical adsorption efficiency. The capacity of biochar to absorb insecticides chemically. Oxygenated functional groups such as carbonyl, carboxyl and hydroxyl groups play a significant role in improving performance of biochar in various applications, including its adsorption capacity for organic pollutants, and interactions occur between these groups through hydrogen bonds and electrostatic attraction [54, 55]. Additionally, ability of biochar to adsorb pesticides is affected

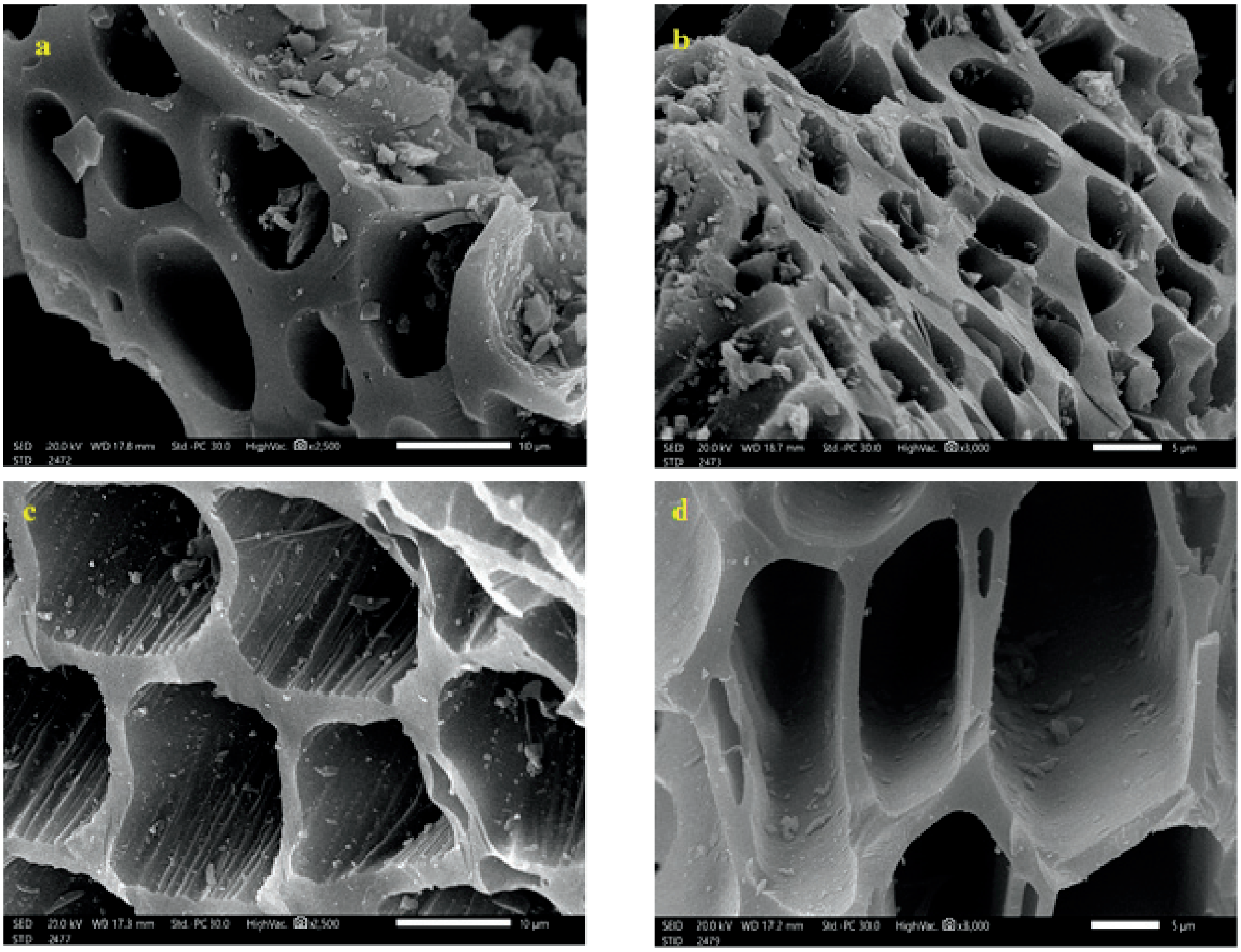

Fig. 4. The microstructure of biochar surface, showing the vessels of both types of biochar, *P. roxburghii* (a & b) and *C. sempervirens* biochar (c & d), at different magnifications. SEM clearly revealed wider vessels in the *C. sempervirens* biochar group than in the *P. roxburghii* biochar group at the same magnification.

by surface charge, and the chemical adsorption of organic molecules involves hydrogen bonding, π–π interactions and other interactions between molecules and biochar. Because of its high electronegativity, the oxygen atom in pesticides' oxygen functional groups can create hydrogen bonds with the functional groups on biochar. Formation of hydrogen bonds is a significant interaction mechanism that enhances stability of pesticides adsorbed on biochar surfaces. This interaction also affects their configuration and charge distribution. As illustrated in Fig. 1, which shows the chemical structure of clothianidin, it has different

Table 3. FTIR bands and corresponding functional groups were observed in spectra of both biochar types

	Assignments Wavenumber (cm^{-1})	*P. roxburghii*	*C. sempervirens*
3000-3700	-OH group (alcohols-phenols)	--------	3616.3444
2800-3000	C-H (methyl)	2984.4865-2875.2252	2969.4828-2875.2491
1900-2000	C=C=C (allene groups)	1900.984	1923.4181
1600-1800	C=O/COOH (carboxylic acids)	1707.1233	--------
~1500	C=C	1548.0609	1519.8156
1317-1375	C-H bending (symmetric and asymmetric or C-O asymmetric of aromatic	----------	1360.4247
1000-1260	C-O	1136.5270	1083.5114
700-900	C-H aromatic (out of plane)	877.0209	877.1605

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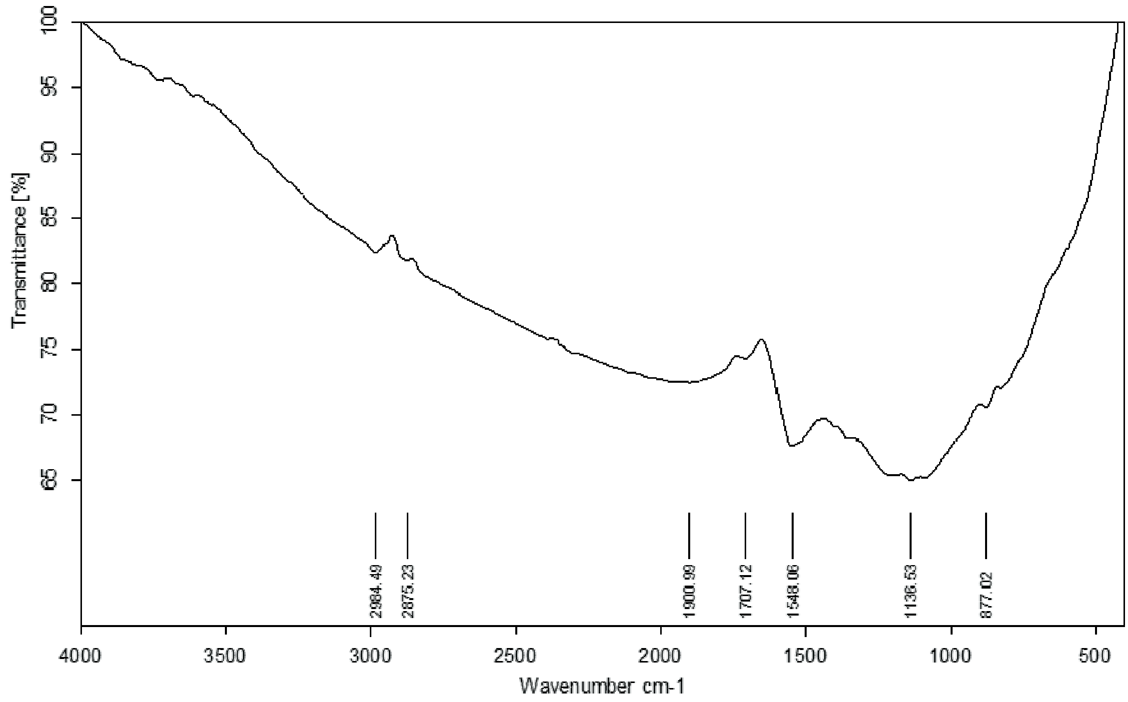

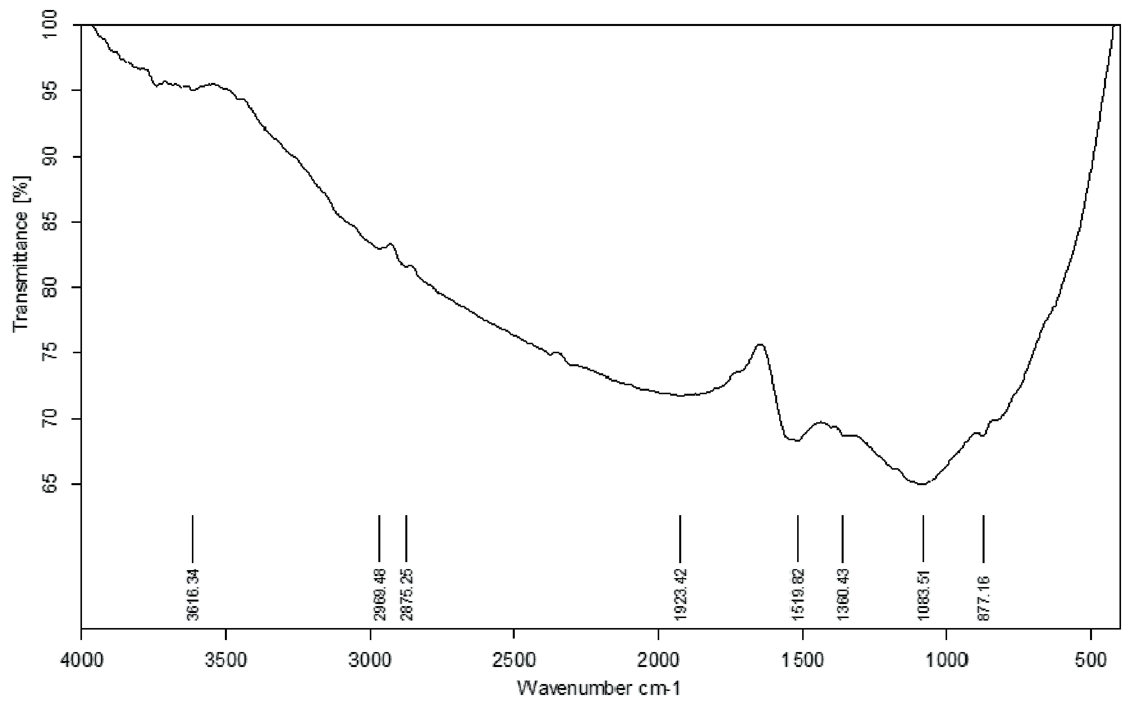

Fig. 5. FTIR spectra for *P. roxburghii* biochar (upper) and *C. sempervirens* biochar (lower).

sites that act as electron donors (electron rich), such as oxygen, sulfur, halide and nitrogen, which support donor-acceptor π–π electron interactions. The π-electron systems are found in pesticides with aromatic rings. This includes delocalized electrons, creating sites in both electron-rich and electron-lacking regions. Clouds of π-electrons interact through π–π stacking when aromatic rings on surface of biochar approach aromatic structure of pesticide molecules. This interaction contributes to stabilizing the adsorbed pesticide on biochar surface, enhancing affinity between the adsorbed pesticide and biochar [22, 23].

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Equilibrium time

A batch adsorption kinetics experiments were conducted to determine equilibration time of clothianidin onto tested soils. Adsorption of clothianidin on the three studied soils versus time at 5 μg/g soil as an initial concentration is illustrated in Fig. 6. The adsorption rate of clothianidin onto tested soils was increased during the first 15 hours, then the adsorption rate was stable from 15 hours to 30 hours. Thus, in the following adsorption experiments in this work, a 24-hour equilibrium time was employed.

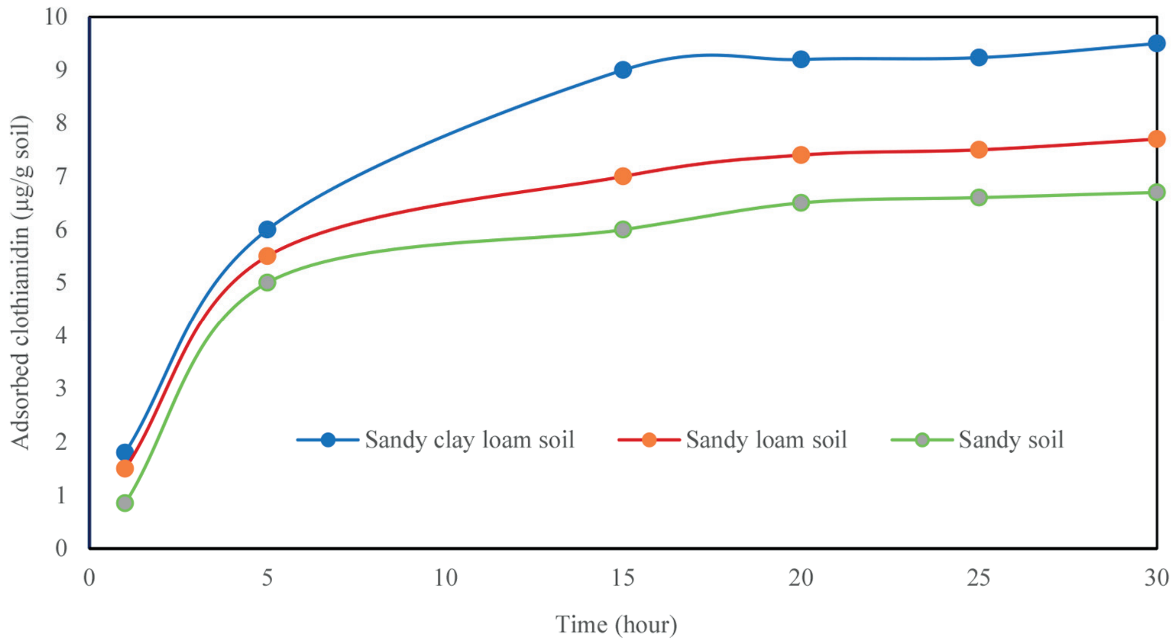

Fig. 6. Equilibrium time of clothianidin on tested soils.

Effects of biochar on adsorption isotherm capacity

In addition to improving clothianidin adsorption, adding biochar to soil altered the soil's sportive properties [56]. Adsorption isotherms of clothianidin on tested soils with and without biochar are shown in Fig. 7. Adding 5% biochar to the types of soil used in the study led to an increase in adsorption, especially the addition of *C. sempervirens* biochar. Both the two forms of biochar and the various soils differed significantly. The way clothianidin adsorbs on biochar's revealed that the materials' ability to adsorb pesticides varied widely [57]. Adsorption of clothianidin was generally concentration dependent, and as the concentration of clothianidin in solution increased, the adsorption reduced. All soil samples sorption data fit the Freundlich sorption model well. The derived model parameters ($1/n$, Log K_F, and SE) are given in Table 4. In general, the slopes ($1/n$) were less than 1.33, suggesting an L-type adsorption isotherm [57, 58], it shown that at low pesticide concentrations, there was a substantial contact between the adsorbent and the adsorbate during the sorption process, and that adsorption reduced as clothianidin's aqueous phase concentration grew. This was explained by fact that there was more competition for adsorption sites, which were immitted as concentration of the solute in solution rose [57]. As expected, given the extremely low OM concentration, the lowest adsorption (the lowest K_F value) was recorded into bulk soils (without biochar). The highest adsorption was clearly detected in sandy clay loam soil (the soil with largest OM-content) [58-64].

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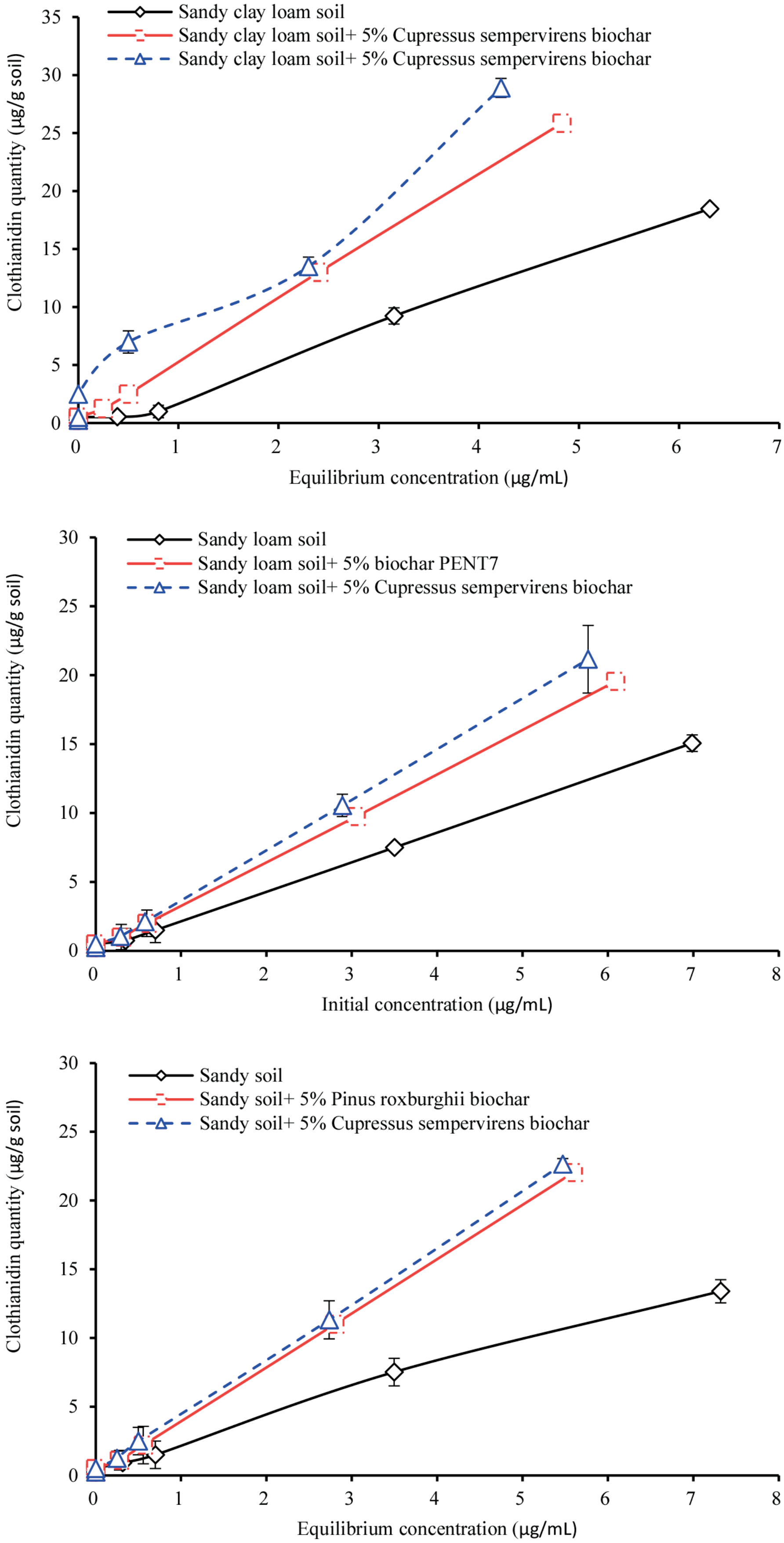

Fig. 7. Adsorption isotherms of clothianidin on the bulk soils and modified soils containing 5% biochar.

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Table 4. Freundlich adsorption isotherm parameters of clothianidin on bulk soils and modified soils with 5% biochar

Freundlich parameters	1/*n*	Log K_F	R^2	SE
Sandy clay loam soil	1.33	0.23	0.99	0.24
Sandy clay loam soil + 5% *P. roxburghii* biochar	1.03	0.71	0.99	0.18
Sandy clay loam soil + 5% *C. sempervirens* biochar	1.14	0.74	0.99	0.43
Sandy loam soil	1.00	0.33	1.00	0.36
Sandy loam soil + 5% *P. roxburghii* biochar	0.98	0.52	1.00	0.34
Sandy loam soil + 5% *C. sempervirens* biochar	1.00	0.56	1.00	0.65
Sandy soil	0.88	0.37	0.99	0.41
Sandy soil + 5% *P. roxburghii* biochar	0.93	0.60	1.00	0.39
Sandy soil + 5% *C. sempervirens* biochar	0.93	0.66	1.00	0.43

Conclusions

Elemental analysis of the biochar revealed that carbon was major element, followed by oxygen. Porous surfaces of the biochar exhibited an organized structural shape. FTIR-analysis revealed presence of methyl groups on aromatic rings, functional groups with double bonds, C-H bending, and C-O groups into both types of biochar. Adding 5% biochar to tested soils increased amount of clothianidin absorbed and thus reduced the amount leached into the groundwater. The adsorption data of all the soil samples were fit with the Freundlich model.

Data Availability: All data are available in the main text or in the Supplementary Information.

Author Contributions: Mohamed R. Fouad: Methodology, resources, data curation, writing, review and editing. Hesham M. Aly and Noura A. Hassan: Preparation and analysis of biochar.

Notes: The authors declare no conflict of interest.

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Additional Information:

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