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Occurrence, persistence and risk assessment of pesticide residues in European wheat fields: A continental scale approach

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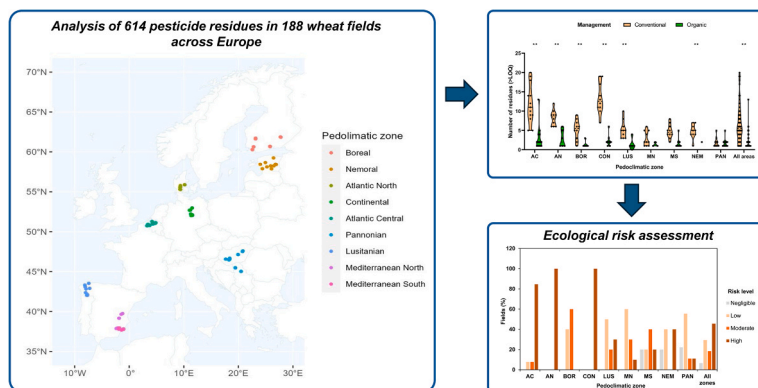
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HIGHLIGHTS

- More than 600 compounds were analyzed in 188 European soils from wheat fields.
- Two or more pesticides were detected in 88 % and 63 % of conventional and organic fields.
- Fenbutatin oxide and AMPA were the most frequently detected pesticide residues.
- Epoxiconazole and boscalid showed the highest level of ecological risk.
- 46 % of conventional fields showed a high level of ecological risk.

GRAPHICAL ABSTRACT



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ABSTRACT

Pesticide residues in agricultural soils represent an environmental concern that requires special attention due to their potential ecological and public health risks. We analyzed 614 pesticides in 188 wheat fields across Europe subjected to both conventional and organic farming systems. At least one pesticide residue was detected in 141 soils. Seventy-eight pesticides or their metabolites were detected. The presence of pesticides was significantly higher in both number and concentration in conventional fields (up to 0.98 mg kg^{-1}) compared to organically managed sites (up to 0.40 mg kg^{-1}). A total of 88 % of conventional fields and 63 % of organic fields contained two or more pesticides. Conversion from conventional to organic farming does not guarantee that soils will be pesticide-free in the short term. Fenbutatin oxide was the most frequently detected pesticide in both farming systems, followed by AMPA. Other substances, such as boscalid, epoxiconazole, diflufenican, tebuconazole, dinoterb, bixafen, and DEET, were found in ≥ 10 % of samples. Some Persistent Organic Pollutants, including dieldrin, endosulfan sulphate, and chlorpyrifos, were also detected. Ecological risks were higher in conventionally managed fields, with 46 % exhibiting high-risk levels, compared to just 1 % in organic fields. Epoxiconazole and boscalid were the substances with the highest risk levels.

1. Introduction

The intensification of agriculture has led to an increased need for soil fertilization, irrigation, and the use of pesticides, among others. The use of pesticides has provided advantages in reducing weeds, diseases, and pests. In fact, it is estimated that pesticides reduce crop losses due to pests and diseases by around 30 % [1]. In general, active ingredients and their transformation products (metabolites) are a wide range of chemical compounds, with differences in their molecular properties or mechanisms of action that can act on a target organism or a broad range of organisms [2–4].

Due to the essential role that pesticides play in improving agricultural production, their usage has increased significantly in recent years. In this context, global pesticide consumption in agriculture rose from 2,846,866 tonnes in 2010 to 3,526,746 tonnes in 2022, representing a 24 % increase. Among the different pesticide categories, herbicides are the most widely used worldwide, accounting for 55 % of total agricultural pesticide consumption, followed by fungicides and bactericides (23 %), and insecticides (22 %) [5].

At the European level, pesticide consumption in agriculture has also risen substantially in recent years, although to a lesser extent than globally. In this regard, over the past decade, pesticide consumption in Europe increased by approximately 12 %, from 402,229 tonnes in 2010–449,038 tonnes in 2022, representing 13 % of global pesticide consumption [5]. This more moderate increase in Europe compared to the global trend is mainly due to stricter regulations and limitations on pesticide use compared to other regions of the world [6]. In this sense, in 2023, 444 pesticides were authorized for use in pest management in

Europe, while 954 were not approved or were banned, and a further 43 were still pending approval [7,8]. Unlike the global trend, in Europe, fungicides and bactericides, as well as herbicides, are the most widely used pesticide groups, each representing 42 % of the total European pesticide consumption, while insecticides account for only 16 % of the total [5].

Although pesticides have undoubtedly contributed to increasing global food production to meet the demand of a continuously growing world population, their misuse and overuse during the last decades have raised concerns about their adverse effects on the environment and public health. In this regard, it has been estimated that less than 5–15 % of pesticides applied to crops actually reach the target pest, while the rest enter the environment freely, leading to soil, water, and air contamination, thus generating a loss of biodiversity, ecotoxicity in non-target organisms, pest resistance and, finally, a loss of food security [9–13]. In addition, and perhaps even more concerning, pesticide residues can enter the food chain through crops and drinking water, potentially leading to adverse impacts on human health, including neurodegenerative issues, cardiovascular, endocrine, respiratory, and renal imbalances, skeletal problems, reproductive system dysregulation, and even cancer [13,14].

Wheat is one of the most important cereal crops in the world, being used as animal feed and as the main source of food for nearly 40 % of the world's population. In Europe, more than 50 % of cereal crops are represented by wheat. In addition, Europe is the region with the highest wheat yield in the world (an average of 4.2 t ha^{-1}), both due to moderately favorable natural conditions and intensive and innovative production systems [15,16]. Despite these conditions, pesticides play a

key role in increasing the quantity and quality of wheat production, including weed, pest, and disease control, and even for wheat grain storage [16,17].

Different guidelines have been developed in recent years to ban some dangerous pesticides, reduce their use, and take measures to increase the protection of human health and the environment [4]. In order to achieve more sustainable agriculture, different agricultural practices, such as organic farming, crop rotation, or ploughing, are suggested as alternatives to reduce the use of chemical fertilizers and synthetic pesticides [18]. While organic agriculture can use only some allowed active substances and compounds (around 28), conventional agriculture has around 444 active substances allowed [8,19,20]. However, due to the high persistence of these substances, organically managed fields can contain residues of different pesticides due to past conventional practices, even up to 20 years later [21], or due to pesticide drift from nearby fields [2,22,23].

The present study aimed to provide information on the occurrence and concentration of pesticide residues in wheat fields across different European pedoclimatic zones, as well as to assess the ecological risks associated with the presence of such pesticide residues. In general, previous similar studies with different agricultural management practices were carried out considering different crops (i.e., [2,24]), or under a regional or national scale (i.e., [23,25,26]), while studies with the same crop usually cover a low number of study areas and pesticide residues analyzed, or similar environmental conditions are studied (i.e., [25]). For these reasons, the occurrence of the residues of 614 pesticides (223 insecticides, 136 fungicides, 169 herbicides, 71 metabolites or degradation compounds, eight plant growth regulators, two nematocides, one rodenticide and one bird repellent, and three unclassified substances) was investigated in the topsoil of 188 agricultural fields across Europe. The samples originated from wheat fields under conventional or organic farming systems in nine European pedoclimatic zones and with different agricultural practices on crop rotation, tillage, and fertilization. Our hypothesis is related to the fact that under different agricultural production systems, such as conventional and organic farming, the different geographical locations are expected to present different pesticide patterns. In addition, we also hypothesize that the occurrence of pesticide residues according to their type and quantities is linked to specific soil characteristics.

2. Materials and methods

2.1. Soil sampling

As part of the European project SoilDiverAgro (<https://soildiveragro.eu/es/>), soil samples were collected at nine European pedoclimatic zones and from different countries, covering 86 % of the EU-27 territory: Boreal (BOR) (Finland), Continental (CON) (Germany), Atlantic Central (AC) (Belgium), Lusitanian (LUS) (NW Spain), Nemoral (NEM) (Estonia), Atlantic North (AN) (Denmark), Mediterranean North and Mediterranean South (MN and MS) (South and Eastern Spain), and Pannonian (PAN) (Hungary and Serbia) (Fig. S1, Supplementary material). In each pedoclimatic zone, an average of 20 wheat plots were selected for sampling (10 from conventional farming and 10 from organic farming). In addition, pairs of conventional and organic plots with similar soil characteristics in the same area were selected to ensure consistency in pedoclimatic conditions. The exact number of plots per farming system and pedoclimatic zone is shown in Table S1 (Supplementary material) (93 and 95 plots for conventional and organic systems, respectively). Soil samples were collected 15–30 days after the wheat harvesting (between July and October 2019). Fields with organic production fulfilled the EU criteria for this type of production [27] (i.e., synthetic fertilizers and pesticides are prohibited in organic farming). In the organic sampling areas, 88 % of the studied fields had been under organic farming for at least 5 years [28]. Although four samples (two from conventional and two from organic farming systems) came from a

non-EU country (Republic of Serbia), guidelines for organic farming systems and pesticide guidelines have been transferred from the European Union to national legislation as an EU-candidate country [29].

Detailed information for each plot with precipitation and temperature data from the previous 12 months and 30 years before collection, agricultural farming system [conventional (Conv) or organic (Org)], number of years of the mentioned system (from 1 to 60 years), crop rotation, tillage system, fertilization practice, and pesticide application, can be found in the Supplementary data (Table S2), in the raw dataset [28,30], and in Peltoniemi et al. [31]. A schematic representation of the sampling procedure was previously provided by Waeyenberge [32]. Briefly, a composite sample (minimum 2 kg) was collected from each plot at a depth of 0–25 cm at approximately 60 random representative points, following a zigzag pattern to cover the entire 1 ha field study site area. A steel auger with a variable diameter (ranging from 13 to 50 mm) was used. During the sampling process, irrelevant materials, such as stones and roots, were removed. Once in the laboratory, soil samples were air-dried, homogenized, sieved through a 2-mm mesh, and stored in the dark until analysis.

2.2. Selection of the pesticide residues

In all studied samples, an initial list of 614 pesticides (223 insecticides, 136 fungicides, 169 herbicides, eight plant growth regulators, two nematocides, one rodenticide, one bird repellent, 71 degradation compounds, and three unclassified substances) was analyzed based on the active substances most often applied to crops, according to the EU Pesticides Database (v3.2) [8] and reported by other authors (i.e., [2,24,26]). Due to chemical characteristics and extraction differences, a differentiation was carried out between multi-pesticide residues and glyphosate compounds. The characteristics of the analyzed pesticide residues are indicated in Table S3 and Fernández-Calviño et al. [28].

2.3. Determination of multi-pesticide residues

The QuEChERS method (Quick, Easy, Cheap, Effective, Rugged, and Safe) [33] for the determination of pesticide residues on foods of plant origin and adapted to measure soil samples [2,34] was used to measure the multi-pesticide residues. This method is based on pesticide extraction with one or several solvents, the cleaning of the sample to avoid interferences, and the determination of pesticides by UPLC-MS/MS (Agilent 1290 Infinity UPLC/AB Sciex 5500 Qtrap MS) and GC-MS/MS (Agilent 7000 Triple Quad GC/MS). Briefly, a 10 g soil aliquot was spiked with 10 mL Millipore water and 10 mL acetonitrile within a 50 mL Greiner tube. This mixture was agitated for 10 min, after which QuEChERS salts were added to the tube (4 g of MgSO_4 , 1 g of NaCl, 1 g of $\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2 \text{H}_2\text{O}$, and 0.5 g of $\text{Na}_2\text{C}_6\text{H}_6\text{O}_7$). Then, the tube was vortexed (10 min) and centrifugated (10 min, 3500 rpm), and the supernatant was transferred to Eppendorf tubes for UPLC-MS/MS and GC-MS/MS analyses. For the GC-MS/MS, 1000 μL of the supernatant were evaporated and recomposed with 1000 μL of internal standard. For the UPLC-MS/MS analysis, 100 μL of the supernatant and 900 μL of formic water 0.01 %: MeOH (80:20; v/v) were added directly into an LC vial to be analyzed. Analytical measurements were conducted at Laboratorio Analítico Bioclinico SL (Almería, Spain).

More information about the UPLC-MS/MS and GC-MS/MS methods, as well as about the chemical reagents used, is shown in Supplementary material (Tables S4 and S5).

2.4. Determination of glyphosate and related compounds

To measure glyphosate and their metabolites (Table S3), the QuPPE-PO-Method for analysis of highly polar pesticides in food involving extraction with acidified methanol and modified to measure soil samples was used [35]. Two aliquots of 5 g were collected from each sample and mixed with an internal pattern, 10 mL water with 2.5 % citric acid and

methanol with 2.5 % citric acid. From this mixture, 1 mL was used to measure through UPLC-MS/MS. The used column was an anionic Polar Pesticide Column (100 mm × 2.1 mm × 5 μm particle size), and the temperature was set at 50°C. Analytical measurements were conducted at Laboratorio Analítico Bioclinínico SL (Almería, Spain).

More information about the UPLC-MS/MS apparatus and conditions, as well as about the chemical reagents used, is shown in [Supplementary material \(Table S4\)](#).

2.5. Quality assurance and quality control

Three sets of multi-pesticide calibration standards were prepared for UPLC-MS/MS and GC-MS/MS multi-methods, as well as for the determination of glyphosate and related compounds. The calibration curves demonstrated satisfactory linearity in the response as a function of concentration, with correlation coefficients exceeding 0.98 and response residuals within ± 20 %. Each sample sequence included 4–5 fortified blank soil samples and 4–5 fortified soil samples. The recoveries obtained from the fortified soils ranged from 70 % to 130 %. Each of the 614 analytes was identified based on a) the retention time and peak shape of the respective reference standard (or the isotopically labeled internal standard) and b) the ion ratio, with the ratios between the quantification and confirmation transitions being within ± 30 % of the average ion ratio of the calibration standards. The responses for the UPLC-MS/MS and GC-MS/MS analytes were normalized to the response of Monuron D6 and PCB-118, respectively, while the response for glyphosate and AMPA was normalized to the response of the isotopically labelled analogues. All determinations were performed in duplicate, and the mean content of both aliquots was considered the content in the sample.

The LOQ for all studied pesticide residues for the UPLC-MS/MS and GC-MS/MS analytical methods was 0.005 mg kg⁻¹, while it was 0.01 mg kg⁻¹ for glyphosate and related compounds.

2.6. Data analysis

Only pesticide residue content equal to or above the respective limit of quantification (LOQ) was considered in the data analysis (data entries where the analyte content was below the LOQ were left empty) [2]. The homogeneity of variance was tested using Levene's test. When data were normally distributed, the statistical significance of differences between regions and farming systems was estimated using one-way ANOVA for equal (Bonferroni post-hoc test) and no equal variances (Games-Howell post-hoc test, which is robust to heteroscedasticity). Statistical tests and graphs were conducted using GraphPad Prism 8 and R 4.4.1 software. In order to understand whether the occurrence of pesticide residues according to their type and quantities is linked to specific soil characteristics (i.e., texture, soil pH, organic matter, total organic carbon, nitrogen forms, and CEC, among others), environmental conditions (average temperature and rainfall during the last 30 years and the 12 months prior to sampling), agricultural management practice [crop residues and legume incorporation, years under the same production system, rotation type (monoculture, short- and long-term rotation) or tillage type]), and wheat yield, a Spearman's correlation analysis was performed using the IBM SPSS v27 software [21].

2.7. Ecological risk assessment

The ecological risk assessment was conducted for the 20 most frequently detected pesticide residues using two distinct approaches: the toxicity exposure ratios (TER) approach and the risk quotients (RQ) approach.

2.7.1. Toxicity exposure ratios (TER) approach

TER values were calculated based on the maximum or mean measured environmental pesticide residue concentrations (MEC_{max} or

MEC_{mean} , in mg kg⁻¹) and the toxicity data for individual pesticides with respect to selected soil species. In this case, based on the available literature, two of the five EFSA soil organisms were selected: the earthworm *Eisenia fetida* and the springtail *Folsomia candida* [36]. Furthermore, the TER approach was used to evaluate both acute and chronic exposure risks. To determine acute exposure risks, TER values were calculated using the median lethal concentration (LC_{50}) values of each pesticide for *E. fetida* (no data were available in the literature for *F. candida*) following Eq. (1), whereas chronic exposure risks were determined based on the no-observed-effect concentration (NOEC) values of each pesticide for *E. fetida* and *F. candida* using Eq. (2) [36,37]:

$$TER_{acute} = \frac{LC_{50}}{MEC_{max\ or\ mean}} \quad (1)$$

$$TER_{chronic} = \frac{NOEC}{MEC_{max\ or\ mean}} \quad (2)$$

where LC_{50} is the median lethal concentration of a given pesticide for a specific soil species (mg kg⁻¹), $NOEC$ is the no-observed-effect concentration (mg kg⁻¹) of a given pesticide for a specific soil species, and MEC_{max} and MEC_{mean} are the maximum and mean values of the measured concentrations of a given pesticide in the soil (mg kg⁻¹).

Through this approach, both general and worst-case scenarios were evaluated by assuming the input of $MECs$ at average and maximum concentrations. In addition, threshold TER values for acute and chronic exposure risks for selected species were set at 5 and 10, respectively. If the calculated TER exceeds these threshold values, the exposure risks to certain species can be interpreted as negligible [23,37]. LC_{50} and $NOEC$ values for each pesticide and for the species *E. fetida* and *F. candida* were obtained from the PPDB database [38] and the literature [23,36,39] and are presented in [Table S6](#).

2.7.2. Risk quotients (RQ) approach

To calculate the ecological risks through the risk quotients (RQ) approach, the chronic toxicity of each pesticide towards the earthworm *E. fetida* and the springtail *F. candida* was considered, taking into account only the most sensitive soil species for each pesticide [39].

The risk quotient (RQ) for single pesticides was calculated according to the following equation (Eq. (3)) [23,40]:

$$RQ = \frac{MEC}{PNEC} \quad (3)$$

where MEC (mg kg⁻¹) is the measured concentration of a given pesticide in the soil, and $PNEC$ (mg kg⁻¹) is the predicted no-effect concentration for soils. The $PNEC$ for each pesticide was calculated as follows (Eq. (4)):

$$PNEC = \frac{NOEC}{AF} \quad (4)$$

where $NOEC$ (mg kg⁻¹) is the no-observed-effect concentration of the most susceptible species and AF represents an assessment factor and is used to reduce uncertainties related to the accuracy, model errors, lack of data used, and inevitable variability between laboratory exposure and field conditions. Based on the EFSA guidelines [41], distinct AF values are used depending on the group of organisms and pesticides considered. In this sense, the assessment factor (AF) can be set at 10, 50, 100, or 1000, depending on the amount of toxicity data available. In this context, an AF of 1000 was chosen if at least one LC_{50} at any ecological level was available; an AF of 100 was used if chronic assays were available; and an AF of 50 or 10 was used if two or three or more $NOEC$ values were available, respectively [36,39]. The AF values used for each pesticide are shown in [Table S6](#).

The RQ values were classified into four different levels of ecological risk: negligible risk ($RQ < 0.01$), low risk ($0.01 < RQ < 0.1$), moderate risk ($0.1 < RQ < 1$), and high risk ($RQ > 1$) [42].

Finally, for each field, the total sum of risk quotients (ΣRQ_{site}),

which denotes the total ecological risk of the mixture for the given locality, was calculated by summing all RQs for each pesticide residue quantified in the soil sample, with the assumption of a concentration addition (CA) effect among pesticide residues in the mixture [39]. The ΣRQ_{site} can be classified into four different levels of ecological risk: negligible risk ($\Sigma RQ_{site} < 0.01$), low risk ($0.01 < \Sigma RQ_{site} < 0.1$), moderate risk ($0.1 < \Sigma RQ_{site} < 1$), and high risk ($\Sigma RQ_{site} > 1$) [42].

3. Results

3.1. Overview of pesticide residues and the influence of agricultural farming systems on their distribution

At least one substance was detected in 141 out of the 188 analyzed fields (75 %), regardless of region or farming system. A total of 78 different compounds or metabolites were detected, primarily fungicides

(31 compounds), followed by herbicides (21 compounds), insecticides (15 compounds), 10 metabolites or degradation compounds, and one bird repellent (anthraquinone) (Tables S7 and S8). Of the detected residues, thirty-one were banned pesticides and their metabolites, while 44 authorized pesticides were detected. A total of 73 different residues were detected in conventionally managed fields, while 35 were detected in organically managed fields. Of all the residues found in organically managed fields, only one was allowed for organic farming: Spinosyn A, an insecticide isolated from *Saccharopolyspora spinosa* [43] (Table S3). However, this compound was only detected in one sample from the AC zone.

Considering all the studied zones, both the number of residues and the total detected residue content were significantly higher ($p < 0.01$) in conventional farming systems compared to organic farming systems (Fig. 1). In this sense, at least one residue was detected in 92 of the 93 sampled conventional fields (99 % of the sampled fields) (Fig. 2 and

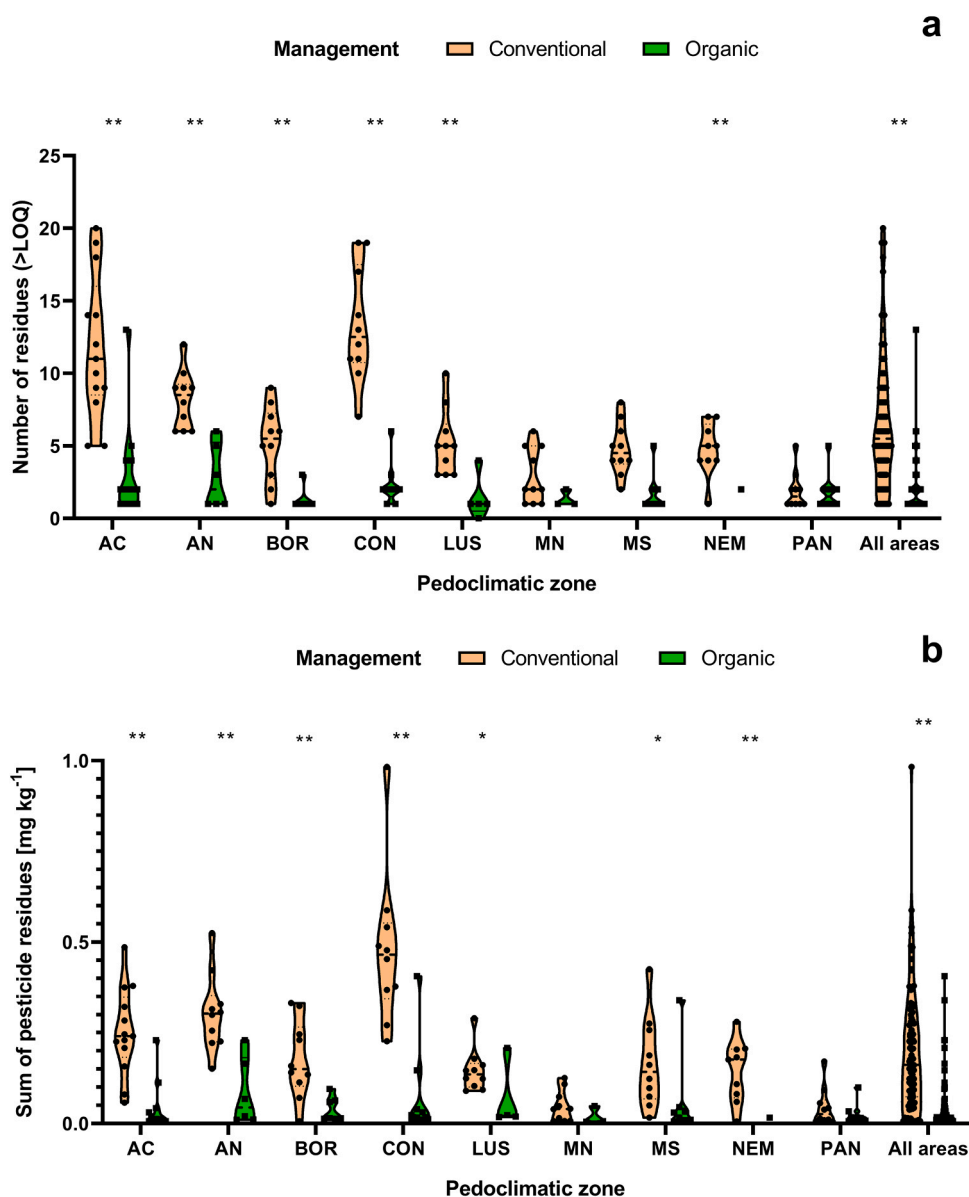


Fig. 1. (a) Number of pesticide residues in wheat fields under conventional and organic farming management system by pedoclimatic zone (Atlantic Central = AC; Atlantic North = AN; Boreal = BOR; Continental = CON; Lusitanian = LUS; Mediterranean North = MN; Mediterranean South = MS; Nemoral = NEM; and Panonian = PAN). (b) Sum of the concentrations of pesticide residues in the studied soils under both farming management systems by pedoclimatic zone. Black dots show each sampling point. If the number or sum of substances detected differed between organic and conventional management systems by pedoclimatic zone, the study zone is indicated by the significant levels of $p < 0.05$ as * or $p < 0.01$ as **.

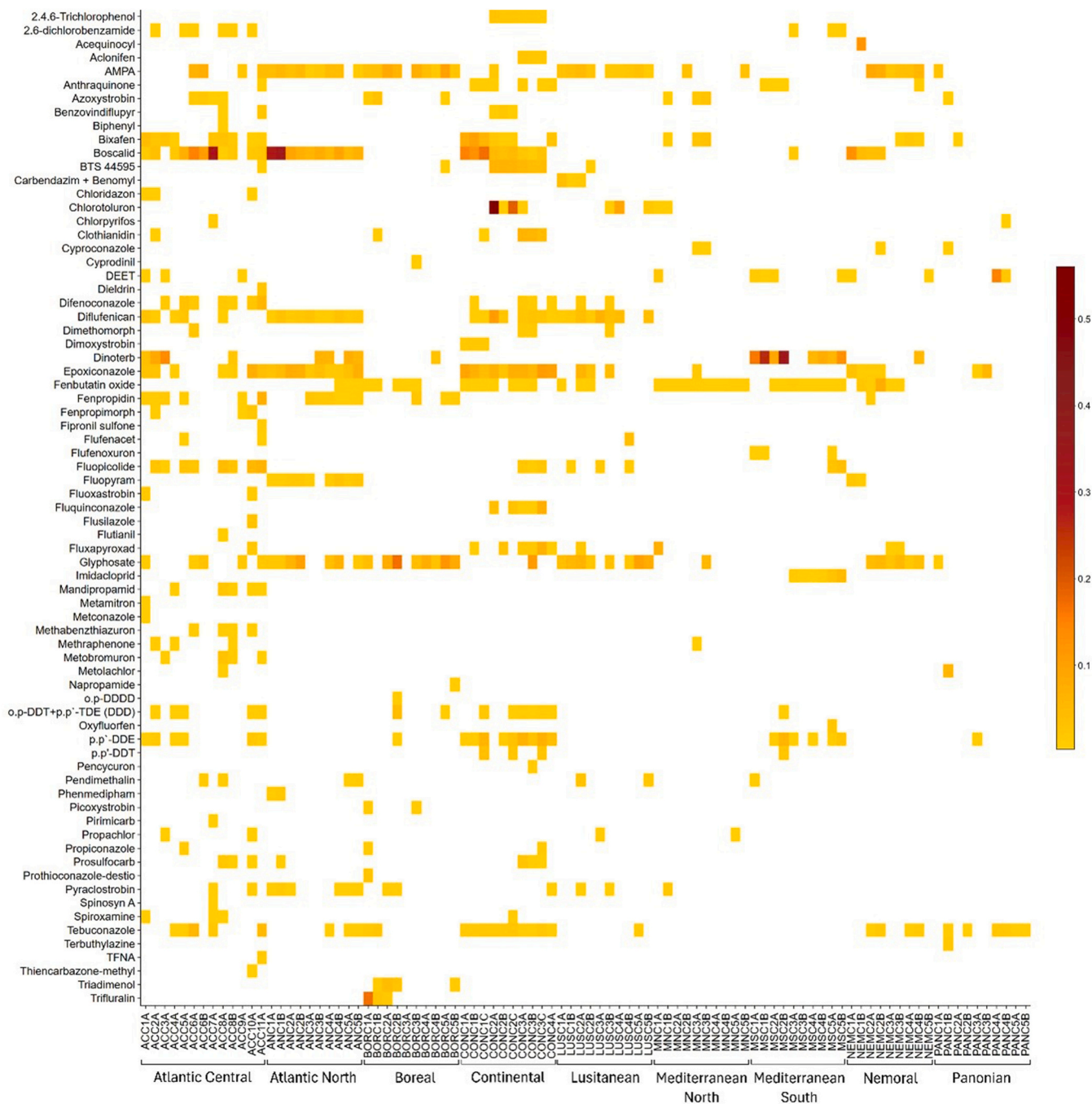


Fig. 2. Individual pesticide concentrations in mg kg^{-1} of the 73 residues detected in the conventionally managed fields. Only fields with at least one pesticide have been mapped. White filling indicates an absence of quantification of the substance (i.e., $< \text{LOQ}$). Rows represent pesticides, while columns represent sampling fields arranged by pedoclimatic zone.

Table S7), while in organic fields, at least one residue was detected in 51 of the 95 sampled fields (53.6 % of the sampled fields) (Fig. 3 and Table S8). Additionally, the maximum concentration of residues detected in an individual soil sample was up to 0.98 mg kg^{-1} for conventionally managed fields, and 0.40 mg kg^{-1} for organically managed fields (Fig. 1b, Tables S7 and S8). Furthermore, significant differences ($p < 0.01$) were also found between the two types of management systems (conventional and organic) within each zone in the AC, AN, BOR, CON, LUS, and NEM zones for both the number of pesticide residues and the sum of all residues. Moreover, significant differences ($p < 0.01$) between the two types of management system (conventional and organic) were also observed for the sum of residues in the MS region

(Fig. 1).

Regarding the differences between the studied zones, on the one hand, for conventional fields, the Continental zone (CON, Germany) showed the highest pesticide residue frequency (average value of 13.5), followed by AC (Denmark) (11 residues), AN (Belgium) (8.5 residues), LUS (NW Spain) (5 residues), BOR (Finland), MS (South-Eastern Spain) and NEM (Estonia) (4.5 residues), MN (South Spain) (2 residues), and PAN (Hungary and Serbia) (1.5 residues) zones (Fig. 1a). A similar pattern was also observed for the sum of pesticide residues, with the CON zone presenting the highest amount (average of 0.46 mg kg^{-1}), followed by AN (average of 0.30 mg kg^{-1}), AC (average of 0.24 mg kg^{-1}), NEM (average of 0.17 mg kg^{-1}), BOR (average of

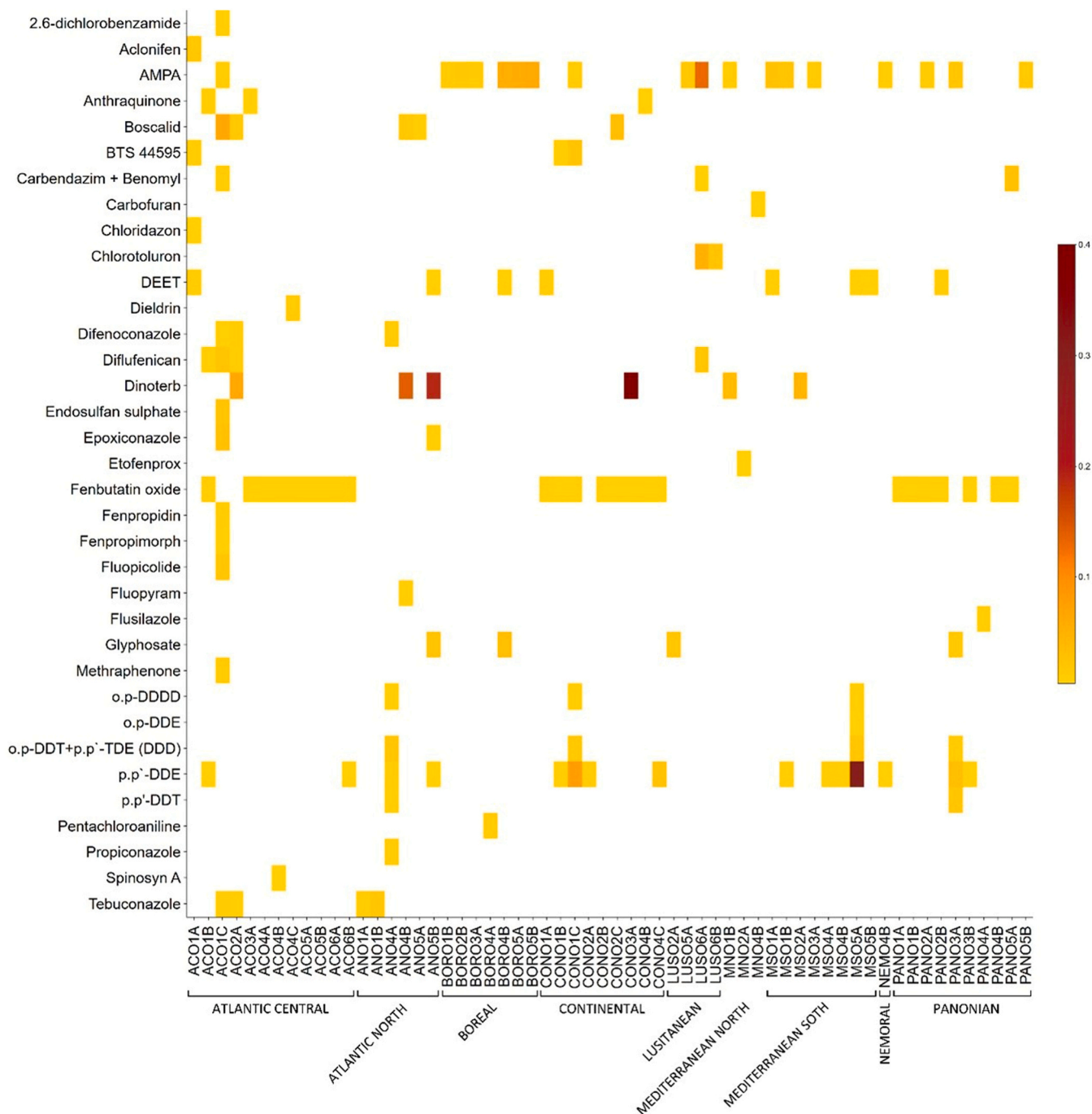


Fig. 3. Individual pesticide concentrations in mg kg^{-1} of the 35 residues detected in the organically managed fields. Only fields with at least one pesticide have been mapped. White filling indicates an absence of quantification of the substance (i.e., $< \text{LOQ}$). Rows represent pesticides, while columns represent sampling fields arranged by pedoclimatic zone.

0.14 mg kg^{-1}), MS (average of 0.14 mg kg^{-1}), LUS (average of 0.13 mg kg^{-1}), MN (average of 0.03 mg kg^{-1}), and PAN (average of 0.02 mg kg^{-1}) zones (Fig. 1b). The most frequently detected compounds in the conventional fields were fenbutatin oxide and AMPA (44 % of the studied samples), glyphosate and epoxiconazole (39 %), boscalid (formerly nicobifen) (37 %), diflufenican and tebuconazole (33 %), bixafen and p,p'-DDE (24 %), dinoterb (19 %), and pyraclostrobin (15 %) (Fig. 2 and Table S7). On the other hand, for organic fields, the AC and CON zones showed the highest pesticide residue frequency (average value of 3 residues), followed by PAN (2.5 residues), BOR, MS and AN (2 residues) zones. LUS, MN and NEM areas showed an average value of less than one pesticide residue since residues only appeared in

some samples (i.e., 4 of 13 samples in LUS) (Fig. 1a). Regarding the sum of pesticide residues, the highest amount was found in the AN zone (average value of 0.04 mg kg^{-1}), followed by CON, LUS and MS (average of 0.02 mg kg^{-1}), BOR (average of 0.015 mg kg^{-1}), AC (average of 0.012 mg kg^{-1}), NEM (average of 0.016 mg kg^{-1}), and PAN (average of 0.014 mg kg^{-1}) zones (Fig. 1b). As indicated for conventional fields, fenbutatin oxide and AMPA were the compounds most frequently detected in organic fields, with detection frequencies of 25 % and 19 %, respectively, followed by p,p'-DDE (15 %), DEET (8 %), dinoterb (6 %), boscalid (5 %), and glyphosate, diflufenican, o,p-DDT+p,p'-TDE (DDD) and tebuconazole (4.2 %) (Fig. 3 and Table S8).

Finally, it should be noted that, in general, both the highest pesticide

residue frequency and the highest concentrations of residues were observed in the regions located in western and middle Europe, specifically in the CON, AC, and AN zones (Fig. 1).

3.2. Influence of soil characteristics, agricultural management, and environmental factors on pesticide residues distribution

Wheat yield was positively and significantly correlated with the number of residues detected ($r = 0.475, p < 0.01$), with the total concentration of pesticides ($r = 0.316, p < 0.01$), and with the fungicide content ($r = 0.495, p < 0.01$) (Fig. 4).

When the relationships between soil properties and pesticide residues were evaluated, it was found that soil pH was correlated positively and significantly with the insecticide content ($r = 0.279, p < 0.01$), while it was correlated negatively and significantly with the total content of pesticide residues ($r = -0.182, p < 0.01$) and with the AMPA content ($r = -0.310, p < 0.05$). Regarding the soil organic fraction, OM and TOC contents were positively and significantly correlated with the glyphosate content ($r = 0.333, p < 0.05$) and with the bird-repellent content ($r = 0.720, p < 0.01$), respectively. With respect to soil texture, the clay content was negatively and significantly correlated with the number of residues ($r = -0.224, p < 0.01$), with the total concentration of pesticides ($r = -0.218, p < 0.01$), with the bird-repellent content ($r = -0.586, p < 0.05$), and with the fungicide content ($r = -0.518, p < 0.01$), while the sand content was negatively and

significantly correlated with the bird-repellent content ($r = -0.723, p < 0.01$). Finally, the silt content was positively and significantly correlated with the bird-repellent content ($r = 0.732, p < 0.01$) (Fig. 4).

Regarding the climatic variables, both T30y and T12m were significantly and negatively correlated with the total amount of pesticide residues ($r = -0.214$ and $p < 0.01$ for T30y, and $r = -0.234$ and $p < 0.01$ for T12m), with the metabolite content ($r = -0.249$ and $p < 0.05$ for T30y, and $r = -0.314$ and $p < 0.01$ for T12m), and with the AMPA content ($r = -0.278$ and $p < 0.05$ for T30y, and $r = -0.288$ and $p < 0.05$ for T12m). Additionally, T12m was also positively and significantly correlated with insecticide content ($r = 0.226, p < 0.05$) and significantly and negatively correlated with glyphosate content ($r = -0.457, p < 0.01$). Regarding precipitation, P30y and P12m were significantly and negatively correlated with glyphosate content ($r = -0.473, p < 0.01$) and insecticide content ($r = -0.210, p < 0.05$), respectively (Fig. 4).

Regarding agricultural management systems, the number of years under the same farming system was positively and significantly correlated with the number of residues ($r = 0.355, p < 0.01$) and with the total pesticide content ($r = 0.202, p < 0.05$). Concerning practices related to crop rotation, monoculture practices were negatively and significantly correlated with the content of bird-repellents ($r = -0.659, p < 0.05$), while short rotation practices were positively and significantly correlated with the content of bird-repellents ($r = 0.747, p < 0.01$). Long rotation practices were positively and significantly

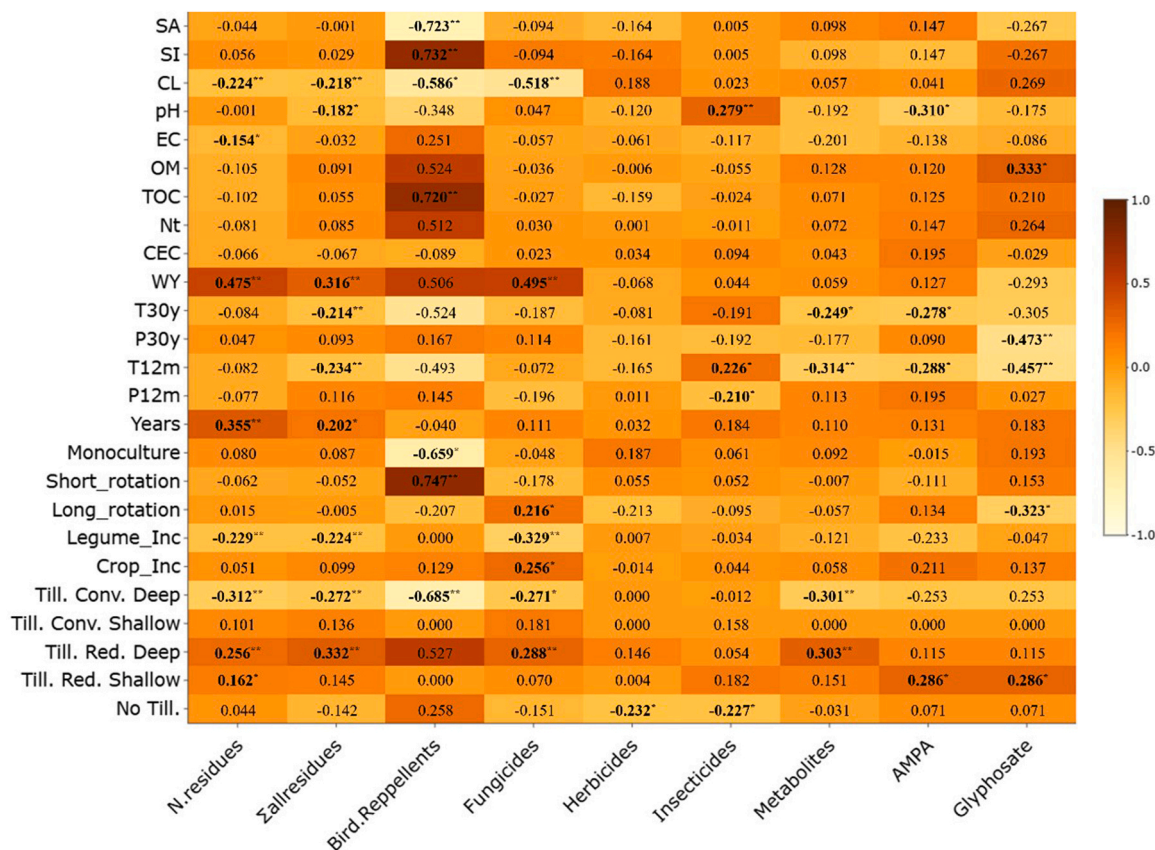


Fig. 4. Spearman's r values for correlations between the occurrence of pesticide residues and soil properties, environmental conditions, agricultural management systems and wheat yield. N. Residues, number of residues; Σallresidues, sum of total residues content; SA, sand content; SI, silt content; CL, clay content; pH, pH in water; EC, electrical conductivity; OM, organic matter content; TOC, total organic carbon content; Nt, total nitrogen content; CEC, cation exchange capacity; WY, wheat yield; T30y, average temperature of the last 30 years before sampling campaigns; P30y, average precipitation of the last 30 years before sampling campaigns; T12m, average temperature of the last 12 months before sampling campaigns; P12m, average precipitation of the last 12 months before sampling campaigns; Years, years with the same farming system; Legume Inc., legume incorporation to the soil; Crop Inc., crop wastes incorporation to the soil; Till. Conv. Deep, conventional deep tillage; Till. Conv. Shallow, conventional shallow tillage; Till. Red. Deep., reduced deep tillage; Till. Red. Shallow., reduced shallow tillage; No Till., no tillage; No Fert., no fertilization; Organic Fert., organic fertilization; Mineral Fert., mineral fertilization; OrgMin. Fert., organomineral fertilization. *Significant at $p < 0.05$. **Significant at $p < 0.01$.

correlated with the content of fungicides ($r = 0.216, p < 0.05$), and negatively and significantly correlated with glyphosate content ($r = -0.323, p < 0.05$). Legume incorporation was significantly and negatively correlated with the number of residues ($r = -0.229, p < 0.01$), with the total pesticide content ($r = -0.238, p < 0.01$), and with the fungicide content ($r = -0.329, p < 0.01$). Regarding tillage type, conventional deep tillage was significantly and negatively correlated with the number of residues ($r = -0.312, p < 0.01$), with the total pesticide content ($r = -0.272, p < 0.01$), with the content of bird-repellents ($r = -0.685, p < 0.01$), with the content of fungicides ($r = -0.271, p < 0.05$), and with the content of metabolites ($r = -0.301, p < 0.01$). On the contrary, reduced deep tillage was positively and significantly correlated with the fungicide content ($r = 0.288, p < 0.01$), and with the metabolite content ($r = 0.303, p < 0.01$), while reduced shallow tillage was also positively and significantly correlated with the number of residues ($r = 0.162, p < 0.05$), and with the contents in AMPA ($r = 0.286, p < 0.05$) and glyphosate ($r = 0.286, p < 0.05$) (Fig. 4).

3.3. Ecological risk assessment

3.3.1. Single risk assessment of pesticides by TERs

Table 1 shows the toxicity exposure ratio (TER) values for both under the general scenario (TER_{mean}) and the worst-case scenario (TER_{max}) calculated for the 20 most frequently detected pesticides in this study. It is important to note that a higher TER value indicates greater safety. In this context, if the calculated TER values exceed the trigger values (5 for chronic toxicity and 10 for acute toxicity), the exposure risks to the tested species can be considered negligible [37].

On the one hand, based on the TER approach, all the pesticides

studied presented negligible acute risk to the species *E. fetida*, both in conventionally managed fields and in organically managed fields, even under the worst-case scenario, with TER values exceeding the established trigger value for acute toxicity in all cases (Table 1).

On the other hand, regarding chronic toxicity, TER values were more frequently found to exceed the trigger value in conventionally managed fields than in organically managed fields. In this sense, in organically managed fields, only epoxiconazole showed exposure risks to *E. fetida*, with TER values below the chronic toxicity trigger value, both under the worst-case scenario ($TER_{\text{max}} = 3.6$) and under the general scenario ($TER_{\text{mean}} = 5.8$) (Table 1). However, in conventionally managed fields, different pesticides showed TER values below the chronic toxicity trigger value both under the worst-case scenario and the general scenario, indicating the presence of risk from certain pesticides to the species studied. Furthermore, based on the TERs, *E. fetida* was much more susceptible to chronic exposure risks than *F. candida*. In this regard, for *F. candida*, only the pesticide epoxiconazole showed a TER value below the trigger value in conventionally managed fields and under the worst-case scenario ($TER_{\text{max}} = 4.5$, Table 1). However, in the case of *E. fetida* under conventionally managed fields, four of the twenty pesticides studied showed TER values below the trigger value under the worst-case scenario, with TER_{max} values of 3.8, 3.7, 3.6, and 0.9 for imidacloprid, boscalid, difenoconazole, and epoxiconazole, respectively (Table 1). Additionally, under the general scenario and in conventionally managed fields, two pesticides, imidacloprid and epoxiconazole, also presented chronic exposure risks to *E. fetida*, with TER_{mean} values of 9.5 and 2.3, respectively (Table 1). Based on the obtained TER values, epoxiconazole is the pesticide that presents the most severe exposure risks to both species studied (*E. fetida* and *F. candida*).

Table 1

Maximal and mean toxicity exposure ratio (TER_{max} , TER_{mean}) values based on the selected soil species (the earthworm *Eisenia fetida* and the springtail *Folsomia candida*) and single pesticides. Trigger value: 10 for acute risk and 5 for chronic risk. TERs below the trigger value are presented in bold font.

Pesticide	<i>E. fetida</i> acute		<i>E. fetida</i> chronic		<i>F. candida</i> chronic	
	TER_{mean}	TER_{max}	TER_{mean}	TER_{max}	TER_{mean}	TER_{max}
Conventionally managed fields						
2,6-dichlorobenzamide	> 1000	> 1000	NA	NA	> 1000	> 1000
AMPA	> 1000	> 1000	694.1	281.2	> 1000	> 1000
Azoxystrobin	> 1000	> 1000	215.4	100.0	> 1000	833.3
Bixafen	> 1000	> 1000	> 1000	> 1000	290.0	77.4
Boscalid	> 1000	> 1000	14.8	3.7	> 1000	> 1000
Chlorotoluron	> 1000	892.9	313.0	55.8	> 1000	348.2
Clothianidin	429.6	216.6	81.3	41.0	NA	NA
Difenoconazole	> 1000	> 1000	10.8	3.6	> 1000	> 1000
Diflufenican	> 1000	> 1000	> 1000	441.8	> 1000	> 1000
Epoxiconazole	> 1000	> 1000	2.3	0.9	11.5	4.5
Fenbutatin oxide	> 1000	> 1000	190.4	22.3	NA	NA
Fenpropidin	> 1000	> 1000	744.2	163.4	> 1000	756.3
Fluopicolide	> 1000	> 1000	> 1000	992.1	> 1000	496.0
Fluopyram	> 1000	> 1000	754	380.7	> 1000	> 1000
Fluxapyroxad	> 1000	> 1000	895	295.8	> 1000	> 1000
Glyphosate	> 1000	> 1000	430.4	125.4	> 1000	> 1000
Imidacloprid	569.1	227.7	9.5	3.8	66.5	26.6
Pendimethalin	> 1000	> 1000	> 1000	> 1000	> 1000	> 1000
Pyraclostrobin	> 1000	> 1000	> 1000	> 1000	> 1000	> 1000
Tebuconazole	> 1000	> 1000	354.9	103.6	> 1000	> 1000
Organically managed fields						
2,6-dichlorobenzamide	> 1000	> 1000	NA	NA	> 1000	> 1000
AMPA	> 1000	> 1000	967.8	216.3	> 1000	> 1000
Boscalid	> 1000	> 1000	42.2	18.4	> 1000	> 1000
Chlorotoluron	> 1000	> 1000	833.3	601.0	> 1000	> 1000
Difenoconazole	> 1000	> 1000	20.0	15.4	> 1000	> 1000
Diflufenican	> 1000	> 1000	> 1000	> 1000	> 1000	> 1000
Epoxiconazole	> 1000	> 1000	5.8	3.6	28.7	17.7
Fenbutatin oxide	> 1000	> 1000	264.7	216.9	NA	NA
Fenpropidin	> 1000	> 1000	> 1000	> 1000	> 1000	> 1000
Fluopicolide	> 1000	> 1000	> 1000	> 1000	> 1000	> 1000
Fluopyram	> 1000	> 1000	> 1000	> 1000	> 1000	> 1000
Glyphosate	> 1000	> 1000	957.8	665.9	> 1000	> 1000
Tebuconazole	> 1000	> 1000	479.0	285.0	> 1000	> 1000

NA: data not available.

3.3.2. Single risk assessment of pesticides by RQ

Fig. 5 shows the percentage of conventionally and organically managed fields recorded for each risk level based on the risk quotients (RQ) approach for individual pesticides. As expected, the ecological risk of the compounds under study was significantly higher in conventionally managed fields than in organically managed fields. Specifically, in conventionally managed fields, the high-risk level was observed for 6 of the 20 most frequently detected pesticides: the fungicides epoxiconazole (with 35 % of conventionally managed fields exhibiting a high-risk level) and boscalid (9 %); the insecticides clothianidin (3 %) and imidacloprid (2 %); the fungicide difenoconazole (2 %); and the herbicide chlorotoluron (1 %) (Fig. 5). However, in organically managed fields, a high-risk level was only observed for the fungicide epoxiconazole (with 1 % of organically managed fields exhibiting a high-risk level) (Fig. 5).

On the other hand, a moderate-risk level was observed for nine pesticides in conventionally managed fields: boscalid (with 27 % of conventionally managed fields showing a moderate-risk level), difenoconazole (12 %), epoxiconazole (5 %), imidacloprid (4 %), clothianidin (3 %), fenbutatin oxide (3 %), bixafen (2 %), chlorotoluron (2 %), and azoxystrobin (1 %) (Fig. 5). However, in organically managed fields, only four pesticides exhibited a moderate-risk level, and at a lower percentage of fields compared to those observed in conventionally managed fields: boscalid (with 4 % of organically managed fields showing a moderate-risk level), difenoconazole (3 %), chlorotoluron (1 %), and epoxiconazole (1 %) (Fig. 5). Regarding the low-risk level, the percentage of fields exhibiting this risk level was lower in all cases for organically managed fields than for conventionally managed fields, similar to what was observed for the other risk levels (moderate and high-risk levels) (Fig. 5).

Finally, it is worth noting that, considering the three risk levels (low, moderate, and high), the insecticide fenbutatin oxide was the pesticide that showed ecological risk in the highest number of fields (45 % and 25 % of the conventionally and organically managed fields, respectively), followed by boscalid (38 % and 5 %), epoxiconazole (40 % and 2 %), tebuconazole (32 % and 4 %), AMPA (29 % and 4 %), and glyphosate (29 % and 2 %). In contrast, 2,6-dichlorobenzamide and pendimethalin showed no ecological risk in any of the fields under study (Fig. 5).

3.3.3. Mixture risk assessment of pesticides by RQ

Fig. 6 shows the percentage of conventionally managed fields (a) and organically managed fields (b) from each pedoclimatic zone categorized based on the total sum of risk quotients (ΣRQ_{site}), which represents the total ecological risk of the pesticide mixture present in each field.

On the one hand, regarding conventionally managed fields and considering all zones, it was found that 46 % of the fields exhibited a high-risk level, 18 % a moderate-risk level, 29 % a low-risk level, and 7 % a negligible-risk level. Additionally, the Continental and Atlantic North pedoclimatic zones exhibited the highest ecological risk levels, with 100 % of the conventionally managed fields in these zones exhibiting a high ecological risk level, followed by the Atlantic Central zone, with 85 % of conventionally managed fields exhibiting a high-risk level. Furthermore, 40 %, 30 %, 20 %, and 10 % of the conventionally managed fields from the Nemoral, Lusitanian, Mediterranean South, and Mediterranean North zones, respectively, exhibited a high-risk level, while the Boreal zone showed no fields with a high-risk level (Fig. 6a).

On the other hand, regarding organically managed fields, the ecological risks observed were much lower compared to those observed for conventionally managed fields. In this regard, considering all pedoclimatic zones, only 1 % of the organically managed fields exhibited a high-risk level, while 60 % showed a negligible risk, 6 % exhibited a moderate risk, and 33 % exhibited a low risk. Regarding the different pedoclimatic zones, the Atlantic Central zone showed the highest ecological risk, with 8 % of the organically managed fields exhibiting a high-risk level, 8 % exhibiting a moderate-risk level, and 75 % exhibiting a low-risk level. In contrast, the Mediterranean North, Mediterranean South, and Nemoral zones showed no organically managed fields with significant ecological risk (Fig. 6b).

4. Discussion

In this study, a high number of pesticides were analyzed following the recommendations of Riedo et al. [21]. Most previous studies only analyzed a few target pesticides or typical substances (between 50 and 200 substances), and not “cocktails” like our study. Additionally, in our study, historical and current agricultural farming system and topographical information were identified. In contrast some multi-country

	Negligible		Low		Moderate		High	
	Conv	Org	Conv	Org	Conv	Org	Conv	Org
2,6-dichlorobenzamide	100	100						
AMPA	71	96	29	4				
Azoxystrobin	88	100	11		1			
Bixafen	78	100	20		2			
Bosclaid	62	95	2	1	27	4	9	
Chlorotoluron	90	98	7	1	2	1	1	
Clothianidin	94	100			3		3	
Difenoconazole	86	97			12	3	2	
Diflufenican	98	100	2					
Epoxiconazole	60	98			5	1	35	1
Fenbutatin oxide	55	75	42	25	3			
Fenpropidin	92	100	8					
Fluopicolide	96	100	4					
Fluopyram	92	100	8					
Fluxapyroxad	97	100	3					
Glyphosate	71	98	29	2				
Imidacloprid	92	100			4		2	
Pendimethalin	100	100						
Pyraclostrobin	85	100	15					
Tebuconazole	68	96	32	4				

Number of soils (NS) for each risk level in %

0 < NS ≤ 10	10 < NS ≤ 25	25 < NS ≤ 50	NS ≥ 50
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Fig. 5. Ecological risk assessment in conventionally (Conv) and organically (Org) managed fields based on risk quotient (RQ) and its corresponding occurrence frequency (in %) for the twenty most frequently detected pesticides.

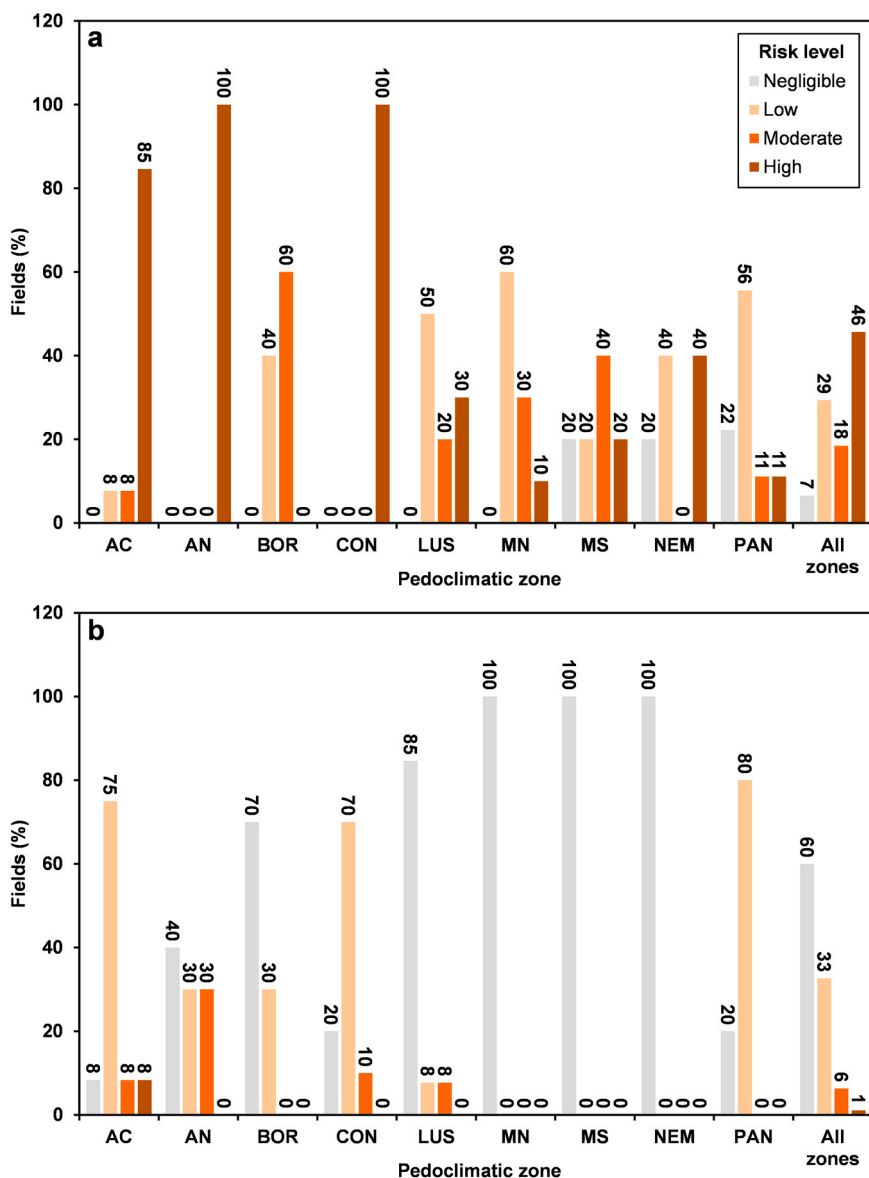


Fig. 6. Percentage of conventionally managed fields (a) and organically managed fields (b) from each pedoclimatic zone classified into the risk levels of negligible ($\Sigma RQ_{site} < 0.01$), low ($0.01 \leq \Sigma RQ_{site} < 0.1$), moderate ($0.1 \leq \Sigma RQ_{site} < 1$), and high ($\Sigma RQ_{site} \geq 1$), based on to the ΣRQ_{site} values, which represent the total ecological risk of the mixture for the given locality.

studies or reviews usually collect published data or lack historical and environmental information from each site (e.g., [24]), making it sometimes impossible to link it with the agricultural farming system.

4.1. Occurrence and distribution of pesticide residues

Fenbutatin oxide or bis[tris(2-methyl-2-phenylpropyl)tin] oxide, an acaricide banned in the European Union since 2015, four years before soil sampling, was the residue most frequently detected in both agricultural management systems (Figs. 2 and 3, Tables S7 and S8). This compound is widely distributed in the studied soils under both farming systems, mainly due to its extremely high octanol-water partition coefficient ($\log K_{ow} = 12.8$), negligible vapor pressure, and high chemical stability (half-life in soils ranging from 0.9 to 3.5 years), which favors its low mobility in soils [44]. This makes fenbutatin oxide a highly persistent pesticide in soils, as reported by different authors across several regions (i.e., [38,44–46]). In both agricultural management systems, fenbutatin oxide levels were very high (up to 0.075 mg kg^{-1}), much higher than previously reported for agricultural soils from the

archipelagos of the Macaronesia region (average of $0.000302 \text{ mg kg}^{-1}$) [44], but lower than those typically found in Mediterranean agricultural soils when its use was still allowed ($0.1\text{--}5 \text{ mg kg}^{-1}$) [45].

Despite their banning in the 1970–1980s in most European countries, DDT metabolites (i.e., p,p'-DDE and o,p'-DDT+p,p'-TDE) have also been found at a frequency of over 10 % (Figs. 2 and 3, Tables S7 and S8). Similar results have also been obtained by Alaoui et al. [47], who also studied the presence of pesticide residues in soils across ten European countries and detected p,p'-DDE in 85 % of the soils analyzed. The reported concentrations were similar, with p,p'-DDE as the predominant compound, as indicated by national-scale studies in Switzerland [48] or Spain [49–50]. These concentrations were lower than the reference value of 0.7 mg kg^{-1} for contaminated soils with DDT [51].

In the samples studied, 31 banned pesticides or metabolites were also detected, some of them included in the Stockholm list of POPs (Persistent Organic Pollutants) [52], such as dieldrin, DDT-related compounds, endosulfan sulphate, and pentachloroaniline (a metabolite of pentachlorobenzene). Chlorpyrifos, a pesticide approved at the time of sampling but banned since then and a potential candidate for POP listing

[53], was also detected in two conventionally managed samples in the AC and PAN zones (Fig. 2 and Table S7). These results, therefore, confirm the high persistence of these pesticides, even 40 years after their ban, as in the case of compounds related to DDT, in line with what has also been observed in various European studies (i.e., [44,47,54–55]). In this regard, most of these highly persistent compounds are chlorinated pesticides. Consequently, the presence of Cl atoms in their composition, which tend to form very strong bonds with other atoms, such as carbon, gives these compounds high chemical stability [56]. Moreover, these compounds have low solubility in water, being highly hydrophobic, with $\log K_{OW}$ values > 3.5 in all cases. As a result, these compounds exhibit strong adsorption to soils, primarily interacting through hydrophobic partitioning with the less polar fractions of soil organic matter [57–60]. This high affinity for soils, especially for soil organic matter, significantly limits both their loss through leaching into groundwater and their bioavailability to soil microorganisms, thus restricting their biodegradation and, ultimately, promoting their accumulation and persistence in soils [58,61].

Other pesticide residues, such as boscalid (formerly nicobifen), epoxiconazole, diflufenican, tebuconazole, dinoterb, bixafen or n,n-diethyl-m-toluamide (DEET), were also found in $\geq 10\%$ of samples. In general, these pesticide residues are typical contaminants in agricultural soils in different regions of the world, both at national (i.e., [21,23,26,62–64]) or multi-country/continental scales (i.e., [2,9,44,47]). The occurrence of some broad-spectrum fungicides such as boscalid, epoxiconazole (banned since 2020), tebuconazole, or pyraclostrobin, is a relatively typical issue in agricultural soils, water bodies or wheat grains (i.e., [21,25,65]), while other pesticides such as bixafen, diflufenican, dinoterb, or DEET are also highly persistent in soils and/or ubiquitous in different environmental matrices [3,66–68].

Glyphosate and its degradation products were widely detected in both managements (Figs. 2 and 3, Tables S7 and S8). Like fenbutatin oxide, glyphosate and aminomethyl phosphonic acid (AMPA), a glyphosate transformative compound, present a very high persistence in soils, even more than a year after its application, which is highly dependent on climatic conditions, persisting 30 times longer under cold and dry conditions than under warm and moist conditions [69]. In addition, the occurrence of glyphosate was accompanied by AMPA, or only AMPA was detected, which indicates glyphosate degradation (Figs. 2 and 3) since AMPA is more persistent in soils than glyphosate [69]. Moreover, they can be easily transferred due to wind and water erosion and redistributed by rain to non-glyphosate applied fields, thus justifying their widespread presence in soils [70].

In general, our data seem aligned with those reported by other researchers for European agricultural fields. Pan-European studies (i.e., [4, 7]) found two or more residues in 58–60% of analyzed samples, while in our case, two or more residues were detected in 88% and 63% of the conventional and organic fields, respectively. Geissen et al. [71], who analyzed 340 soil samples from Spain, Portugal and the Netherlands, found that 80% of the conventional fields contained residues of two or more pesticides. Other single-country studies also found similar or higher amounts of pesticide residue types, as indicated for France [26], the Czech Republic [62–63,72], Spain [73], Finland [23] or Switzerland [21]. In our study, AMPA was more frequent than glyphosate, similar to those reported worldwide [74], continental [47,70] or by national studies [23,75–76]. According to the literature, this pattern is expected since: i) DT_{90} for glyphosate is estimated at around 170 days while for AMPA around 1000 days [26], ii) AMPA persists 11–21 times longer than glyphosate under warm and moist conditions [69], and iii) given that AMPA is a byproduct of microbial breakdown, larger concentrations would be anticipated in plots subjected to repeated GBH treatments [77].

4.2. Pesticide residues and their persistence

For both management systems, banned compounds were still

frequently found in soil samples, highlighting the high persistence, in accordance with those reported by different authors [3,21,78]. In addition, the fact that pesticides occur on organically managed fields, where synthetic pesticides are not allowed, could be mainly due to: a) the short age of these fields under organic management, thus presenting pesticide residues derived from previous conventional management, and b) transfer processes [21,70]. In this sense, wind and water erosion processes can act as dispersers of pesticide residues, as highlighted by Silva et al. [70] for glyphosate and AMPA. In fact, our results indicated that pesticides can be detected in fields within 20–30 years under organic management or in areas not associated with previous agricultural practices (Fig. 7). This is aligned with Riedo et al. [21], who reported similar amounts of pesticide residues in conventional and organic fields, including organic fields with long-term under the same management (> 20 years), which presented up to 16 pesticide residues. In our case, the maximum amount of pesticide residues under organic management was found in a field recently converted (around one year) from conventional to organic management, with 12 pesticide residues. However, in fields with organic management over 10 years, 5–6 pesticide residues were still present; a similar number was found by Geissen et al. [71] from organic fields in Spain, Portugal and the Netherlands. In this sense, Riedo et al. [79] indicated that in the first 20 years after conversion to organic management, there are still pesticide residues from previous applications that can remain in the soil and can be detected. Furthermore, in the northern Europe, in Finland, pesticide residues were found in fields under organic farming for at least 10 years but at 75–90% lower concentrations than in conventionally farmed fields [23].

On the other hand, a significant increase in the number of residues can be observed over time for conventional fields, especially in the 20–30 years, which can be maintained over time, which agrees with what was observed by the significant correlation between pesticide residues and years under the same management (Figs. 4 and 7).

Organically managed sites showed a slight reduction both in the number of residues and in the pesticide content over time, although these differences were not significant ($p > 0.05$). These findings highlight the importance of pesticide persistence in the environment. Therefore, the occurrence of pesticide residues even in fields under long-term organic farming is expected.

4.3. Relationship between pesticide residues occurrence and wheat yield, soil properties, climatic variables, and agricultural management

Our results show higher yields in wheat production associated with increased pesticide applications, which reflect the critical role of pesticides in protecting crops from pests and diseases (Fig. 4). With regard to soil properties, the negative correlations between soil pH and total pesticide residues and AMPA contents (Fig. 4) suggest that alkaline soils may favor pesticide breakdown, especially for glyphosate into AMPA, as observed by Muskus et al. [80], while acidic soils may favor the persistence and accumulation of pesticide residues in soils. In this context, it is well known that adsorption is one of the main processes that govern the environmental fate of pesticide residues once they reach the soil [61]. Thus, higher adsorption of pesticides to soil constituents implies a lower bioavailability and, consequently, lower degradation. Similarly, higher pesticide adsorption also implies lower mobility towards other environmental compartments and, consequently, greater persistence of these compounds in soils [61,81]. Therefore, it is expected that pesticide residues tend to accumulate or persist in more acidic soils since the adsorption of most pesticides increases as the soil pH value decreases [81]. In addition to the pH effect discussed earlier, the association between OM and TOC with glyphosate and bird-repellents (Fig. 4), respectively, highlights the role of organic components in adsorbing and retaining these compounds in soils (i.e., [80,82–83]). In this sense, the crucial and positive role of soil organic matter in the retention of glyphosate and other pesticides was highlighted in previous

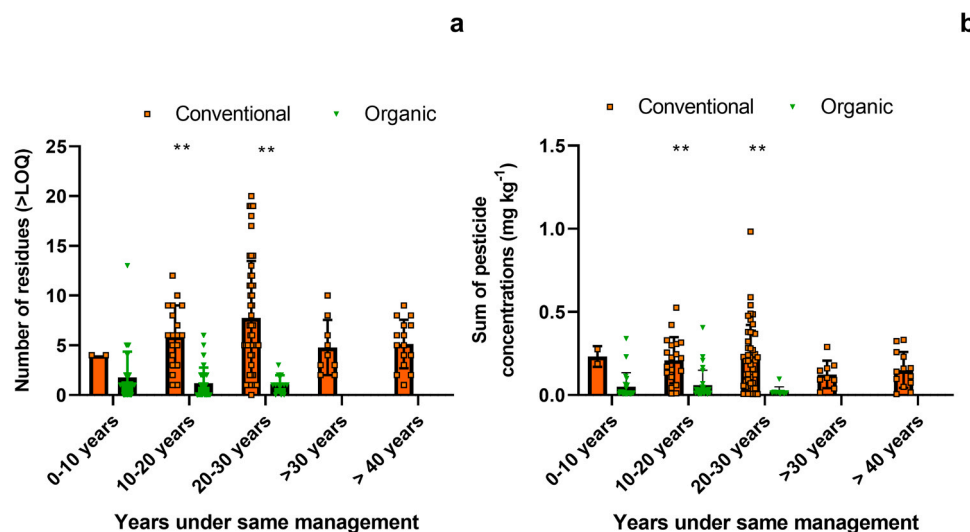


Fig. 7. Number (a) and sum of the concentrations (b) of pesticide residues detected in soils from conventional and organic management. The boxes represent the mean values of each management practice with their standard errors. Each dot represents a field. All sites are grouped in 10-year time intervals. *Significant differences between managements at $p < 0.01$.

studies [84–87]. Therefore, it is expected that the soils richest in organic matter will be those with the highest glyphosate residues.

Regarding soil texture, although the finest soil fraction (silt and clay) are widely recognized for their role in pesticide retention in soils [61, 88–89], and more specifically for glyphosate adsorption [90–91], in the present study no relationship was found between the presence of glyphosate or other pesticide residues and the clay content of the soils (Fig. 4). In this context, it should be noted that the role of clay as pesticide sorbent is less important when the contents of soil organic matter are moderate or higher [89,92].

Regarding climatic factors, correlation results indicated that the zones with higher temperatures and precipitation had lower residues concentration (Fig. 4). On the one hand, it is well known that a higher precipitation rate causes greater leaching of pesticides through the soil into groundwater, as well as greater runoff into surface water, thus leading to a decrease in pesticide concentrations in soils [93–94]. On the other hand, an increase in temperature also causes an acceleration of different processes related to the dissipation of pesticides in soils. First, an increase in temperature generally causes an increase in the pesticides solubility, thereby increasing the rates of uptake by plants and leaching through the soil, leading to a decrease in the concentrations of pesticide residues in soils. Secondly, an increase in temperature usually leads to an increase in the chemical and biological degradation of pesticides [95]. Therefore, pesticide residues are expected to exhibit higher persistence in soils from dry and cold regions compared to those from humid and warm regions [4]. This finding aligns with the higher detection frequencies and concentrations of pesticides observed in the European zones (CON, AC, and AN) of the northern latitudes compared to the zones (PAN, LUS, MN, and MS) of the southern latitudes in this study (Fig. 1).

Another factor that appears to play a key role in pesticide behavior in soils is agricultural management practices (Fig. 4). For example, a potential accumulation of pesticide residues over time is expected in monoculture or static systems. In addition, the incorporation of crop residues into soil can lead to an increase in pesticide retention, thus increasing their persistence in soils. In this sense, Alletto et al. [96] indicated that crop residues generally exhibit pesticide adsorption capacities 10–60 times higher than soil, primarily due to their higher organic matter content, which provides an increased surface area and functional groups such as hydroxyl, carboxyl, and phenolic groups, thereby enhancing pesticide binding. In addition, these crop residues may also contain pesticides, thus incorporating them into the soil. With

regard to the type of crop rotation implemented, the incorporation of legumes results in the fixation of atmospheric nitrogen, thus providing an additional source of nutrients for microorganisms, leading to an increase in microbial activity and diversity, which may favor the biodegradation of pesticide residues [97].

In general, conventional deep tillage practices resulted in a lower occurrence of pesticide residues, both in number and quantity. In contrast, more conservative agricultural practices, such as reduced deep tillage or reduced shallow tillage, resulted in a higher occurrence of pesticide residues (Fig. 4), indicating that reduced soil disturbance can enhance residue retention since this type of practice decreases the availability of pesticides for biological degradation [96]. In addition, the organic carbon content is higher at the soil surface and gradually decreases with depth. Therefore, considering that soil organic carbon content favors the adsorption and persistence of pesticide residues in soils, it is expected that higher pesticide residues persistence will occur at the surface under conservation tillage than under conventional tillage [96]. In addition, the nature and composition of organic matter, and therefore its reactivity, is also affected by the type of tillage. In this sense, different authors have observed that under conservation tillage soil organic matter presents a higher number of reactive functional groups compared to soil organic matter under a conventional tillage system. Similarly, the humification processes of organic matter are also more advanced in deeper soil layers [98].

4.4. Ecological risk assessment

The assessment of the negative impacts of pesticides on certain soil species, such as earthworms or springtails, is of special relevance, mainly due to the important functions of these soil organisms in terrestrial food chains, energy, and nutrient cycling. Thus, negative impacts on these soil organisms can lead to a significant loss of soil ecosystem services [99].

The fungicide epoxiconazole, which has not been approved for agricultural use since 2020 [100], is the pesticide that presents the highest risk to both earthworms and springtails, showing a high level of risk even in organically managed fields (Table 1 and Fig. 5). Despite the fact that epoxiconazole is not currently approved for use, its high environmental persistence, with a half-life of 362 days in soils [101], means it continues to be frequently detected in agricultural soils [102–105]. These findings are consistent with those obtained by Panico et al. [105], who evaluated the toxicity of contaminated soils in France

towards invertebrates and observed that epoxiconazole posed a significant risk to soil meso- and macrofauna (earthworms, enchytraeids, springtails, oribatid mites, and terrestrial snails). Similarly, Vaščková et al. [39] also indicated that long-term exposure of earthworms to epoxiconazole could present a high risk, leading to changes in earthworm communities and resulting in a loss of biodiversity.

Another pesticide that showed a high ecological risk in the present study was the fungicide boscalid (Table 1 and Fig. 5), in agreement with the findings of Alaoui et al. [47], who also reported moderate to high-risk levels for boscalid in agricultural soils from Europe and Argentina. In this same line, Zuo et al. [106], who evaluated the ecological risk of pesticides currently in use in vegetable soils, also determined that boscalid poses a chronic toxicity to *E. fetida*. In this sense, Han et al. [107] found that boscalid, especially in coexistence with other contaminants such as amoxicillin, causes damage to the intestinal barrier of earthworms, thus increasing their bioaccumulation and, finally, leading to severe oxidative stress and metabolic disorders in earthworms. These ecological risks of boscalid should be treated with special attention since boscalid is a recently used fungicide that is highly persistent in soils, with an estimated half-life of 254 days [103], as well as one of the most frequently detected pesticides in agricultural soils worldwide [71,102–103,108].

Other pesticides that posed a high risk to earthworms included the fungicide difenoconazole and the insecticides imidacloprid and clothianidin (Fig. 5). These findings are consistent with those of Panico et al. [105], who observed that azole fungicides and the insecticide imidacloprid contributed significantly to ecological risks in soils under conventional agricultural practices. Similarly, Alaoui et al. [47] also reported a high-risk level for difenoconazole in European soils, while Mu et al. [36] found that imidacloprid and clothianidin posed chronic exposure risks to soil biota, such as *E. fetida*. In fact, it is important to note that due to their toxic effects on bees and other pollinators, the European Union recently restricted the outdoor use of imidacloprid and clothianidin in all EU member states [109].

On the other hand, it is also worth noting that the insecticide fenbutatin oxide and the fungicide tebuconazole, although not presenting a high-risk level in any soil, exhibited a low-risk level in a significant number of soils (Fig. 5). In this regard, for fenbutatin oxide, which was highly detected in the soils analyzed in the present study, ecological risks are not commonly reported in the literature, which can mainly be attributed to the limited number of studies focusing on this pesticide. However, for tebuconazole, the results obtained in the present study align with those reported by Alaoui et al. [47], who found the same risk level (low risk) for tebuconazole in European soils. More specifically, Mu et al. [36], who assessed the ecological risks of different pesticides towards soil biota, concluded that tebuconazole and three other compounds (abamectin, chlorantraniliprole, and chlorpyrifos) were the most hazardous pesticides for soil biota, indicating that tebuconazole residues may cause chronic effects on *E. fetida*. Furthermore, Karas et al. [110] found that tebuconazole causes decreases in populations of ammonia-oxidizing bacteria and archaea, thus leading to a loss of soil biodiversity, as well as a decrease in soil ecosystem services.

It should be noted that the pesticides that posed a higher ecological risk were fungicides or insecticides, while herbicides such as glyphosate and AMPA presented only a low-level of risk (Fig. 5). This is in agreement with Zuo et al. [106], who showed that fungicides and insecticides had greater adverse effects on soil organisms compared to herbicides. In this sense, although both glyphosate and AMPA were among the most detected pesticides, being thus ubiquitous in agricultural soils, previous studies have also observed only low [47] or negligible ecological risks [23,64] for soils contaminated with these compounds. Indeed, Pelosi et al. [64] indicated that *E. fetida*, the most used earthworm species in risk assessment analysis, is less sensitive to pesticides than other earthworm species, so the calculated risks may be underestimated. In fact, these authors also reported that glyphosate-based herbicides at low concentrations can negatively affect certain earthworm species in terms

of growth, reproduction and activity. In this sense, Dominguez et al. [111] demonstrated that the presence of AMPA residues in soils causes the loss of body weight in juveniles of *E. andrei*, as well as a reduction in cocoon production.

5. Concluding remarks

The results highlight that pesticides are ubiquitous in agricultural fields and may impact soil health, leading to a loss of ecosystem services and, ultimately, posing potential risks to human and ecological health. Furthermore, it should be noted that fields located in the northern latitudes of Europe showed a higher presence of pesticides compared to those located in the southern latitudes. In addition, authorized and banned pesticides appeared in both farming management systems, including long-term organically managed fields that may retain pesticide residues from previous conventional management or by pesticide drift by nearby fields. Although organic fields had lower pesticide content than conventionally managed fields, they were not free of pesticide residues. This highlights the persistence of these compounds and their easy transfer between different environmental compartments, which enables them to be incorporated into the food chain, posing potential risks to human and ecological health.

Multi-national/continental, national or regional/local studies should consider the analysis of more substances and a wide range of pesticides, especially banned or non-used pesticides, due to their higher persistence. According to our results, it is expected to find several pesticides, even after several years from the last potential applications due to both their high persistence and wind and water erosion. Therefore, benchmarks for pesticide residue mixtures in agroecosystems should be defined. In addition, the potential transfer from soil to soil organisms and to water bodies should also be assessed.

Environmental implication

The presence of pesticides in agricultural soils is a major global concern, as it can lead to biodiversity loss, a decline in ecosystem services, and a decrease in the long-term sustainability of agricultural systems, posing a significant threat to food security. However, most studies focus on small areas and few pesticides due to testing costs. Thus, this study examines more than 600 pesticides in 188 soil samples from wheat fields across Europe. It also assesses contamination in areas where pesticide use is not authorized, revealing widespread pesticide residue presence and the persistence of banned substances in untreated soils.

CRedit authorship contribution statement

David Fernández Calviño: Writing – review & editing, Supervision, Resources, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Eva Lloret:** Writing – review & editing, Investigation, Data curation. **Marleena Hagner:** Writing – review & editing, Investigation, Data curation. **Irene Ollio:** Writing – review & editing, Investigation, Data curation. **Krista Peltoniemi:** Writing – review & editing, Funding acquisition, Data curation, Conceptualization. **Claudia Campillo-Cora:** Writing – original draft, Investigation, Data curation. **Merrit Shanskiy:** Writing – review & editing, Funding acquisition, Data curation, Conceptualization. **Vanesa Santás-Miguel:** Writing – original draft, Investigation, Data curation. **Marian Pöldmets:** Writing – review & editing, Investigation, Data curation. **Antía Gómez-Armesto:** Writing – original draft, Investigation, Data curation. **Kaire Loit:** Writing – review & editing, Investigation, Data curation. **Manuel Arias-Estévez:** Writing – review & editing, Writing – original draft, Data curation. **Kristian Koefoed Brandt:** Writing – review & editing, Funding acquisition, Data curation, Conceptualization. **Paula Pérez-Rodríguez:** Writing – review & editing, Writing – original draft, Visualization, Investigation, Formal analysis, Data curation. **Stefan Schrader:** Writing – review & editing, Investigation, Funding acquisition, Data curation,

Conceptualization. **Andrés Rodríguez-Seijo**: Writing – review & editing, Writing – original draft, Visualization, Formal analysis, Data curation. **Manuel Conde-Cid**: Writing – review & editing, Visualization, Investigation, Formal analysis, Data curation. **Lieven Waeyenberge**: Writing – review & editing, Investigation, Funding acquisition, Data curation, Conceptualization. **Raúl Zornoza**: Writing – review & editing, Funding acquisition, Data curation, Conceptualization. **Silvia Martínez-Martínez**: Writing – review & editing, Investigation, Data curation.

Ethical Approval, Consent to Participate and Consent to Publish

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Availability of data and materials

The authors confirm that the data supporting the findings of this study are available within the article and supporting information as a reusable data file that can be found at Fernández-Calviño et al. [28] (Available dataset at ZENODO <https://doi.org/10.5281/zenodo.10467153>).

Declaration of Competing 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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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.jhazmat.2025.138291](https://doi.org/10.1016/j.jhazmat.2025.138291).

Data Availability

I have shared the link to the data in the manuscript.

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