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Field dissipation of pesticides used in Norwegian carrot production and pesticide residues in carrots in Norway compared to China, the European Union and Germany

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Preface and Acknowledgment

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Ås

Summary

The use of pesticides has not only positive effects on yield and food safety but also poses a risk to the environment and human health. Residues of pesticides are frequently detected in food crops, water, and soil, but a lack of knowledge regarding field dissipation and residues in soil under current agricultural practices was identified by Silva et al., (2019).

The aim of this study was to assess the status of pesticide residues in carrots and their dissipation in soils from carrot production in Norway under current agricultural practices and to compare it to available data from the EU, Germany, and China. To achieve this, data on pesticide residues in carrots from Norway, the EU, Germany, and China was compiled and compared. To determine the dissipation of pesticides in soil under current agricultural practices a field study was conducted on two carrot fields in southeast Norway. Soil samples were taken from May 2019 – April 2020 and extracted with acetonitrile. Dissipation rates were modelled and compared to existing data.

From the literature study it was found that most pesticides detected in carrots in the EU are fungicides. In all three regions investigated (Norway, the EU, Germany) boscalid was the most detected pesticide in carrots. When pesticide residues in Europe were compared to China, it was found that many pesticides used in China are banned in the EU. Unlike in the EU, mainly insecticides were detected in China. In all regions, residues of non-approved and sometimes long banned pesticides were found in carrots and vegetables.

In the field study, boscalid, cyprodinil, fludioxonil, metribuzin, and pyraclostrobin were sprayed in 2019 and detected in the soil samples. Additionally, several background pesticides were detected, of which some had been sprayed prior to 2016, indicating long persistence in soils with calculated half-lives of over 1000 years. Of all pesticides detected in the soil, most were fungicides. For pesticides that had been sprayed in 2019, metribuzin and pyraclostrobin showed similar half-lives to those reported in the EU. For boscalid the half-life was found to be shorter, probably as a result of uptake into carrots and potentially leaching events during winter. Cyprodinil and fludioxonil were found to have a longer persistence in soils than previously reported, which was concluded to be a result of cold climate conditions due to application late in the season, or lower amounts taken up by plants.

Table of contents

Preface and Acknowledgment.....	2
Summary	3
List of Abbreviations.....	6
1. Introduction	7
Hypothesis and aim of the study.....	10
2. Background information	11
2.1 Glossary.....	11
2.2 Fate of pesticides in soil	12
2.2.1 Sorption and desorption	13
2.2.2 Degradation and persistence of pesticides	14
2.3 Kinetic models for describing degradation and dissipation of pesticides	16
2.4 Pesticide legislation	17
2.4.1 Legislation in Norway, the EU, and Germany	17
2.4.2 Pesticide legislation in China	19
3. Material and Methods.....	20
3.1 Project description	20
3.2 Literature study.....	20
3.3. Field study	21
3.3.1 Location.....	21
3.3.2 Soil properties	22
3.3.3 Agricultural practices	23
3.3.4 Soil sampling.....	27
3.4 Analysis of pesticides in carrots	28
3.5 Extraction of pesticides from soil.....	28
3.5.1 Recovery study in soil	28

3.5.2 Final extraction.....	29
3.6 Modelling of DT50 values in soil.....	30
4. Results	34
4.1 Literature study	34
4.1.1 Pesticides approved in carrots	34
4.1.2 Pesticide residues in carrots	37
4.2 Recovery study	49
4.3 Field study	50
4.3.1 Pesticide residues in carrots from HV	50
4.3.2 Pesticide residues in soil at HV and HØ	50
4.3.4 Calculated dissipation for pesticides detected in the field study.....	55
5. Discussion	61
5.1 Comparison of pesticide residues in carrots and vegetables	61
5.2 Pesticide residues in soil at HV and HØ.....	64
5.2.1 Cooccurrence of pesticides in soil.....	64
5.2.2 Pesticides sprayed in 2019 and their concentration in the soil.....	65
5.3 Field dissipation and DT50 values of pesticides sprayed in 2019.....	66
5.4 Persistency of pesticides sprayed in previous years (before 2019)	69
5.5 Consequences and significance of pesticide residues in food crops and soil.....	71
5.6 Uncertainties and limitations	71
6. Conclusion.....	73
References	74
Appendix	85

List of Abbreviations

EFSA	European Food Safety Authority
MRL	Maximum Residue Level
JOVA	The Norwegian Agricultural Environmental Monitoring Programme (Program for jord- og vannovervåking i landbruket)
CAKE	Computer Assisted Kinetic Evaluation
PPDB	Pesticide Properties Database of the University of Hertfordshire
K_{oc}/K_{foc}	Soil organic carbon sorption coefficient
IPM	Integrated Pest Management
GAP	Good Agricultural Practices
BVL	Bundesministerium für Verbraucherschutz und Lebensmittelsicherheit
WRB	World Reference Base of Soil Resources
SFO	Single-First-Order kinetics (degradation kinetics)
FOMC	Gustafson-Holden kinetics (First-Order-Multi-Compartment)

1. Introduction

Yield loss due to pests had always been a struggle in agriculture, but with the extensive use of synthetic pesticides since the 1960s, yields of major crops have been increasing to more than double (Oerke, 2006). Without any crop protection yield losses would be substantial, with the highest potential yield loss due to weeds at around 34 % (Oerke, 2006). As a result, copious amounts of pesticides are used every year. Since the 1990s, the amount of pesticides sprayed worldwide increased from over 2 Mt sprayed in 1990 to over 4 Mt in 2018 with herbicides being the main group followed by fungicides and insecticides (FAOSTAT, 2020). With an increasing population expected to reach over 9 billion people by 2050, food production and yields need to further increase to meet the demand (Carvalho, 2006; Zhang et al., 2011). Additionally, climate change could further increase the use of pesticides with pests like weeds, pathogens, and animal pests possibly altering their distribution as a response to climate change (Bebber et al., 2013). Northern climatic zones especially might see improved overwintering conditions for pests and invasive species, which could result in an increased use of pesticides (Stenrød et al., 2016). Overall, pesticides contribute to food security world-wide but their (incorrect) use can have negative aspects.

Of applied pesticides, little reaches its target. It had been estimated that it can be as little as 0.1 %, with the rest entering the environment (Pimentel, 1995), resulting in environmental problems and human health issues. In a study about the effects of intensive agriculture in Europe, pesticides were found to have the most negative consequences, due to significant reduction in diversity of plants and animals (Geiger et al., 2010). Further threats include effects on aquatic organisms, soil biota, and food webs (Mahmood et al., 2016), as well as the development of resistant organisms (Carvalho, 2006). Pesticide residues can be detected in water, air, soil, and could potentially reach humans. As a result, numerous negative effects of pesticides on human health had been reported. This can include acute effects such as food poisoning and long term effects such as cancer, allergies, neurotoxicity, and damage to organs (Mahmood et al., 2016). For humans, the main routes of exposure are direct through occupation or indirect through drinking water, air, and food (Kim et al., 2017).

The amount of pesticides entering the environment depends on application rates, which vary greatly between countries and are much higher in China (13.07 kg/ha), compared to Germany

(3.77 kg/ha), the EU (1.06 kg/ha), and Norway (0.76 kg/ha) (FAOSTAT, 2020). China is also one of the largest consumer and producer of pesticides in the world (Lin et al., 2020), and the largest producer of vegetables and carrots in the world (FAOSTAT, 2020). Fruits and vegetables have high rates of pesticide application among food commodities (Zhang et al., 2011), and consequently a high percentage of samples show residues. In the report of the European Food Safety Authority (EFSA) from 2017; 62.1 % of fruits and nuts, and 43.1 % of vegetables had quantifiable residues (EFSA, 2019). For China, Chen et al. (2011) reported that 26 % of fruits and vegetables contained residues at or below the Maximum Residue Level (MRL), and 11.7 % contained residues over the MRL. Additionally, Fantke et al. (2012) reported higher health impacts from pesticide use in grapes, fruits, and vegetables in Europe compared to cereals, maize and potatoes.

In an effort to minimize negative effects associated with pesticides, most countries have rules and regulations in place which often include MRLs and monitoring programs for pesticide residues in food and in the environment. The EU coordinated control program on pesticide residues in food, or the JOVA program for residues in water bodies in Norway can be mentioned as examples. Furthermore, both China and the EU, among others, are attempting to make policy for sustainable use of pesticides. The EU has proposed a “farm to fork strategy” and a “pollution-free Europe”, with the goal of 50 % reduction in pesticide use by 2030 (EC, 2019). In China, the Action Plan for Zero Growth of Pesticide Use by 2020 was passed in 2015 (Shuqin & Fang, 2018). However, little attention was given to pesticide residues in soil, despite the important role of soils and microbial communities in pesticide degradation. The transport of pesticides via leaching or runoff into water bodies (Walker et al., 2012), and the potential uptake of residues from soil into food crops, are also important aspects resulting from pesticide residues in soils. Especially crops with edible parts in direct contact with soil, such as carrots, have a higher potential of accumulating pesticides and other contaminants from the soil (Trapp & Legind, 2011).

Studies on pesticide residues in soil, often focus on specific classes of pesticide like organochlorine pesticides (Zhou et al., 2013), or single pesticides like metribuzin (Conn et al., 1996), or glyphosate (Bento et al., 2016), without considering other pesticides. Nevertheless, a recent study from Europe (Silva et al., 2019) found, that over 80 % of analysed soils, contained pesticide residues. The same study also found that soils from the Southern regions of the EU had the highest frequency of soils without residues compared to the Northern, Western, and

Eastern EU regions. A study from Czech, where soils from agricultural sites were analysed from 2013-2017, reported that up to 69 % of residues could be inherited from the previous growing season (Kosubová et al., 2020).

Ideally, pesticides applied to agricultural fields should not accumulate in soil but should be degraded. The degradation of pesticides depend on different factors such as properties of the pesticide (Hvězdová et al., 2018), but also climatic conditions and soil properties like organic carbon, clay content, microbial activity, and pH (Kah et al., 2007). Laboratory studies tend to overestimate the degradation compared to field studies due to the elimination of some of these factors (Díez & Barrado, 2010), making field dissipation studies an important part of environmental exposure risk assessments. Especially in Norway, environmental factors play a key role. The combination of soils poor in organic carbon, with low microbial biomass, and low to moderate soil temperatures were found to lead to very long persistence of pesticides in soils in Norway, and it has been suggested that Norwegian conditions are not well represented in the climatic scenarios used for approval of pesticides in the EU (VKM, 2015). Field trials are often carried out in unrepresentative countries like Germany, Switzerland, and Spain (Almvik et al., 2014), yet those studies are used for the risk assessment. Risk assessments include harmful effects to humans, the environment, and persistence in the environment (EC, 2009b). To take differences in climatic conditions into consideration, different climatic zones are used for the approval and modelling of fate of pesticides in the EU.

Norway is part of the Nordic Zone together with Denmark, Finland, Sweden, Estonia, Latvia, Lithuania, and Iceland (VKM, 2015). However, the zones are very broad and Norway alone has 3-4 different climatic zones, with most of them falling into the worst case or extreme worst-case categories for temperature and precipitation (FOCUS, 2001). In Almvik et al. (2014) it was demonstrated that the DT50 values for boscalid on a European level were reported within 27-208 days, but under Nordic conditions DT50 values ranged up to 1000 days, pointing towards colder climates having a longer persistency of pesticides in soils than previously thought.

Degradation of pesticides depend on many different factors, but dissipation rates are often determined in lab studies or with field trials, rather than real world scenarios. A lack of data on pesticide residues in soil under common agricultural practices have been identified (Silva et al., 2019), which is important for further exposure analysis, and to reduce the risks associated with pesticide residues in the environment.

Hypothesis and aim of the study

The thesis was part of the project LowImpact at NIBIO, which aims towards a climate smart vegetable production with reduced pesticide residues in food, soil and water in China and Norway. The main objective of the thesis was to assess the current situation of pesticide residues and dissipation rates in soils under a real-world scenario, as well as the current level of pesticide residues in carrots. To address the objectives, soil samples were taken from a carrot field in south-eastern Norway over the course of one growing season and screened for pesticides and metabolites. A software (CAKE) was used to model their dissipation. Additionally, information from monitoring programs on pesticide residues in carrots in Norway, the EU, Germany, and China was compiled and compared to assess the current situation of pesticide residues in carrots.

The Hypotheses were

- a) The dissipation of pesticides under current agricultural practices in Norway will be slower compared to field dissipation in the EU and China, due to cold climate conditions.
- b) Differences in approved pesticides, as well as agricultural practices, pests, and regulations, will influence pesticides detected in food commodities and therefore lead to differences among regions. It was hypothesised, that more residues will be found in produce from China compared to produce from countries in the EU, due to usage of more toxic substances, higher application rates and differences in food safety regulations.

2. Background information

2.1 Glossary

MRL

Maximum Residue Levels (MRL) are defined as the upper legal level of a pesticide concentration in food resulting from authorised agricultural practices. Food with residues at or below the MRL are considered safe for the consumer (EFSA, 2010).

DT50/ DT90

The time it takes for 50 %/ 90 % of a substance to disappear from an environmental compartment (water, soil, etc.) by dissipation processes.

MF-value

Concentration level which indicates potential negative effects to aquatic organisms. Used for risk assessments of pesticides.

Degradation

The process of breaking down a substance via microbial degradation, hydrolysis, and photolysis, by transforming them into degradation products (FOCUS, 2006). It also includes oxidation, transformation into microbial biosynthates, or polymerisation products.

Dissipation

The overall process leading to the eventual disappearance of substances from the environment. It includes two main types of processes: transfer processes, such as volatilisation, leaching, plant uptake, run-off, erosion as well as transferring substances to different environmental compartments, and degradation processes (FOCUS, 2006).

Half-life

Time taken for 50 % of a substance to dissipate from a compartment described by single first-order kinetics.

2.2 Fate of pesticides in soil

Pesticides can enter the soil via different pathways; through drift when applying a pesticide to a crop, through wash-off from treated crops, or granulate that is directly applied to the soil (Fig. 1). After entering the soil, the fate and persistency of pesticides in the environment is affected by degradation, sorption, and transportation (Arias-Estévez et al., 2008). Pesticides can be transported or leach through the soil via water flow into rivers, streams, and groundwater and are frequently detected in water sources (Stenrød, 2015). Leaching occurs when pesticides are dissolved in the soil water. Consequently the properties of the soil and the pesticide determine if the pesticide will be in the water phase or sorbed to soil minerals (clay) and/or organic matter (OM) (Walker et al., 2012). In this study the focus will be on sorption and degradation, but in the schematic overview given in Fig. 1, transportation and leaching are included to give a more complete overview of the processes.

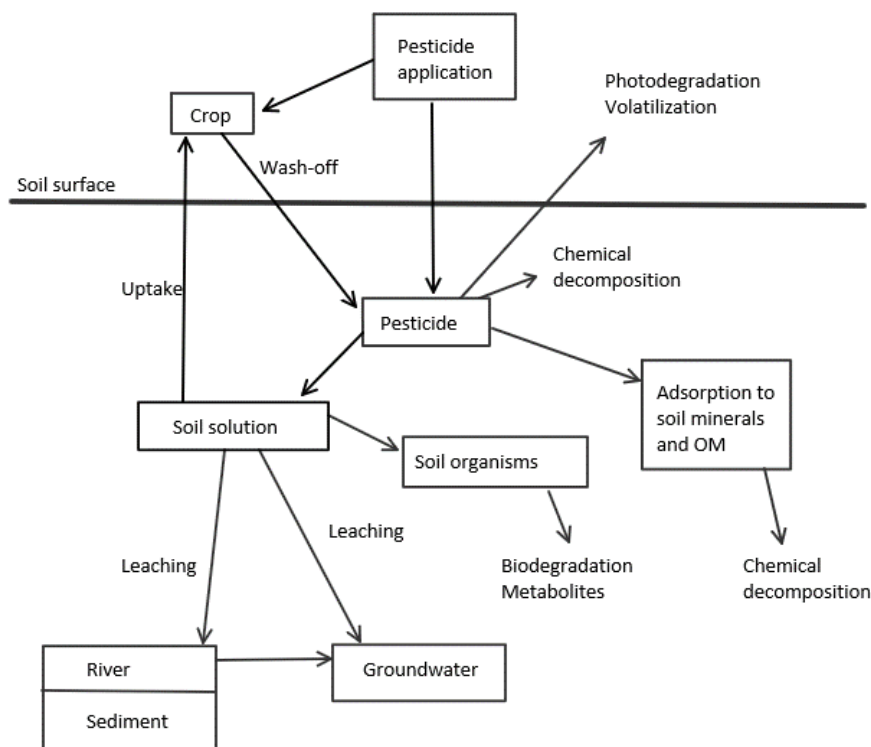


Figure 1: Pathways of pesticides entering the environment and their fate in soil (after Arias- Estévez et al. (2008) and Blume et al. (2016)).

2.2.1 Sorption and desorption

The availability and fate of a pesticide is determined by their sorption capacity. Sorption of pesticides in soils is a result of interactions based on different mechanisms like van der Waals forces, hydrogen bonds, and charge exchange. The formation of chemical complexes, covalent or ionic bonds, and ligand exchange also play an important role. Moreover, micro- and nanopores of soil particles contribute to sorption and desorption kinetics (Blume et al., 2016). Especially clay minerals and organic matter (OM) have large surface areas with a high sorption capacity, making them important factors for sorption dynamics of pesticides, and therefore restricting their availability and movement in the soil (Walker et al., 2012).

Typically, the topsoil (A horizon) is richer in OM and often has a higher biological activity, increasing the capability of microorganisms to degrade pesticides. However, the sorption to OM might decrease the availability of pesticides with a strong sorption affinity, but at the same time the degradation of pesticides with a low sorption ability might be promoted (VKM, 2015).

To be able to compare the sorption ability of substances, sorption coefficients (K_d) are used (Blume et al., 2016), and describe the tendency of a chemical to bind to soils. Due to the influence of organic carbon on sorption, corrections can be made which are expressed as soil organic carbon sorption coefficients (K_{oc} and K_{foc}). The values are dependent on the soil type, soil pH, acid-base properties of the pesticide, and the type of organic matter in the soil. K_{oc} is a linear and K_{foc} is a non-linear parameter (PPDB, 2007). To compare the K_{oc}/K_{foc} values, pesticides are divided in different categories: <15 = Very mobile, $15-75$ = Mobile, $75-500$ = Moderately mobile, $500-4000$ = Slightly mobile, > 4000 = Non-mobile (PPDB, 2007).

Generally lipophilic pesticides tend to adsorb to soil particles and hydrophilic pesticides tend to dissolve in water (Walker et al., 2012).

Important soil properties that influence the fate of pesticides are the pH of the soil and the activity and distribution of microorganisms (Kah et al., 2007). The soil pH both influences the the sorption ability of pesticides as well as the activity of microbes are influenced by the soil pH, for example a pH higher than 8-8.5 seems to restrict the growth of microorganisms. The behaviour of pesticides is also influenced by the pH and an increased rate of degradation of ionizable compounds with increased pH was observed (Kah et al., 2007). An overview of factors influencing the fate of pesticides in the environment is given in Table 1.

Table 1: Factors influencing the fate of pesticides in soil, after Arias-Estévez et al. (2008) and modified.

Pesticide	Soil	Climate
Chemical nature	Site	Temperature
Volatility	- Elevation, slope, geographical location	Rainfall, humidity, evaporation
Solubility	- Plant cover (species, density)	Seasons/ seasonal variation
Formulation	- Microbial populations	Solar radiation
Concentration	- Agricultural practices (tillage, fertilizer, ...)	Wind, air movement
Application	Soil type	
- Method	- Texture (especially clay)	
- Time of the year	- Structure, compaction	
- Frequency	- Organic matter	
- Amount	- Soil moisture	
	- Mineral content	
	- pH	

Once a pesticide is sorbed, different processes like changes in pH and electrolyte concentration as well as elevated microbial activity can lead to desorption, mobilisation, and leaching (Blume et al., 2016). Additionally, freezing-thawing cycles have been shown to increase risk of leaching under Norwegian conditions by promoting desorption (Holten et al., 2019; Stenrød et al., 2008). Desorbed pesticides are bioavailable and can be transported, degraded, or taken up by plants, making this process important for environmental exposure.

2.2.2 Degradation and persistence of pesticides

Pesticides can be degraded or transformed either chemically or biological. Chemical degradation of pesticides takes place in form of hydrolysis, oxidation, isomerisation, volatilisation, and photochemical breakdown (Walker et al., 2012). For a pesticide to be biodegraded it needs to be available to microorganisms (bioavailable), hence not sorbed to soil particles or OM, but dissolved in the soil water.

Over time, the bioavailability and extractability of pesticide can decrease, and non-extractable residues can be formed via covalent bonds and incorporation into humic substances. This leads to sequestration or aged sorption, which has the potential to decreasing the dissipation time of pesticides in the environment, and therefore reduces the risk of exposure for soil and water organisms (Blume et al., 2016).

Properties of pesticides that influence the degradation are molecule size and structure, functional groups, solubility, polarity, charge distribution, and acid-base properties. Especially hydrophilic pesticides are easily transported in water and therefore readily available to microorganisms, usually making them not very persistent (Walker et al., 2012).

Soil factors influencing the microbial populations in the soil are temperature, humidity, pH, and nutrient status (Blume et al., 2016). Generally, environmental conditions that favour microbial growth also favour biodegradation of pesticides, making degradation much faster under aerobic conditions and higher temperatures compared to anaerobic conditions and lower temperatures (Blume et al., 2016). In an ideal case, the pesticides are mineralized to water and CO₂, with interim metabolites being formed (Blume et al., 2016). Metabolites behave differently in the environment compared to their parent component, and have the potential to be more toxic (Andreu & Picó, 2004), but little is known about degradation products of pesticides.

Persistency, together with bioaccumulation and toxicity (PBT), are criteria in environmental risk assessments. To describe the persistence of a substance, dissipation half-life (DT₅₀) values are used. DT₅₀ values express the amount of time needed for the initial concentration of a substance to be reduced by 50 %. Degradation rates were found to be strongly dependent on the compound and the soil type (Kah et al., 2007), and an increased persistence of pesticides had been observed in soils with high clay and/or OM content, and under low temperatures (Walker et al., 2012). For Norway, the characteristics of the pesticide, physiochemical properties of the soil, and climatic conditions have been identified as the main factors influencing the degradation of pesticides (VKM, 2015). This leads to site specific degradation rates and variations in fate of pesticides due to complex dynamics of physical, chemical, and biological processes such as sorption-desorption, uptake by plants, run-off, leaching, volatilization, and chemical and biological degradation.

2.3 Kinetic models for describing degradation and dissipation of pesticides

Different kinetic models are available to describe the degradation or dissipation of pesticides in laboratory and field experiments, such as single first-order kinetics (SFO) and different bi-phasic kinetics (First-Order-Multi-Compartment model (FOMC), Double First-Order in Parallel model (DFOP), and Hockey-Stick Model (HS)). Guidelines for estimating degradation kinetics and the decision process for adopting or rejecting a model are described in FOCUS (2006).

SFO kinetics is a simple exponential equation with only two parameters estimated; the total amount of chemicals at time $t=0$ (M_0) and the degradation rate constant (k). The rate of change in pesticide concentration is assumed to be directly proportional to the actual concentration in the system at any time. SFO kinetics have been preferred for deriving regulatory degradation endpoints (FOCUS, 2006). When the dissipation of a pesticide and its metabolite are modelled together, SFO kinetics are thought to be the most robust model, and in most cases describe the dissipation reasonable well (FOCUS, 2006).

However, in some cases, SFO kinetics will describe the dissipation poorly. Low availability of pesticides over time, spatial variability, and changes in microbial activity can lead to distinct phases of degradation. An initially fast degradation followed by a phase with slower degradation is referred to as a bi-phasic pattern (FOCUS, 2006), of which three different models (FOMC; DFOP, HS) are available. The FOMC model has three parameters that are determined; M_0 , the shape parameter determined by coefficient of variation of k values (α), and a location parameter (β). Dissipation is faster for larger values of α and for smaller values for β . The number of sampling dates plays a role in choosing a model, usually a minimum of 6 to 8 sampling dates should be available, but often simpler models with fewer parameters (SFO, FOMC) are preferred over models with more parameters (DFOP, HS).

The main tools for assessing the goodness of fit and for comparing different models are visual assessment and the chi-square (χ^2) test. The χ^2 test accounts for measurement error by considering the deviation between observed and predicted values. Ideally the error value at which the best-fit model passes the χ^2 -test should be below 15 % and the fit must be visually acceptable. However, this value should not be considered an absolute criteria, and in some cases the error value to pass the χ^2 -test can be larger, but the fit still represents a reasonable description of the degradation behaviour (FOCUS, 2006).

2.4 Pesticide legislation

Increasing international connections and trade of food commodities made internationally harmonized standards for pesticide residues in food important. Since 1963 the CODEX Alimentarius, a collection of international standards, practices, and guidelines related to food production and food safety has existed. So far, over 4300 MRLs have been established, covering nearly 200 pesticides (FAO/WHO, 2020). To harmonise pesticide standards worldwide, the International Code of Conduct on Pesticide Management was initiated in 1985 (FAO & WHO, 2014), aiming towards harmonising pesticide standards worldwide. The Code of Conduct on Pesticide Management includes encouragement of responsible pesticide trade, promotion of practices to reduce risk of exposure, the effective use of pesticides, promotion of integrated pest management strategies (IPM), and assistance to countries with no legislation in place, as well as a life cycle approach for the development, selling and handling of pesticides. As a result, the number of countries without pesticide legislation has decreased (Handford et al., 2015). The Code of Conduct on Pesticide Management also includes toolkits to support countries with testing of pesticides, reduction of environmental risks associated with pesticides, distribution and trade of pesticides. In 1998, the Rotterdam Convention was held, with the goal of banning hazardous chemicals in international trade such as DDT and dieldrin (Rotterdam Convention, 2010) and had so far been signed by 162 parties. In 2001, the Stockholm Convention was held to limit the use of persistent organic pollutants (POP) (Stockholm Convention, 2019). The list was expanded in 2011 and several new substances, such as endosulfan, were added.

2.4.1 Legislation in Norway, the EU, and Germany

The EU has one of the stricter pesticide regulatory systems in the world (Handford et al., 2015). In 1991, the “Council directive 91/414/EEC for placing pesticides on the market” was passed. Following that, the EU harmonized standards for pesticide registrations in 2009, where a precautionary based approach for the approval of pesticides was adopted. The approach requires scientific evidence of a pesticide being beneficial for plant production, having no unacceptable effects on the environment, and having no harmful effects on humans (EC No 1107/2009). Regulation 1107/2009 is the central regulation for pesticides in the EU.

Before a pesticide is approved for use in the EU, risk assessments must be made which include harmful effects on humans and the environment but also persistence in the environment (EC, 2009b). Criteria used are persistence, bioaccumulation, and toxicity (PBT).

If all three of these criteria are met, the substance will not be approved. If the substance meets two PBT criteria, it will be approved for use, but classified as a Candidate for Substitution (CfS). An active ingredient is considered persistent if the half-life in soil is longer than 120 days (EC, 2009b).

The process of approving an active ingredient usually takes 2.5 to 3.5 years (EC, 2020b). New substances will initially be approved for a maximum of 10 years, followed by the possibility of renewal for a maximum 15 years. After an active ingredient is approved at the EU level, plant protection products with one or more active ingredient need to be authorised in each EU country before they can be made commercially available. To make this process more efficient, the EU is divided in three different zones (EC, 2020b): the Northern Zone, of which Norway is part of, the Central Zone to which Germany belongs, and the Southern Zone. Due to differences in climate and other factors, the different zones have the possibility for appropriate evaluation of active ingredients according to their specific situation and requirements (Northern Zone, 2020). To further simplify the process of approval, an active ingredient approved in one Northern Zone country is also automatically given zonal authorisation in all Northern Zone countries, even though the climatic conditions might not be comparable (VKM, 2015). After the zonal authorisation, individual member can decide on whether the active ingredient will be authorised on a national level or not. The national laws for approving a plant protection product in Norway and Germany are “Matloven” (2003) and the “Pflanzenschutzgesetz” (2012), respectively.

Furthermore, Directive 2009/128/EC has as a goal to “achieve a sustainable use of pesticides by reducing the risks and impacts of pesticide use on human health and the environment and promoting the use of integrated pest management and of alternative approaches or techniques such as non-chemical alternatives to pesticides.”

MRLs have been established and harmonised among member states in Regulation EC No 396/2005 (EC, 2005). MRL are defined as; the upper legal levels of a pesticide residue concentration in food and feed that is thought to not pose a health risk for the consumer (EFSA, 2010). Risk assessment must be conducted to prove that the criteria for MRLs are upheld. If no MRL is defined, the regulation default of 0.01mg/kg is used. The MRLs are set to not be exceeded if good agricultural practices (GPA) are followed. GAP include; following national recommendations, safe use of plant protection products under actual conditions at any stage of production, storage, transport, distribution and processing as well as following principles of integrated pest management (IPM), and applying the minimum quantity of pesticides necessary (EC, 2005).

Reasons for MRL exceedances of approved pesticides were identified as a lack of GAP, including the use of plant protection products on crops for which no authorisation was granted, wrong application rate, inappropriate pre-harvest intervals, inappropriate number of applications, or wrong method of application (EFSA, 2016). Other reasons for exceedance of MRL can include spray drift from neighbouring fields or unfavourable weather conditions which are associated with reduced degradation (EFSA, 2010). If residue levels above the MRL are detected, the commodity cannot be traded.

2.4.2 Pesticide legislation in China

Pesticide regulations were established recently in China, compared to the EU. Stricter regulations were partly a response to import bans on food commodities from China, as well as incidences of food poisoning, resulting in the introduction of a new food safety law in 2009 (Jia & Jukes, 2013). The Ministry of Agriculture and Rural Affairs handles the registration and approval of pesticides and has used scientifically based MRLs since 2009 (Handford et al., 2015). Since 2001 China has also implemented a series of policies aimed at reducing pesticide residues in food, including the Pollution-free Food Action Plan, the Food Safety Law of the People's Republic of China, and the Action Plan for Zero Growth in Pesticide Use by 2020 (Liu et al., 2020). According to the Ministry of Agriculture, these regulations increased the percentage of food samples within legal limits from 91.4 % in 2005 to 97 % in 2017 (Liu et al., 2020). GAPs were also established and if pesticides are applied according to them, it is unlikely that they pose a threat to public health (Chen et al., 2012). However, food safety incidences continue to occur (Jia & Jukes, 2013).

Part of China's problem in meeting the standards of the EU and other countries are problems related to the enforcement of pesticide regulations, especially in rural areas. Results from a survey showed that laws regulating the types of pesticides allowed in agriculture are widely followed, but other unsafe practices like improper disposal of containers are common (Yang et al., 2014; Zhou & Jin, 2013). In addition, 59 % of farmers reported that they would commonly sell vegetables within two or three days after spraying (Yan et al., 2016). When farmers were asked about their use and selection of pesticides, Yang et al. (2014) found, that almost 50 % of farmers trusted their own experience more than government regulations. Overall, pesticide residue levels were linked to local policymaking and implementation, highlighting the important role of local governments to ensure food safety (Liu et al., 2020).

3. Material and Methods

3.1 Project description

The master thesis is part of the project “ChiNor solutions for Low Impact climate smart vegetable production with reduced pesticide residues in food, soil and water resources”, short LowImpact (NIBIO, 2019). The project started in January 2019 and is scheduled to end in June 2022. The aim of the project is to improve vegetable production in China and Norway by minimising negative effects of agriculture on the environment and improve food safety by reducing pesticide exposure by use of biochar. The hypothesis of the project is that *“Soil C and N transformation processes and pesticide exposure concentrations under current production practices pose critical challenges to environmental quality and food safety that may be alleviated through novel use of biochar applications”*. Different work packages were established to study the various aspects of the project including pesticide exposure assessment tools, biochar for improved nutrient management and reduced pesticide exposure, as well as impacts on food safety, soil and water quality. The master thesis is part of the work package “pesticide exposure assessment tools” with the aim of optimizing analytical methods for exposure assessment of pesticides and their metabolites in vegetable production systems in Norway.

3.2 Literature study

Information on approved pesticides in Norway and Germany was collected in a literature study, using information from Mattilsynet and Plantevernguiden for Norway (Mattilsynet, 2020; plantevernguiden, 2020), and information from the Bundesministerium für Verbraucherschutz und Lebensmittelsicherheit (BVL) for Germany (BVL, 2020c). Information on approved pesticide in carrots in China was obtained through personal communication of Marit Almvik with an expert at the Chinese Academy Of Agricultural Sciences (CAAS) (Almvik, 2020).

Data on pesticide residues in carrots in Norway, the EU, Germany, and China was collected. For Norway, data from NIBIO and Mattilsynet was used (Mattilsynet, 2019) and for the EU, reports on pesticide residues in food from the European Food Safety Authority (EFSA) were used. Since carrots are reported every third year, only the reports from 2008, 2011, 2014, and 2017 were considered (EFSA, 2010; EFSA, 2014; EFSA, 2016; EFSA, 2019). For Germany, information was obtained from official reports from the BVL (BVL, 2020b). For China, scientific papers on pesticide residues in vegetables from market samples were used.

3.3. Field study

3.3.1 Location

The field study was conducted between May 2019 and April 2020. All agronomic decisions, including what pesticides were sprayed and when were made by the farmer.

The sites were situated in the south-eastern part of Norway in Skien, Telemark. The climate is classified as temperate with maritime influence and moderate precipitation (Blenkinsop et al., 2008). The average temperature and precipitation is 6.4°C and 840 mm respectively. Lowest amount of precipitation occurs in February and the most precipitation in October. On average July is the warmest month and February the coldest. The averages are based on data from 1971-2000 (KSS, 2020). The greatest amount of precipitation usually occurs in winter (30 %) followed by spring (25 %).

Weather data for the period May 2019 - May 2020 was taken from LandbruksMeteorologisk Tjeneste (LMT, 2020). Data from the weather station Gjerpen and Gvarv was used since the precipitation for Gjerpen is only measured during the growing season (01.04-30.09). Temperature was always available for the station at Gjerpen.

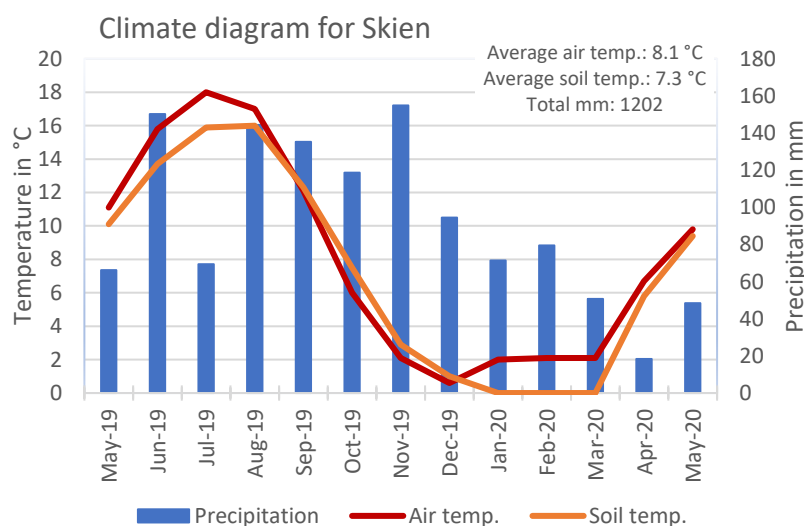


Figure 2a: Climate diagram for Skien, Norway from May 2019 - May 2020. The data is from LMT from the station Gjerpen and Gvarv. Air temperature was measured at a height of 2 m. Soil temperature was measured at a depth of 10 cm.



Figure 2b: Overview map of Norway with the red dot marking Skien. The map on the is from norgeskart.no.

The average temperature between May 2019 and April 2020 was 8.1 °C, with the highest temperature measured being 32.4 °C (26.07.19) and the lowest -9.9 °C (21.03.20). The total precipitation was 1202 mm. Both temperature and precipitation were above the average from 1972-2000.

The soil temperature at 10 cm depth reached its maximum in July and August with 16°C, and its minimum from January to March with 0 °C. The average soil temperature over the study period was 7.3 °C.

3.3.2 Soil properties

The two sampling sites are called HV and HØ and located closely together, divided only by a road with HV situated on the left site and HØ on the right site (Fig. 3). Soil analyses of the top layer (0-10 cm) was conducted by Eurofins Agro Testing Norway AS in Moss in the end of September 2019 with soil samples from May 2019.

Table 2: Soil properties (soil texture and grain size distribution (clay, silt, sand), total organic carbon (TOC), and pH) presented for the two sampling sites HV and HØ. World Reference Base (WRB) classification were taken from kilden.nibio.no.

	HV	HØ
Area	2.1 ha	3.6 ha
Soil texture	Silty loam	Silty loam
WRB classification	Umbric Epistagnic Albeluvisol (Siltic)	Epistagnic Albeluvisol (Siltic)
Clay	16 %	18 %
Sand	26 %	20 %
Silt	59 %	64 %
Total organic carbon (TOC)	2.4 %	2.2 %
pH	7.8	7.9

HV is intersected by an area (with 6-12 % inclination) containing silty loam (letteirer), but soil samples were only taken in the silty part of the field as marked in Fig. 3. From the soil analysis both sites are classified as silty loam, even though at kilden.nibio.no they were shown as being different with HV belonging to the texture group silt (Fig. 3).

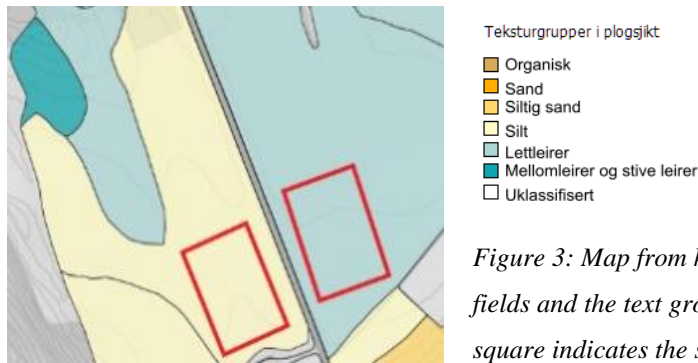


Figure 3: Map from kilden.no showing the placement of the carrot fields and the text group for HV (left) and HØ (right). The red square indicates the sampling area.

Both sites belong to the most common subunit of Albeluvisol in Norway, the Epistagnic Albeluvisols (Siltic) (VKM, 2015). They are periodically saturated with water as a result of decreasing macropores with depth and dense layers, stopping vertical transport of water. HV also has an umbric horizon. Umbric horizons are relatively thick, dark-coloured surface horizons that show a low base saturation and moderate to high contents of organic matter. It consist of mineral materials (WRB, 2015).

3.3.3 Agricultural practices

In 2019, the farm had a contract with Findus to deliver 600 t of carrots for frozen mixes and 600 t of carrots for fresh consumption. The spraying plan for the growing season was, that the first herbicides would be applied at high dosage 3-5 days after sowing, followed by a lower herbicide dosage 3-4 times after seedling emergence. The herbicides aclonifen, metribuzin, and depending on weeds also cycloxydim and clomazone are used in carrots. In mid-July, herbicide application would be finished, and fungicides would be applied in August using boscalid, pyraclostrobin, and azoxystrobin. Insecticides were not be sprayed, due to the usage of an insect net from mid-June until August. The net was removed before fungicide application, but herbicides were applied on it. The actual spraying plan varied for different reasons such as weather, weeds, and pests. Carrots are sown at the sites every 5-6 years to prevent fungal colonies from being established. Previous crops include root cabbage, potatoes, and cereals. In the following section the agricultural practices and the spraying for both fields are described in depth.

Location HV

At HV, carrots were planted on 35 daa (decare) on 29.05.19. The variety was Yello Mello industry, which was sown on raised flat beds with three rows per bed, using an Agricola carrot seeder. An insect net was laid out in June and removed in mid-August. The herbicides aclonifen and metribuzin were applied. The carrots were harvested on 26.09.19 with a share lifter (skjærløfter) after the green tops were removed by a topper (avtoppingsmaskin). After harvesting the carrots were sent to a washing facility for cleaning. The carrots were delivered to Findus.

Additional information on spraying and other agricultural operations can be found in Table 3. Information regarding crop rotation and previous pesticide application can be found in Table 4.



Picture 1: HV with raised flat beds and insect net visible in June. Pictures were taken by M. Almvik (May) and K. Lang (June, August).

Table 3: Soil sampling and spraying information for HV in 2019.

Date pesticide application	Pesticide applied	Dosage	Soil sampling date	Comment
			03.05.19	Before carrots were planted out/ sprayed
03.06.19	Aclonifen, Metribuzin (Fenix+Sencor WG 70)	70ml Fenix 3g Sencor WG 70	04.06.19	29.05: sowing 10.06: Insect net
18.06.19	Aclonifen, Metribuzin (Fenix+Sencor WG 70)	12ml Fenix 2g Sencor WG 70	18.06.19	
29.06.19	Aclonifen, Metribuzin (Fenix+Sencor WG 70)	30ml Fenix 4g Sencor WG 70	12.07.19	17.07, 03.08, 04.08: irrigation 16.08: 20kg/daa fertilizer (12-4-18)
			30.08.19	20.08: insect net removed
			05.09.19	
			26.09.19	Carrots were harvested
			25.10.19	
			16.04.20	Bare soil

Table 4: Spraying journal from 2016-2018 for HV

Year	Commercial product	Active ingredient	Dosage	Class	DT50 (days)	Crop
HV						
2016	Steward	Indoxacarb	8.5 g	I	5.97	Cauliflower
	Fastac 50	Alfacypermetrin	25 ml	I	42.6	
	Lentagran WP	Pyridate	90 g	H	2.2	
	Biscaya OD 240	Thiacloprid	40 ml	I	8.1	
	Decis Mega EW 50	Deltametrin	15 ml	I	21	
2017	2 x Karate 5 CS	Lambda-cyhalothrin	2 x 13 ml	I	26.9	Broccoli
	Sumi-Alpha	Esfenvalerate	14 ml	I	19.2	
2018	2x Decis Mega EW 50	Deltametrin	15+12 ml	I	21	Broccoli
	Conserve	Spinosad	20 ml	I		
	Steward	Indoxacarb	8.5 g	I	5.97	

Location HØ

At HØ, carrots were planted on 34.3 daa on 28.05.19. The variety was Namdal C4, which was sown in ridges with one seed row per bed. An insect net was laid out in June and removed in mid-August. The herbicides aclonifen and metribuzin were applied, and four different fungicides (boscalid, pyraclostrobin, fludioxonil and cyprodinil). The carrots at HØ were not harvested and sold.

More information on spraying and fertilizer application can be found in Table 5. Crop rotation and previous pesticide application can be found in Table 6.



Picture 2: HØ with topped seed beds and insect net visible. Pictures were taken by M. Almvik (May, July) and K. Lang (August).

Table 5: Soil sampling and spraying information for HØ in 2019.

Date pesticide application	Pesticide applied	Dosage	Sampling date	Comment
			21.05.19	Before carrots were planted out/ sprayed
03.06.19	Aclonifen, Metribuzin (Fenix+Sencor WG 70)	70ml Fenix 3g Sencor	04.06.19	28.05: sowing 10.06: insect net
18.06.19	Aclonifen, Metribuzin (Fenix+Sencor WG 70)	12ml Fenix 2g Sencor	18.06.19	
29.06.19	Aclonifen, Metribuzin (Fenix+Sencor WG 70)	30ml Fenix 4g Sencor	12.07.19	
20.08.19	Boscalid, Pyraclostrobin (Signum)	100 g Signum	30.08.19	20.08: insect net removed
03.05.19	Fludioxonil, Cyprodinil (Switch 62.5 WG)	80 g Switch	05.09.19	
			26.09.19	
			25.10.19	
			16.04.20	Bare soil

Table 6: Spraying journal from 2016-2018 for HØ.

Year	Commercial product	Active ingredient	Dosage	Class	DT50 (days)	Crop
HØ						
2016	Steward	Indoxacarb	8.5 g	I	5.97	Cauliflower
	Fastac 50	Alphacypermethrin	25 ml	I	42.6	
	Lentagran WP	Pyridate	90 g	H	2.2	
	Biscaya OD 240	Thiacloprid	40 ml	I	8.1	
	Decis Mega EW 50	Deltamethrin	15 ml	I	21	
2017	2 x Karate 5 CS	Lambda-cyhalothrin	2 x 13 ml	I	26.9	Broccoli
	Sumi-Alpha	Esfenvalerate	14 ml	I	19.2	
2018	Sumi-Alpha	Esfenvalerate	12 ml	I	19.2	Cauliflower
	Lentagran WP	Pyridate	100 ml	H	2.2	
	Conserve	Spinosad	20 ml	I		
	Decis Mega EW 50	Deltamethrin	12 ml	I	21	
	Steward	Indoxacarb	8.5 g	I	5.97	

3.3.4 Soil sampling

Soil samples were taken from selected sites in the fields marked with a red rectangle in Fig 3. For HV that area included 18 flat three-rowed seedbeds (Fig. 4) and for HØ 32 topped single seedbeds (Fig. 5). The difference in number of seedbeds was due to wider seedbeds at HV compared to HØ. The topsoil samples (0-10 cm) were taken using a grass plot sampler. 10 samples were sampled randomly across the selected site and mixed in a plastic bag. This was repeated 2 times, resulting in 3 mixed samples from each site per sampling date were obtained. The soil was stored in a freezer at -20 °C at NIBIO.

Soil samples were only taken from the top of the beds and not from in between the rows. Soil samples were taken on nine dates throughout the year (Table 3 + 5). The first samples were collected in May 2019 and the last samples in April 2020. The sampling period covered in total 350 days at HV and 332 days at HØ.

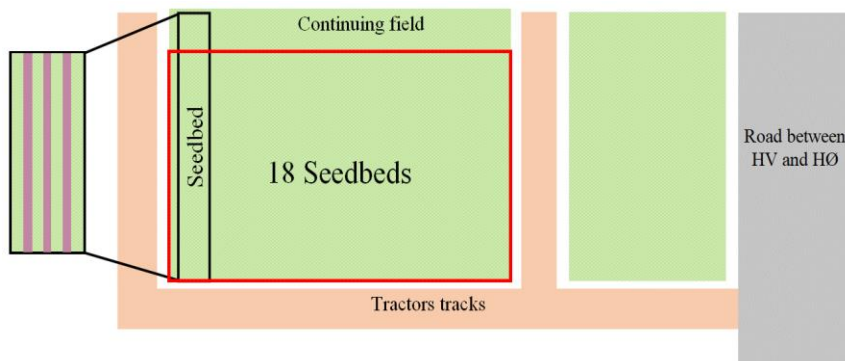


Figure 4: Schematic drawing of HV. The sampling site is marked with red. The sampling area covered 18 seedbeds with each of them having three rows of carrots on top (purple).

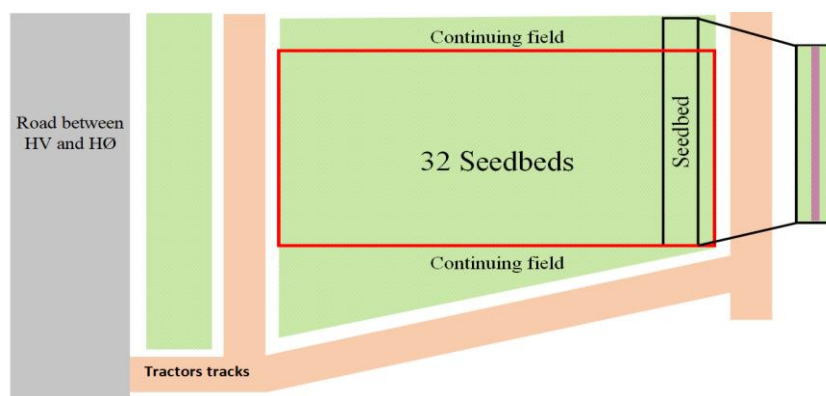


Figure 5: Schematic drawing of HØ. The sampling site is marked with red. The sampling area covered 32 seedbeds with each seedbed having one row of carrots on top (purple).

3.4 Analysis of pesticides in carrots

20 carrot samples were taken from HV on the day they were harvested (26.09.19). The green leaf tops were removed before the carrots were taken up from the soil. Carrot samples were randomly taken from the harvested carrots. HØ was not harvested and therefore no carrot samples were taken from HØ.

The carrots were stored in a cooling room at 2-4 °C at NIBIO and extracted and analysed on the 30.09.19. QuEChERS (Anastassiades et al., 2003) was used for extracting pesticides from the carrot samples.

The carrots were cut in half and one side was analysed with the peel on, the other half was analysed with the peel removed. The samples were homogenised with a blender and then extracted with acetonitrile. Citrate buffer (Supel™ QuE 55227-U tube) was added for improved transfer of the pesticides into the acetonitrile solution. The methods used for determining pesticide concentrations in carrots were NIBIO method M86 (238 pesticides using Agilent LC-MS/MS) and NIBIO method M93 (108 pesticides using Agilent GC-MS/MS), both accredited (ISO 17025) quantitative methods at NIBIO. Karin Prestmoen, NIBIO performed the extraction, and the analysis was performed by Nina O. Svendsen, NIBIO. The Limit of Quantification (LOQ) was 0.01 mg/kg.

3.5 Extraction of pesticides from soil

3.5.1 Recovery study in soil

Before the analysis of the soil samples, different extraction methods were tested to optimize the extraction and clean-up of pesticides in the soil. Extractions with acetone, acidified acetone, and acetonitrile were tested, as well as solid phase extraction (SPE), which is also used in determination of pesticide residues in soils (Andreu & Picó, 2004). Properties of sorbents used for the SPE are presented in Table 7.

Table 7: Properties of different sorbents used for method testing for solid phase extractions with soils from May 2019 from HV.

Sorbent	Polymer type	Surface area (m ² /g)	Pore size (nm)	Particle size (µm)
Lichrolut EN (ENV)	PS-DVB	1200	3	40-120
Oasis HLB	PVP-DVB (polyvinylpyrrolidone)	830	8	30-100
Strata X	Pyrrolidone PS-DVB	800	8.5	33
Strata X-AW (weak anion mixed mode)	Pyrrolidone skeleton with ethylene diamine groups	800	8.5	33
Strata X-CW (weak cation mixed mode)	Pyrrolidone skeleton with carboxylic acid groups	800	8.5	33

3.5.2 Final extraction

In the end it was decided to use a modified version of the original QuEChERS method by Anastassiades et al. (2003), using acetonitrile.

10 g of soil were weight in 50 ml centrifuge tubes. Soil from HV (May 2019) was used for the blank and for the control sample. The control sample was spiked with 166.67 µl of a pesticide standard mix (level 3000 ng/ml) which was equal to 25 ng/ml in the final extract. All the samples had 20 µl of internal standard (IS) (level 50 µg/ml) were added to achieve 50 ng IS/ml in the final extract. 10 ml acetonitrile was added to the samples, which were then extracted for 20 min on a horizontal shaker. After that a citrate buffer (Supel™ QuE 55227-U) was added for improved transfer of pesticides into the acetonitrile solution. The samples were extracted for an additional 10 min on the horizontal shaker and then centrifuged at 3000 rpm for 5 minutes. This was followed by a clean-up step where 1.5 ml of the extract was taken up with a syringe and 0.7 ml extract was pushed through an attached Oasis HLB Plus Light filter into a waste bottle. Afterwards a VWR PTFE 0.2 µm syringe filter was placed on top of the Oasis filter and the remaining extract was pushed into a vial. The extracts were analysed on LC-QExactive, and quantified with internal standard batch calibration in the range 0.4-300 ng/ml, using deuterium-labelled pyraclostrobin (D₃) at a level of 50 ng/ml as internal standard. Pesticide concentrations were calculated as mg pesticide/kg dry soil. The method used for determining pesticide concentrations was M119 (350 pesticides and metabolites using Thermo Scientific LC-Q-orbitrap (model QExactive)) in the positive electrospray ionization mode. The pesticide aclonifen is not included in the analysis method. The analysis was performed by Marit Almvik, NIBIO. The LOQ was 0.1-1.0 µg/kg fresh soil, depending on the compound.

3.6 Modelling of DT50 values in soil

The dissipation rates of pesticides detected in the soils of HV and HØ were modelled with Tessella Computer Assisted Kinetic Evaluation (CAKE) 3.4 software (Tessella Technology and Consulting, 2020).

For the calculation of the dissipation rate, the date after the last application of a pesticide was chosen as the starting time ($t=0$). This resulted in fewer dates being used in the modelling of some pesticides, and therefore simpler models (SFO, FOMC) were preferred.

As an initial step, SFO kinetics were fit to the field data, using the individual replicates for each date.

$$M = M_0 e^{-kt}$$

Formula 1: Equation for SFO kinetics as described in FOCUS (2006).

M = total amount of chemical present at time t

M₀ = total amount of chemical present at time t=0

k = rate constant

To analyse the goodness of fit, visual assessments and chi-square (χ^2) test were used. Ideally the best-fit model should have an error value below 15 % and the fit must be visually acceptable (FOCUS, 2006).

If the SFO model was not accepted, FOMC kinetics were fit to the field data. The FOMC model has three parameters that are determined; M₀, the shape parameter determined by coefficient of variation of k values (α) and a location parameter (β). Dissipation is faster for larger values of α and for smaller values for β .

$$M = \frac{M_0}{\left(\frac{t}{\beta} + 1\right)^\alpha}$$

Formula 2: Equation for FOMC kinetics as described in FOCUS (2006).

M = total amount of chemical present at time t

M₀ = total amount of chemical applied at time t = 0

α = shape parameter determined by coefficient of variation of k values

β = location parameter

When modelling degradation and dissipation, sinks for pesticides and their metabolite must be considered (Fig. 6). Sinks account for processes where components are lost, such as photolysis on the soil surface, loss through volatilisation and/or leaching to deeper soil layers. In field studies, loss of mass balance can occur due to lack of accounting for volatiles or bound residues and sinks should in those cases not be included in the fit (FOCUS, 2006). In the initial fit, all flows to sink compartments were included but removed later if necessary, for example when mass balance was incorrect.

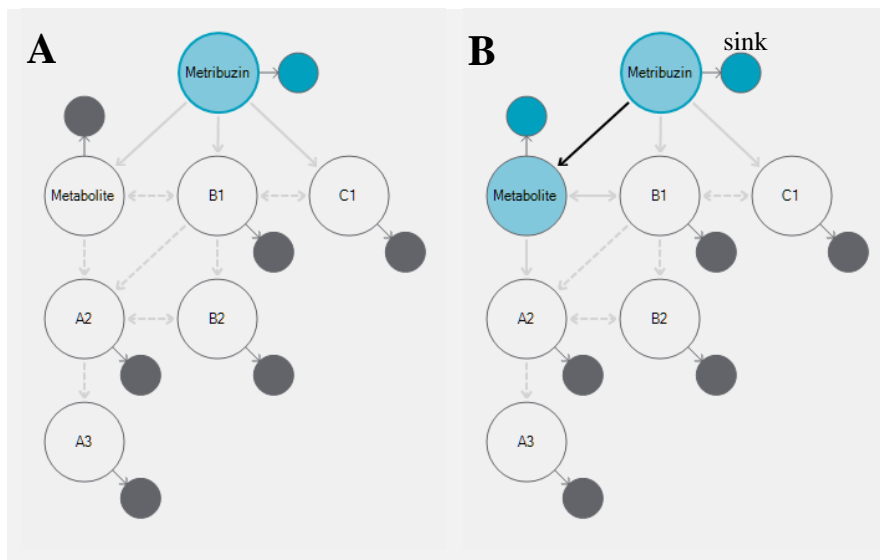


Figure 6: Topography of the CAKE software for modelling dissipation of pesticides. (A) is an example of pesticides where no metabolite was detected and (B) is an example for pesticides with a metabolite. Screenshot from the software CAKE 3.4.

If the fit was still not satisfactory, outliers were removed e.g. datapoints that clearly deviate from earlier or later samples, which can occur due to sampling error or natural variability in the soil. In the initial fitting process these outliers were included but some were eliminated later (Table 8). The outliers were visually identified showing clearly higher concentrations than the rest of the measurements on that date.

Table 8: Properties of data points that were removed are shown with date and value.

Location	Pesticide	Date	Measurments	Value in [$\mu\text{g}/\text{kg}$]
HØ	Fludioxonil	25.10.19	3	225.875
	Imidacloprid	04.06.19	3	19.247
	Pyridafol	05.09.19	2	3.134

To demonstrate the decision-making process for adopting or rejecting a model and the removal of outliers, the fitting process for fludioxonil is given as an example. The SFO and FOMC model initially gave an ok fit, but the χ^2 values were not $< 15\%$ as preferred (Fig. 7A + B, Table 9). One data point on day 51 was visually identified as an outlier and removed. Afterwards the fit improved and the χ^2 value was $< 15\%$, for both models but the SFO model was preferred, and therefore used.

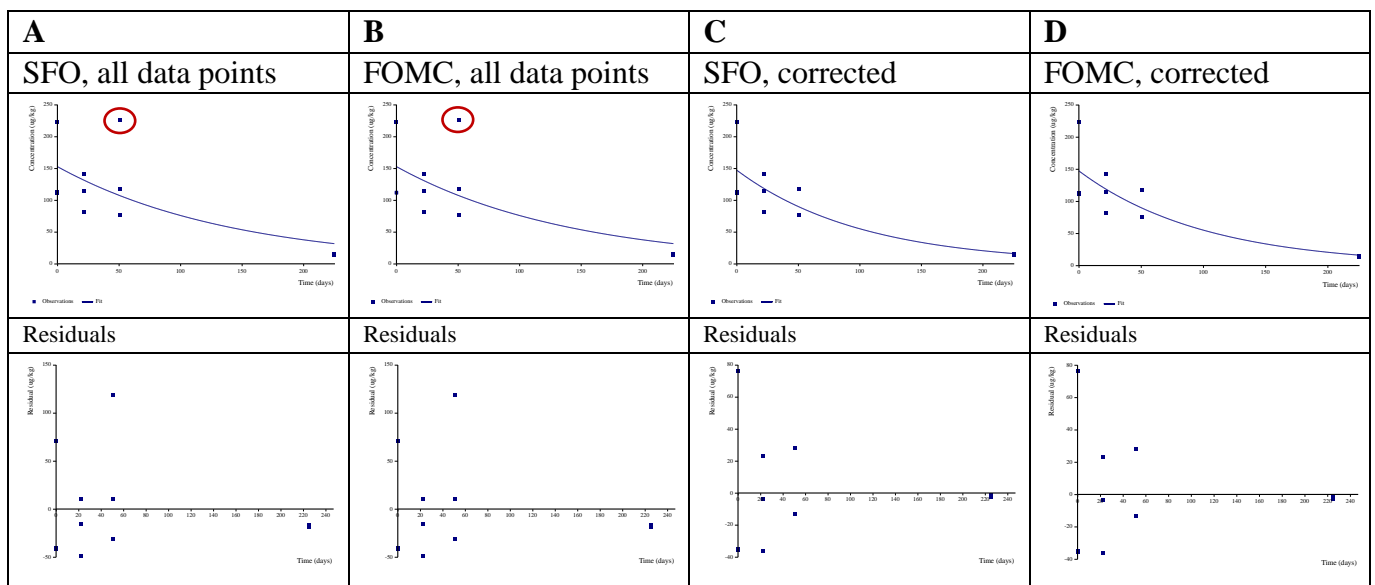


Figure 7: SFO and FOMC models with residues for fludioxonil measured at HØ. (A) shows the SFO model with all measurements from HØ after it had been sprayed on the 03.05.19, (B) showing the same as (A) but for the FOMC model. (C) shows the SFO model for the same data but with one out of three measurements removed at day 51, and (D) shows the FOMC model with the removed data point.

Table 9: Information and statistics on models for fludioxonil with optimized values (modelled M_0 =start concentration, k = degradation rate constant), standard error (σ) and goodness of fit model variables: r^2 (square of Pearson's correlation coefficient), χ^2 , RSS (residual sum of squares). DT_{50} and DT_{90} values are also shown.

Model	Variable	Optimized value	σ	r^2	χ^2	RSS	DT_x (days)		
A	SFO	M_0	152.8	23.91	0.5216	16.3	27270	DT_{50}	99.2
		k	0.006987	0.003818				DT_{90}	330
B	FOMC	M_0	152.8	24.27	0.5214	20.4	27280	DT_{50}	99.1
		α	278.1	183.3				DT_{90}	330
		β	39700	17400					
C	SFO	M_0	147.2	17.53	0.7226	4.27	11190	DT_{50}	70.5
		k	0.00983	0.004164				DT_{90}	234
D	FOMC	M_0	147.3	18.49	0.7226	5.35	11190	DT_{50}	70.5
		α	368	nd				DT_{90}	235
		β	37400	nd					

4. Results

4.1 Literature study

4.1.1 Pesticides approved in carrots

The number of pesticides that were officially approved in carrots varied greatly between Norway, Germany, and China. For carrots, 16 different active ingredients were approved in Norway, 43 were approved in Germany, and nine were approved in China. Interestingly, the main class of pesticides approved in carrots in Norway and Germany were fungicides with 44 % and 35 % respectively, followed by herbicides and insecticides. In China, no fungicides were officially approved in carrots, with insecticide being the dominating class (Fig. 8).

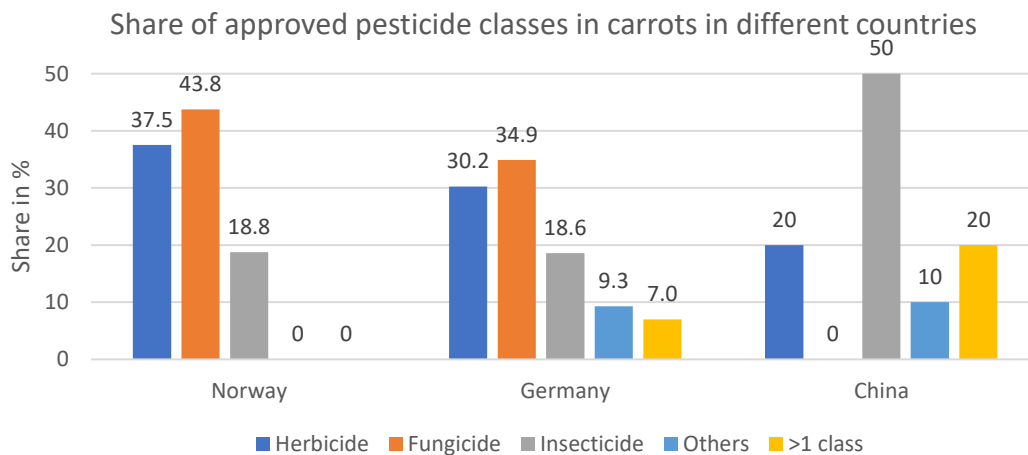


Figure 8: Distribution of classes among approved pesticides in Norway, Germany, and China. Herbicides, fungicides, and insecticides were the main classes. Other classes (others) include rodenticides and molluscicides. Some pesticides belonged to more than one class (>1 class). Information is from Mattilsynet (2012) and *Plantevernguiden.no* (Norway), *BLV* (Germany), and from personal communication ((Almvik, 2020), China).

All active ingredients approved for carrots in Norway were also approved in the EU. In Germany, three out of the 16 active ingredients approved in Norway were not approved for use in carrots (clethodim, deltamethrin, metalaxyl).

Table 10: Pesticides that were approved in 2019 in carrot production in Norway sorted by class (herbicides, fungicides, insecticides). Information about approval is from *plantevernguiden.no* (2020) and *Mattilsynet* (2019).

Herbicides	Fungicides	Insecticides
Aclonifen	Azoxystrobin	Deltamethrin
Clethodim	<i>Bacillus amyloliquefaciens</i> (former subtilis) QST 713	Lambda-cyhalothrin
Clomazone	Boscalid	Spirotetramat
Cycloxydim	Cyprodinil	
Metribuzin	Fludioxonil	
Propaquizafop	Metalaxyl-M	
	Pyraclostrobin	

Other approved classes included molluscicides (MO) and rodenticides (RO), none of which were approved in Norway. Germany and China also had some pesticides that did belong to more than one class. Dazomet for example, is classified as a fungicide, herbicide, and nematicide, and is approved in Germany.

Noticeably more pesticides were approved for use in carrots in Germany compared to Norway and China. One of them was not approved at the EU level (quizalofop-P). Among approved active ingredients were biopesticides namely *Bacillus amyloliquefaciens*, *Bacillus thuringiensis* as well as the fungi *Clonostachys rosea* and the fungal parasite *Coniothyrium minitans*.

Table 11: Pesticides approved in carrots in Germany, sorted by herbicides, fungicides, insecticides, others which includes repellents (RE), rodenticides (RO) and molluscicide (MO). Pesticides that belong to more than one class (>1 class) include nematicides (N). Information on approval was from the BVL (2020c).

Herbicides	Fungicides	Insecticides	Others	>1 class
Aclonifen	Azoxystrobin	Alpha cypermethrin	Calcium carbide (RE)	Dazomet (F, H, N)
Clomazone	<i>Bacillus amyloliquefaciens</i> (former subtilis) QST 713	<i>Bacillus thuringiensis</i> subspecies aizawai GC-91	Calcium phosphide (RO)	
Cycloxydim	Benzoic acid	Cyantraniliprole	Metaldehyde (MO)	
Fluazifop-P	Boscalid	Lambda-cyhalothrin	Zinc phosphide (RO)	
Glyphosate	<i>Clonostachys rosea</i> strain J1446	Pirimicarb		
Haloxyfop-P	<i>Coniothyrium minitans</i> Strain CON/M/91-08	Spirotetramat		
Metobromuron	Cyprodinil	Tau-fluvalinate		
Metribuzin	Difenoconazole			
Pendimethalin	Dimethomorph			
Phenmedipham	Fludioxonil			
Propaquizafop	Fluopyram			
Quizalofop-P	Fluxapyroxad			
	Isopyrazam			
	Mancozeb			
	Metiram			
	Pyraclostrobin			
	Quizalofop-P-ethyl			
	Tebuconazole			
	Trifloxystrobin			

Compared to Norway and Germany, China had very few pesticides approved for use in carrots. Three of them were banned in the EU (phoxim, chlorfluazuron, choline chloride). None of the pesticides were approved for use in carrots in Norway, however four were approved for other crops (imidacloprid, fenoxaprop-P-ethyl, acetamiprid, abamectin). In Germany, two were approved for use in carrots (*Bacillus thuringiensis*, pendimethalin) and three for use in other crops (abamectin, acetamiprid, imidacloprid).

Table 12: Pesticides approved in carrots in China according to their class: herbicide, insecticide, others including rodenticides (RO), and pesticides belonging to more than one class, including acaricide (A). Information on approval is from personal conversation with an expert at CAAS (Almvik, 2020). Information on class was form the PPDB(2007).

Herbicides	Insecticides	Others	>1 class
Fenoxaprop-P-ethyl	Acetamiprid	Choline chloride (RO)	Abamectin/Avermectin (I, A)
Pendimethalin	<i>Bacillus thuringiensis</i> subspecies aizawai GC-91		Pyridaben (I, A)
	Chlorfluazuron		
	Imidacloprid		
	Phoxim		

4.1.2 Pesticide residues in carrots

Pesticide residues in food commodities are closely monitored in Norway, the EU, and Germany. In the following section results for pesticide residues in carrots from national monitoring programs as well as the EU monitoring program will be presented. For China, general findings on pesticide residues in vegetables are reported.

Pesticide residues in carrots analysed in Norway

Of carrots analyses in Norway, 50 % showed no detectable residues between 2008 and 2018. Among the analysed carrots, the number of samples without residues was on average higher for imported carrots compared to carrots grown in Norway (Fig. 9C). A general trend towards an increased number of carrot samples with pesticide residues were observed over the years. In 2008, 37 % of Norwegian carrots and 14.3 % of imported carrots had residues. In 2018, the number of samples with residues increased to 59.1 % of Norwegian carrots and to 50 % of imported carrots.

Exceedances of MRLs were very rare. From 2008 to 2018, there were two cases of pesticide residues in carrots that were above the MRL. The first incident was in 2010 with methamidophos in carrots from Israel showing a concentration of 0.04 mg/kg (MRL: 0.01 mg/kg). The other incident occurred in 2015 in Norwegian carrots with diazinon showing a measured concentration of 0.021 mg/kg (MRL: 0.01 mg/kg).

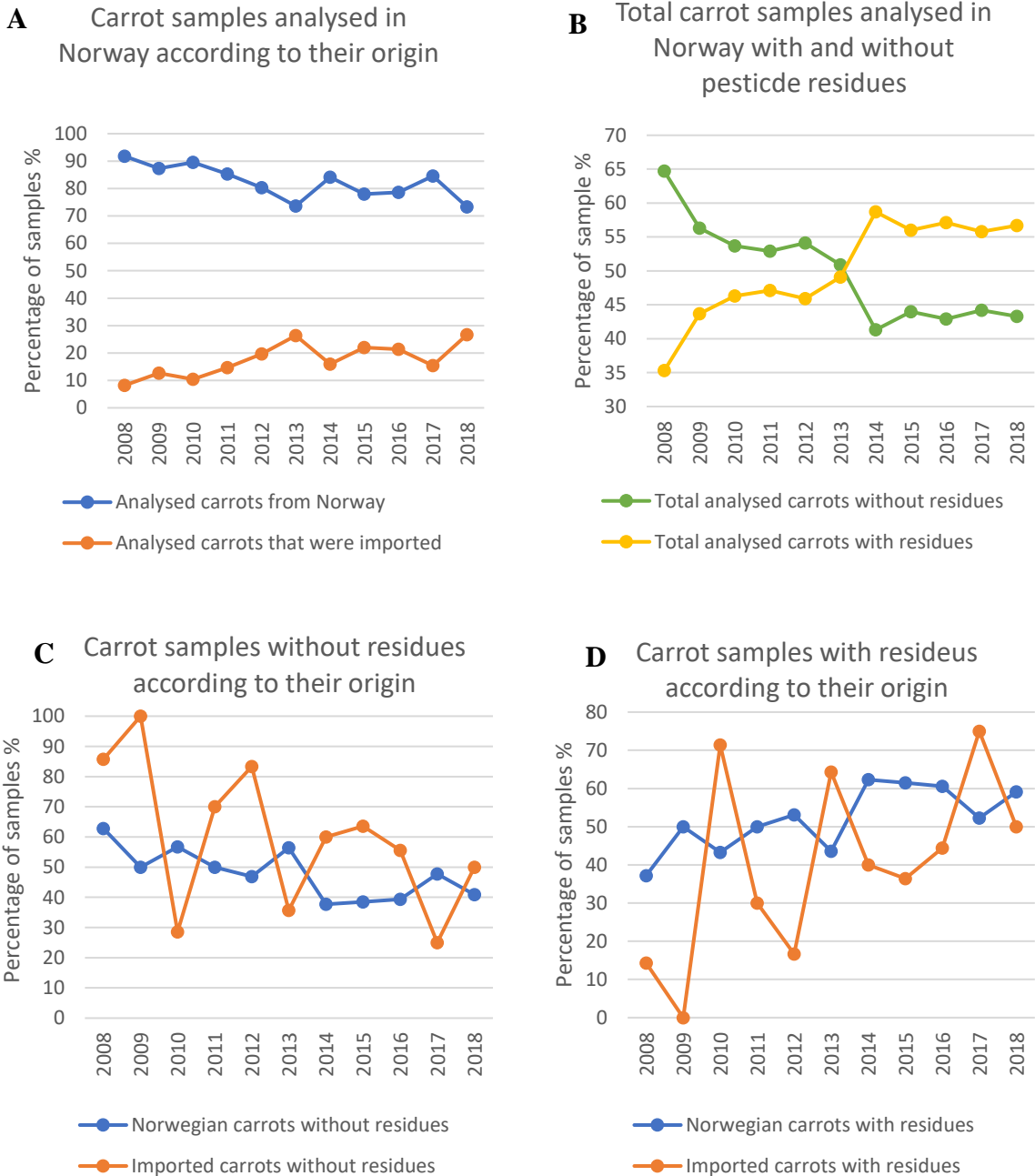


Figure 9: Development of pesticides residues in carrots analysed in Norway from 2008-2018, with (A) carrot samples according to their origin, (B) total sample with and without residues, (C) carrot samples without residues according to their origin and (D) samples with residues according to their origin (Mattilsynet, 2019).

Most samples were carrots grown in Norway, with around 16 % of samples per year being imported carrots (Fig. 9A). Imported carrots were mainly from the EU, but also from countries outside of the EU, specifically Israel, the US and Kenya. Within the EU, imported carrots were mainly from Denmark, the Netherlands, Italy, Portugal and Spain.

Residues of different pesticides were detected every year. In the period investigated, 21 different pesticides were detected in carrots, with on average 7.7 substances per year. Mostly fungicides were detected (57.1 %), followed by insecticides (28.6 %) and herbicides (14.3 %). Of those 21 pesticides, 10 (47.6 %) were not approved in Norway. Pesticides not approved include chlorpyrifos, DDT, diazinon, ethirimol, linuron, metalaxyl, methamidophos, oxamyl, pendimethalin and triadimefon and triadimenol.

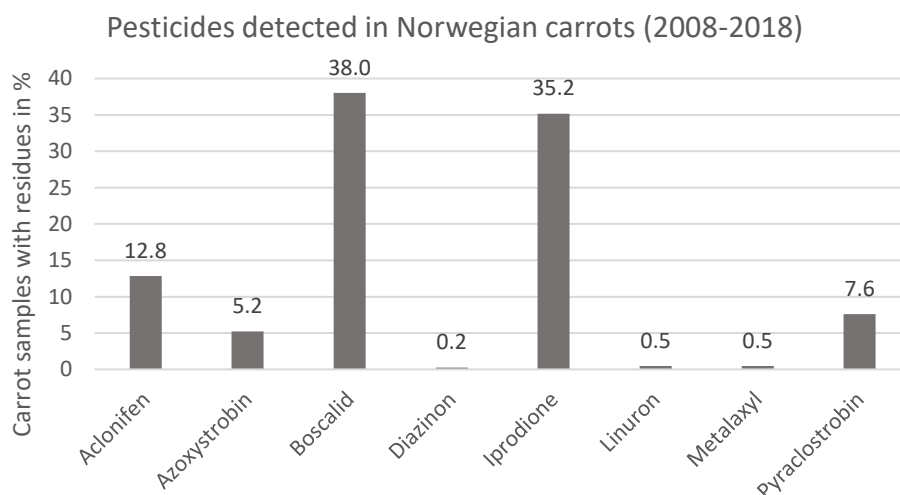


Figure 10: Pesticides detected in carrots from Norway from 2008-2018 (Mattilsynet, 2019).

Looking at pesticides present in carrots grown in Norway, eight different pesticides were detected over the whole period. Boscalid, followed by iprodione and aclonifen were the pesticides most frequently detected (Fig. 10). Most pesticides detected in Norwegian carrots are approved for use in carrots (Table 10).

Imported carrots, had considerably more detected pesticides, in total 20. The most detected pesticides were boscalid, linuron and iprodione (Fig. 11). Iprodione appeared to be more common in carrots from Norway, while linuron was frequently detected in imported carrots, but only occasional in carrots from Norway.

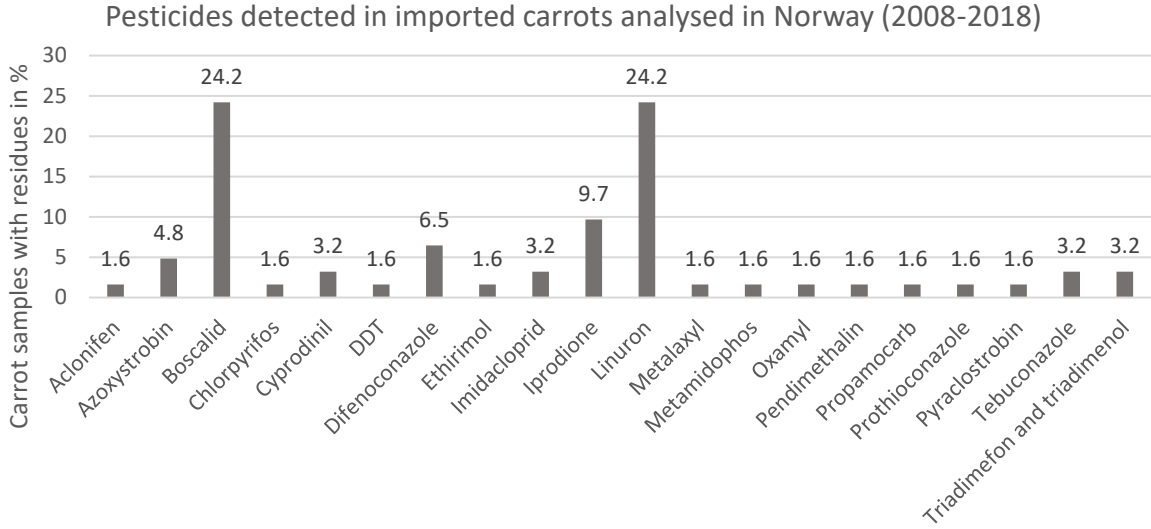


Figure 11: Pesticides detected in imported carrots analysed in Norway from 2008-2018 (Mattilsynet, 2019).

Looking at the change in pesticide residues of carrots analysed in Norway, boscalid was the most detected substance in most of the years, followed by iprodione. The rest of the remaining pesticides varies from year to year and no clear trend could be observed (Fig. 12).

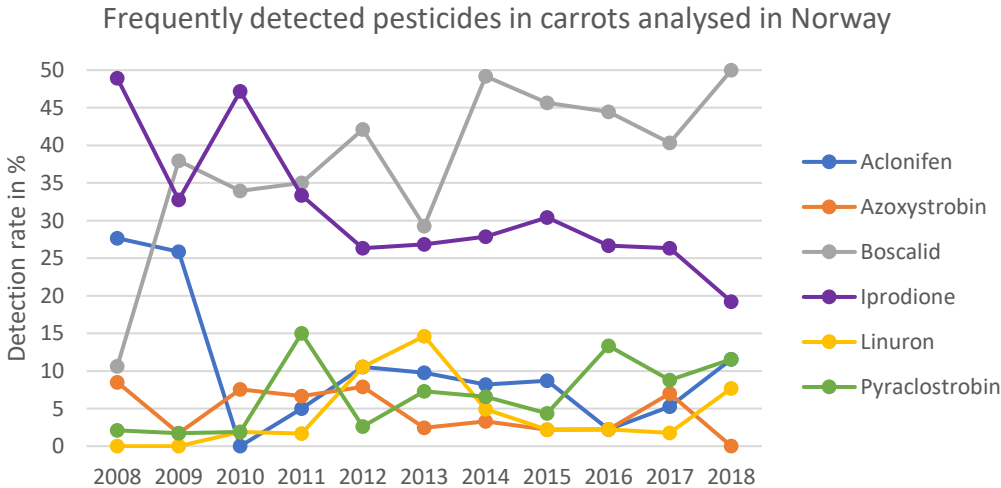


Figure 12: Frequently detected pesticides in carrots analysed in Norway (Norwegian and imported) from 2008 to 2018. (Mattilsynet, 2019)

A steady decline in the detection rate of iprodione is visible with the lowest point occurring in 2018. This was probably connected to the expiration of the approval of iprodione on 05.08.2018 in Norway. For linuron the approval in the EU expired on 31.07.2017, yet the detection rate appeared to increase slightly from 2017 to 2018.

Iprodione and boscalid were the most common combination of cooccurring pesticides. Multiple residues occurred in about 25 % of samples with five pesticide residues being the highest number, observed in a single sample. Most samples with multiple residues were from Norway, which could be related to the influence of temperature on degradation rates. It could also be a result of fewer pesticides being approved in Norway, which might lead to higher application rates of single pesticides compared to the application of more pesticides in lower concentrations. The highest detected concentrations were from Norwegian carrots with 0.77 mg/kg in boscalid. Each year the highest concentrations of residues in Norwegian carrots were reported for iprodione or boscalid ranging from 0.083-0.77 mg/kg.

Pesticide residues in carrots in the EU

In the period 2008-2017, on average 65 % of carrot samples in the EU were pesticide free, while 34 % showed residues and 1.9 % had residues over the MRL (Fig.13). In 2008, a higher percentage of carrot samples had no residues compared to the following years. From 2011-2017, few changes in samples with and without residues was observed.

The overall MRL exceedance rate for samples originating from countries outside of the EU was four times higher than for products in the EU, with many of the MRL exceedances being related to pesticides no longer approved in the EU (EFSA, 2014). Non-approved pesticides in carrots, showing residues over the MRL were mainly endosulfan, diazinon, procymidone and dieldrin. Other pesticides with frequent exceedances of MRL in carrots included chlorpyrifos, iprodione, fipronil, linuron and dimethomorph.

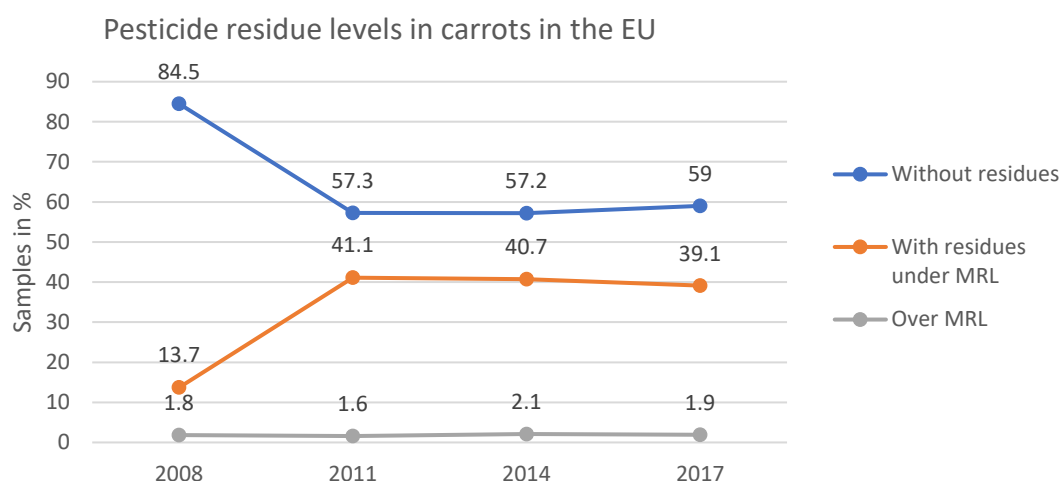


Figure 13: Residues of carrot samples in the EU as reported in the EFSA reports from 2008, 2011, 2014 and 2017. Values for carrots are reported every third year.

Commonly detected pesticides in carrots included boscalid, azoxystrobin and linuron (Table 13). In 2011, bromide ions were detected in over 50 % of the samples, which can occur naturally or as a metabolite of methyl bromide which has not been approved in the EU since 2009. Of frequently reported pesticides, all but linuron were fungicides.

Multiple residues were reported in 16.2 % of carrot samples in 2014, with two cooccurring pesticides being the most common (10.2 %). Similar numbers were reported for 2017, with 18.4 % of carrot samples having multiple residues and two being the most frequent number of cooccurring pesticides (10 %). For the other years no information was available.

Table 13: Most reported pesticides in the EFSA reports in carrot samples in 2008, 2011, 2014 and 2017.

	2008	2011	2014	2017
Common pesticides in carrot samples	Tebuconazole	Bromide ions (55.7 %)	Boscalid (23 %)	Boscalid (25.5 %)
	Iprodione	Boscalid (18.9 %)	Linuron (7.7%)	Azoxystrobin (9.3 %)
	Azoxystrobin	Linuron (14.4 %)	Azoxystrobin (7.6 %)	Difenoconazole (7.5 %)
			Difenoconazole (6.9 %)	Linuron (6.2 %)
				Tebuconazole (5.5 %)

Compared to the average for vegetables, carrots had a higher percentage without residues in 2008 and 2017 (Fig. 14). For 2008, fruits and vegetables were reported together which might explain the higher number of samples with residues compared to carrots. Vegetables had on average more samples exceeding the MRL compared to carrots. Particularly green leafy vegetables were reported to have higher rates of MRL exceedances and a higher percentage of samples with residues. Yet, carrots were among the commodities with the most frequent exceedances of MRLs, together with oranges, pears, rice, beans with pods and spinach. All together the short-term exposure to pesticide residues was found to be neglectable and thought to not pose any harm to consumers in the EU in the years reported.

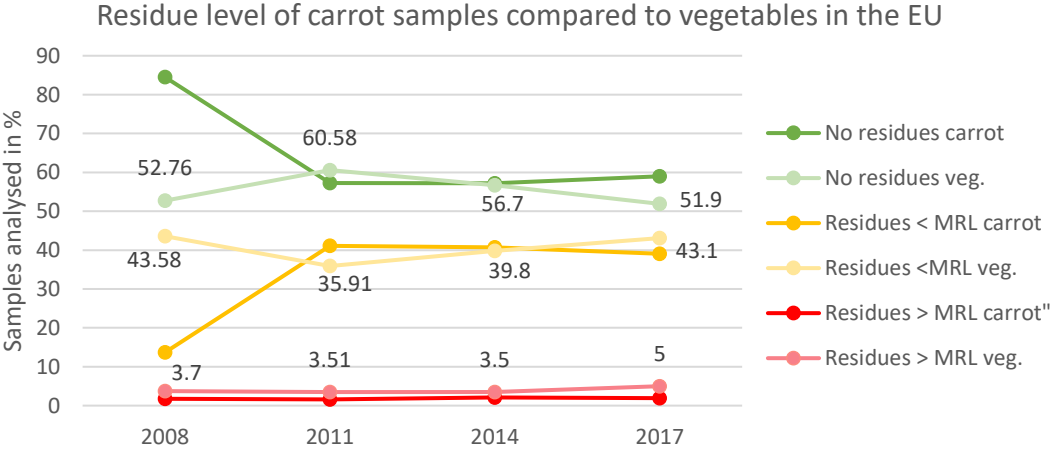


Figure 14: Comparison of the average of pesticides residues in vegetables with carrots in the EFSA reports in 2008, 2011, 2014 and 2017. The numbers in the graph are the percentage for vegetables. The exact numbers for the average for carrots can be found in Fig. 13. For 2008, fruits and vegetables were reported together, but in all other years it was only vegetables. The average numbers for vegetables were 55.5 % (no residues), 40.6 % (<MRL), 3.9 % (>MRL) (EFSA, 2010; EFSA, 2014; EFSA, 2016; EFSA, 2019).

Pesticide residues in carrots analysed in Germany

In Germany, 51.1 % of samples analysed from 2013–2018 had no residues. Imported carrots a higher average percentage of samples without pesticide residues (59.3 %) compared to samples with origin in Germany (46.7 %). The average of samples exceeding the MRL showed little difference between imported carrots (0.2 %) and carrots from Germany (0.23 %).

Most imported samples had their origin in the EU but also Israel, the US, and South Africa. Within the EU most imported carrots came from the Netherlands, Italy, and Spain.

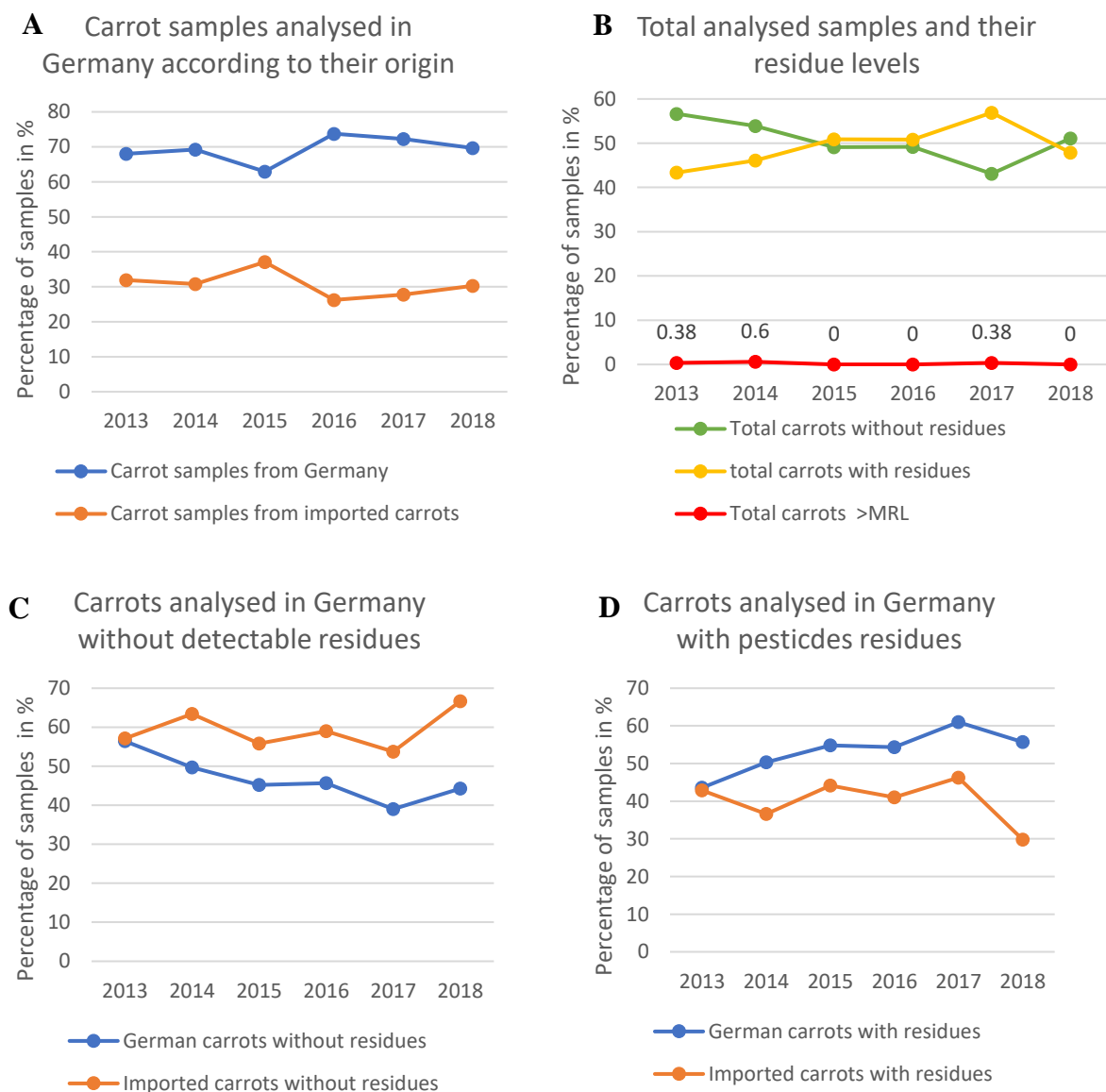


Figure 15: Carrot samples analysed in Germany (A) according to their origin, (B) total residue levels, (C) samples without residues and (D) samples with residues according to their origin (BVL, 2020b).

The number of samples without residues decreased steadily from 2013 to 2017 (Fig. 15C), then increased again in 2018. The same trend was observed for carrots from Germany and imported carrots. Yet, carrot samples from Germany always had a lower percentage of samples without residues compared to imported carrots and consequently always a higher percentage of samples with residues (Fig. 15D).

Exceedances of MRLs occurred in a low percentage of samples and were reported for chlorate, triticonazole, chlorpyrifos, dimethoate, fosetyl and benzalkonium chloride. Benzalkonium chloride was the only pesticide above the MRL without approval in the EU or Germany. Chlorate was not counted as a pesticide since it can have other origins as well. However, the exceedance of the MRL by chlorate is a known problem in the EU and in 2015 EFSA published a scientific opinion on risks for public health related to the presence of chlorate in food (EFSA, 2015). It was concluded that chlorate can lead to long term effects and especially foetuses, neonates, and individuals with low iodine uptake are likely to have effects due to its properties as a competitive inhibitor of iodine uptake.

In total, 96 different pesticides were detected from 2013-2018, with a yearly average of 42.5 different pesticides. In 2016, the highest number of different pesticides (53) were detected and in 2015 the lowest number (36). Of all detected substances, 42 % were fungicide, 22 % herbicides, 24 % insecticides, and around 15 % were metabolites. The full list of detected substances can be found in the Appendix.

The most detected pesticides were boscalid, aclonifen and difenoconazole (Fig. 16). It should also be pointed out, that copper ions were frequently detected in carrots (8.8 %), which can have sources other than pesticides. On average 23 % of carrot samples had multiple residues with two being the most common number of cooccurring residues. In most years a few samples showed ten or more cooccurring pesticides (0.1-0.9 %).

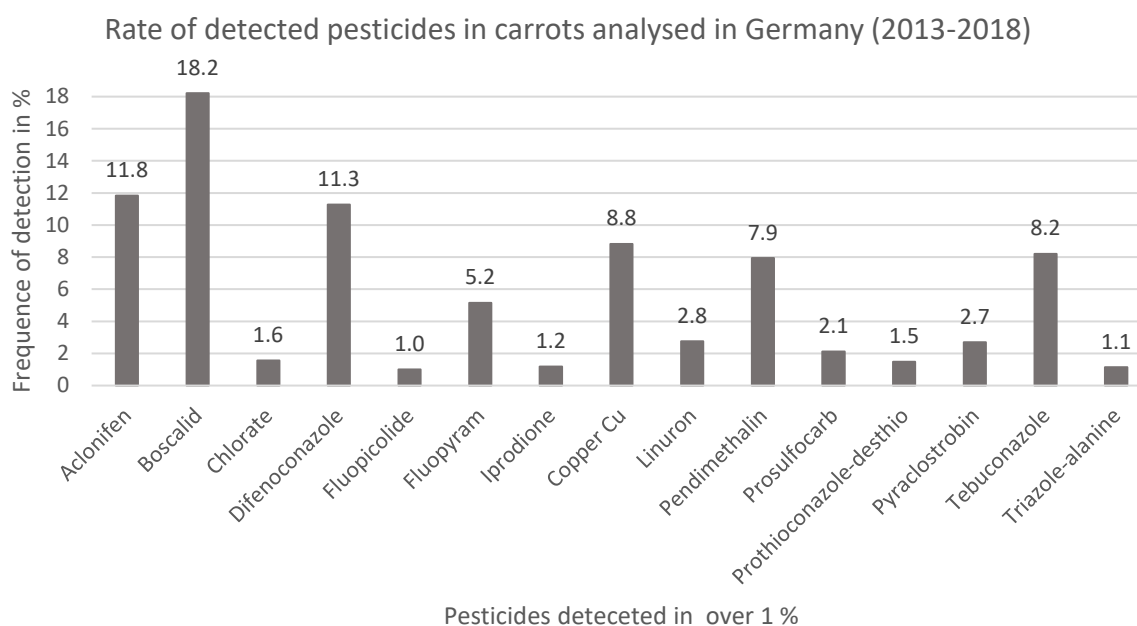


Figure 16: Pesticides detected in carrots in Germany in over 1 % of samples from 2013-2018 (BVL, 2020b).

Looking at all the detected substances in carrots analysed in Germany, each year several non-approved pesticides were detected. The pesticides found were often banned or lost their approval before 2010. DDT, dieldrin, fluazifop, quizalofop, triadimefon and triadimenol which were all detected in low quantities in at least three of the years.

Pesticide residues in vegetables in China

No official data is available for pesticide residues in carrots. One study, reported results of 846 carrot samples with 14.3 % showing residues and 0.24 % having residues over the MRL (Wu et al., 2017). However, several studies have reported on pesticide residues in vegetables sampled from markets in different regions in China (Chen et al., 2011; Liu et al., 2020; Qin et al., 2015; Wang et al., 2013; Wu et al., 2017), presented in the table below.

Table 14: Articles reporting on pesticide residues in vegetables in market samples from China.

	Chen et al., 2011	Liu et al., 2020	Qin et al., 2015	Wang et al., 2013	Wu et al., 2017
Samples	Veg. + fruits	Veg. + fruits	Veg.	Veg.	Veg.
Year(s)	2006-2009	2013-2014	2010-2013	2010	2010-2014
Region	Southeast	Different	West	Northwest	Northwest
Without residues	62.3 %	29.4 %	69.76 %	71.94 %	64.15 %
With residues	26 %	67.7 %	25.59 %	22.1 %	31.97 %
Residues over MRL	11.7 %	2.9 %	4.94 %	5.96 %	3.88 %
Common no. cooccurring pesticides	2	2	2	2	2
Dominant class	I	I	I	I	F
No. pesticides analysed	22	733	21	33	48

Apart from Liu et al. (2020), over 60 % of vegetable samples were without detectable residues. The exceedances of MRLs in vegetables from China, ranged from 2.9 % - 11.7 %. In general green, leafy vegetables had higher residue levels (Wang et al., 2013; Wu et al., 2017).

In most of the studies, insecticides were the most detected class, but often vegetable samples were only screened for commonly used pesticides, which were mainly insecticides. Pesticides detected in three or more of the publications mentioned include acephate, chlorpyrifos, omethoate, phorate (Chen et al., 2011; Qin et al., 2015; Wang et al., 2013), and cyhalothrin (Chen et al., 2011; Qin et al., 2015; Wu et al., 2017). All of them are insecticides or acaricides with no approval in the EU (EU Pesticides Database, 2020). In Liu et al. (2020), it was found, that some detected pesticides had been banned before 2014, but could still be found in fruits and vegetables. In addition, 7.4 % of samples contained highly toxic pesticides. Highly toxic pesticides were classified according to the Chinese classification standards for pesticide toxicity, which is based on acute toxicity (LD50 (median lethal dose) < 50 mg/kg (oral, rat) or < 200 mg/kg (skin, rabbit) (Liu et al., 2020). In Chen et al. (2011), unauthorized pesticides were detected over the MRL. Even though toxic and unauthorized pesticides were detected, the majority of the studies concluded, that there was most likely no risk associated with pesticide residues and consumption of vegetables (Chen et al., 2011; Lin et al., 2020; Liu et al., 2020; Wu et al., 2017).

In the EFSA reports, commodities that were imported from China commonly included tea leaves, broccoli, pomelo, goji berries, mushrooms (dried), and passion fruit. For commodities from China, high numbers of cooccurring pesticides and exceedances of MRL were frequently reported. In goji berries from China for example, 29 different pesticide residues were reported, which was the highest amount of cooccurring pesticides in 2018 (EFSA, 2020). China was frequently listed with countries where over 10 % of the samples exceeded the MRLs. In 2014, 44.5 % of samples from China had residues of pesticides under the MRL and 18 % had residues above the MRL (EFSA, 2016). As a result of frequent exceedances in imported products from China and other countries, import controls for certain countries increased as stated in Regulation (EC) No 669/2009. This led to rejections of products from China by the EU and countries outside the EU. Japan raised their standards for pesticides residues on imported spinach, which lead to a direct loss of US \$3 million for Chinese export enterprises (Liu & Guo, 2019). As a response, China tries to improve their food safety law and the execution of it, but still has ongoing difficulties in meeting EU standards (Jia & Jukes, 2013). In 2018, 17.2 % of samples from China were still above the MRLs set by the EU (EFSA, 2020).

4.2 Recovery study

Among the different solvents tested, acetonitrile showed the best results. The recovery for different pesticides and metabolites was generally better using acetonitrile, acetone and acidified acetone (Fig. 17) compared to recovery with SPE (Fig. 18).

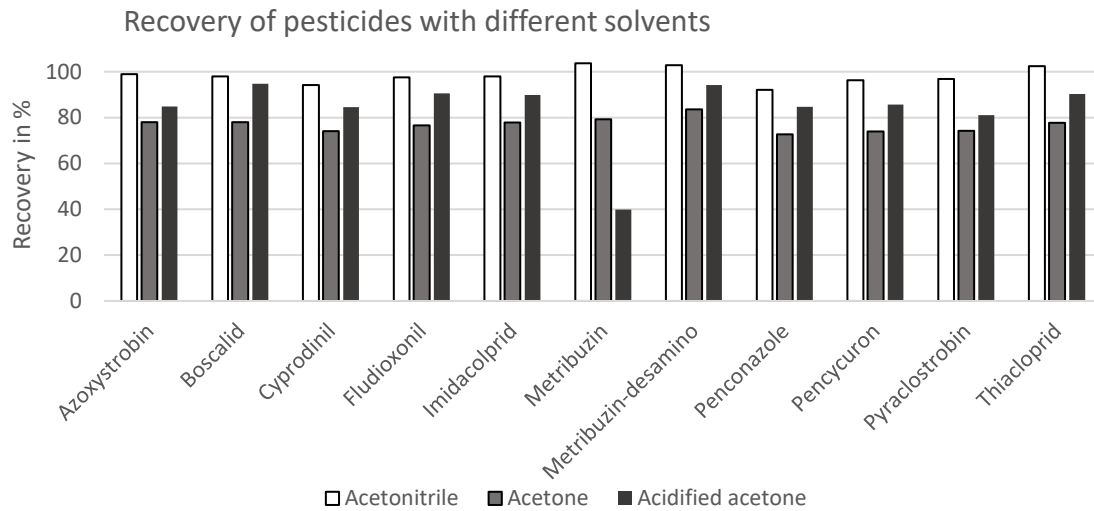


Figure 17: Recovery in % of pesticides from soil, spiked with 25 µg/kg pesticide mix using extraction solvents acetonitrile, acetone, and acidified acetone respectively. Results are average of three soil replicates [n=3].

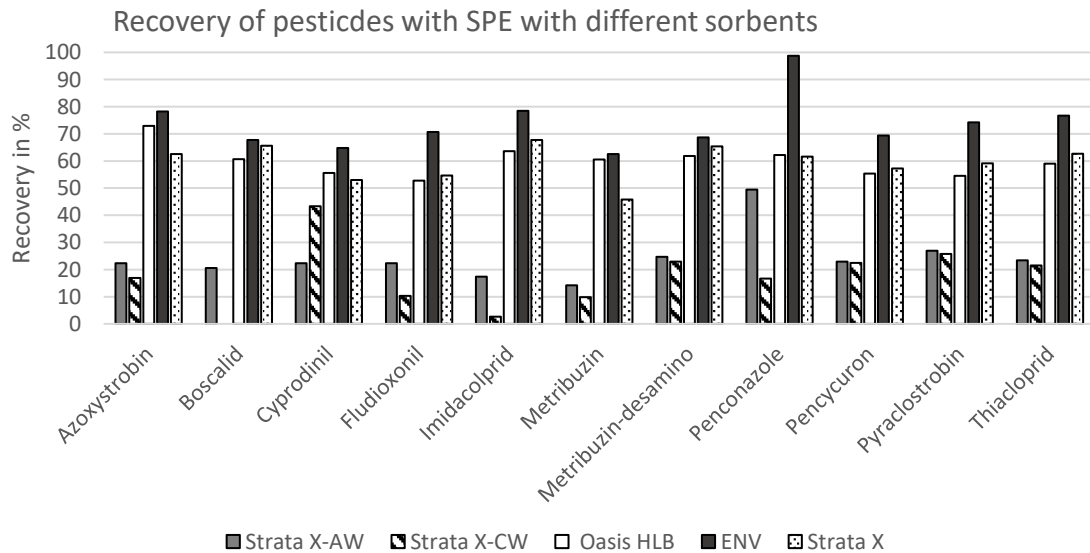


Figure 18: Recovery of pesticides in % with different sorbents for solid phase extraction from soil spiked with 25 µg/kg pesticide mix. Results are average of three soil replicates [n=3].

4.3 Field study

4.3.1 Pesticide residues in carrots from HV

No pesticide residues were detected in the carrot samples from HV in September 2019.

4.3.2 Pesticide residues in soil at HV and HØ

In total nine different pesticides and five degradation products (metabolites) were detected in the soil samples. Their properties can be found in the table below (Table 15). Of all the pesticides and metabolites, 60 % were fungicides, 20 % insecticides, and 20 % herbicides. Most of the metabolites were degradation products of fungicides.

Table 15: Properties of the pesticides detected in the soil at HV and HØ. The information is from the PPDB (2007). Chronic environmental risk values (miljøfarlighet – MF) were from the NIBIO webpage. M: metabolite

Active substance	Class	Structure group	K_{foc} (ml g ⁻¹)	DT ₅₀ field (days)	Water sol. 20°C (mg l ⁻¹)	Mobility	Persistence	Chronic MF value (µg/l)
Azoxystrobin	F	Strobilurin	423	180.7	6.7	Moderate	Persistent	0.95
Boscalid	F	Pyridinecarboxamide	772	254	4.6	Slightly mobile	Persistent	12.5
Cyprodinil	F	Anilinopyrimidine	2277	45	13	Slightly mobile	Moderately	0.18
Fludioxonil	F	Phenylpyrrole	132100	20.5	1.8	Non-mobile	Non-persistent	0.05
Imidacloprid	I	Neonicotinoid	225	191	610	Moderate	Persistent	0.2
Metribuzin	H	Triazinone	48.3	19	10700	Mobile	Non-persistent	0.058
Penconazole	F	Triazole	2205	90	73	Slightly mobile	Moderately	6
Pyraclostrobin	F	Strobilurin	9315	33.3	1.9	Non-mobile	Moderately	0.4
Pyridafol	H/ M	Pyridazine	41.5	97	23800	Moderate	Non-persistent	1
Thiacloprid	I	Neonicotinoid	615	8.1	184	Slightly mobile	Non-persistent	0.064

Of the detected substances, most were approved in Norway, but imidacloprid has not been approved for field use since 2017 but can still be used in greenhouses. Thiacloprid lost its approval recently and can be used until 03.02.2021 in Norway (Mattilsynet, 2012). Pyridafol is a metabolite of pyridate which was allowed for use in Norway until 31.08.18 (Mattilsynet, 2012). Some of the applied substances are Candidates for Substitution, namely acclonifen, cyprodinil, fludioxonil and metribuzin (EC, 2020a), meaning they meet two out of the PBT criteria used in risk assessments.

Residues of pesticides sprayed in 2019

Metribuzin was sprayed at HV and HØ and was detected on both sites before spraying. Metribuzin-desamino was detected in low concentration at HV (0.2 µg/kg) in the beginning of the study period, but not at HØ. No other pesticide was sprayed at HV and consequently it was the pesticides with the highest measured concentration among detected pesticides at HV with 36.7 µg/kg (04.06.19).

Comparing the detected concentrations of metribuzin at HV and HØ, differences can be observed (Fig. 19). More metribuzin was detected in the soil at HV after the first spraying compared to HØ. On the other two spraying dates, the concentration measured in the soil was higher at HØ. The concentration of metribuzin was lower after the second and third application compared to the first one.

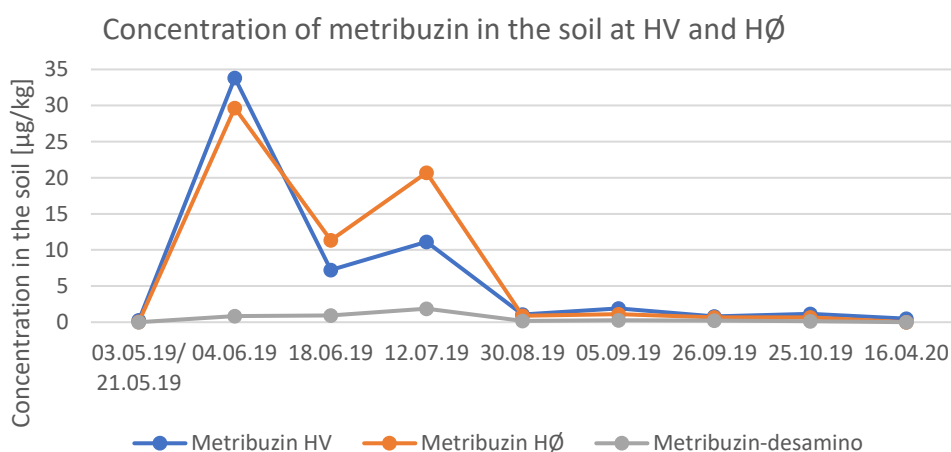


Figure 19: Comparison of metribuzin concentration at HV (blue) and HØ (orange). The average of the three measurements on each date from a sampling depth of 0-10 cm is shown. The concentration of metribuzin-desamino was similar on both sites so one line is for both is shown (grey). The spraying dates were 03.06, 18.06, and 29.06.

Metribuzin-desamino from HV was included in the graph but not metribuzin-desamino from HØ. Its concentration showed little difference between the sites, with 0.32 µg/kg being the biggest difference (12.07.2019). No residue of metribuzin were detected at HØ in April 2020. Metribuzin-desamino was also no longer detected in April 2020 at all.

At HØ, the fungicides boscalid, pyraclostrobin, cyprodinil, and fludioxonil were sprayed in 2019. A strong increase in their concentration is visible after their application. High concentrations of boscalid, cyprodinil, and fludioxonil were detected (Fig. 20). The concentration of pyraclostrobin increased as well but not as much as for the other fungicides. Boscalid, cyprodinil, and fludioxonil showed a steep decrease in concentration in the soil from October to April.

The highest concentration in a single sample was measured for cyprodinil with 312.05 µg/kg soil (05.09.2019), two days after it was sprayed.

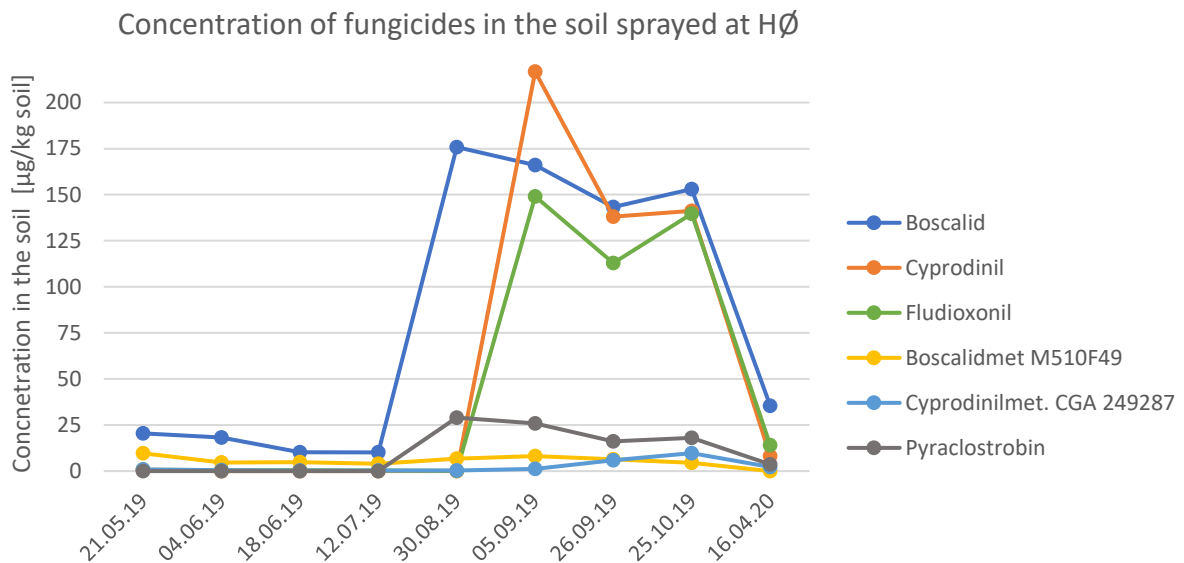


Figure 20: Average pesticide concentration of the three measurements per sampling date for pesticides sprayed in 2019 at HØ. The spraying dates were 20.08 and 03.05. The sampling depth was 0-10 cm.

Some of the fungicides that were sprayed had residues in low concentrations prior to spraying in 2019 (boscalid, cyprodinil, fludioxonil). Residues of boscalid ranged from 20 µg/kg in May to 10 µg/kg in July. Cyprodinil was detected in May and then again after spraying in September 2019. Pyraclostrobin was not detected before spraying. The detected metabolites, cyprodinilmet. and boscalidmet. were detected in low concentrations before spraying. On the last sampling date in April 2020, most substances still had residues, but boscalidmet. was no longer detected.

Soil background pesticides

At HV, several substances were detected that had not been sprayed in 2019 (Fig. 21). The substances were azoxystrobin, boscalid, boscalidmet M510F49, cyprodinil, cyprodinilmet. CGA 249287, fludioxonil, imidacloprid, penconazole, pyraclostrobin, pyridafol, thiachloprid, and thiachloprid-amide M02. Of those pesticides and metabolites, ten were not sprayed since 2016, one was sprayed between 2016 – 2018 (thiachloprid, 2016) of which also a metabolite was detected (thiachloprid-amide M02). Additionally, pyridafol was found, which is a metabolite of pyridate which was sprayed in 2016. Most of the substances were detected throughout the study period (May 2019- April 2020), but pyraclostrobin was no longer detected in 2020.

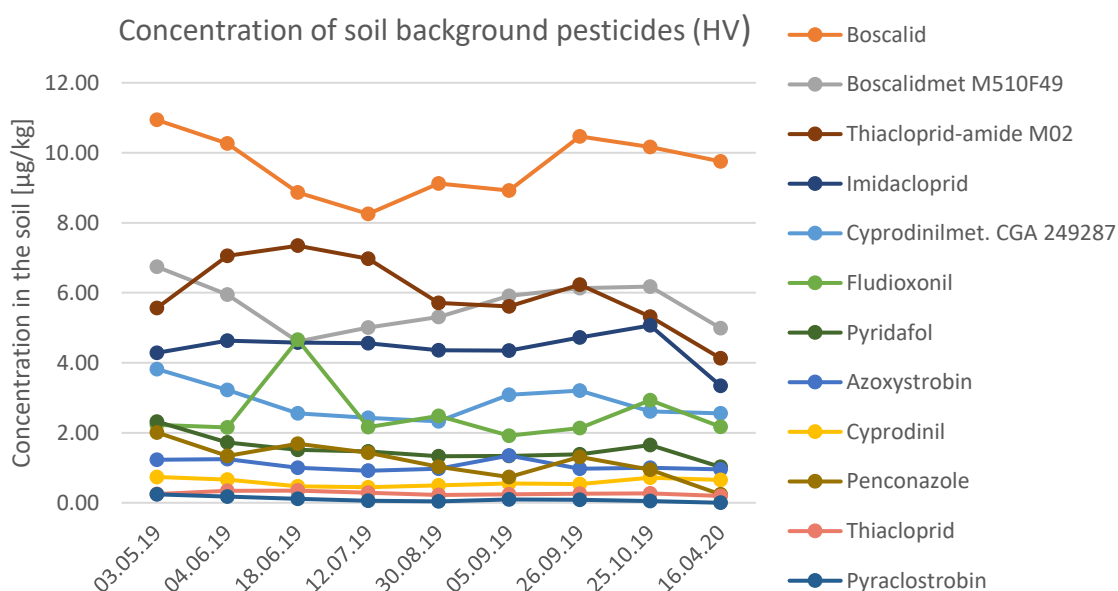


Figure 21: Average concentration of the three measurements on the different sampling dates of soil background pesticides in 2019 at HV. The sampling depth was 0-10 cm.

Boscalid showed the highest concentration in the soil throughout the study period, followed by thiacloprid-amide and boscalidmet. M510F49. For some pesticides very little change in concentration occurs, thiacloprid and pyraclostrobin for example showed a constant concentration. In general, the higher the concentration measured, the more variation was observed.

Background pesticides at HØ included thiacloprid, thiacloprid-amide M02, imidacloprid, penconazole, and pyridafol. Penconazole was only detected in the sample from May 2019 and not thereafter. Imidacloprid showed the highest concentration on 04.06.2019 and declined afterwards. On 05.09.2019, a slight increase in measured concentration can be observed for all pesticides except penconazole (Fig 22).

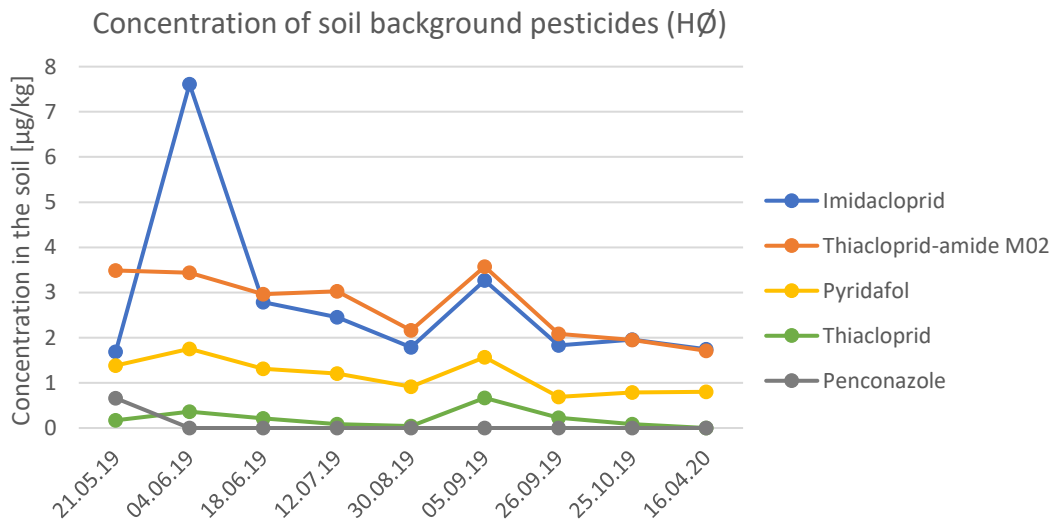


Figure 22: Average concentration of the three measurements on the different sampling dates of detected pesticides that were not sprayed in 2019 at HØ. The sampling depth was 0-10 cm.

4.3.4 Calculated dissipation for pesticides detected in the field study

Pesticides sprayed in 2019

All pesticides sprayed at HV and HØ in 2019 could all be described by SFO kinetics (Table 16). Most pesticides were within the 15 % error value for χ^2 . Metribuzin and metribuzin-desamino were slightly above (16.2, 18.3), but still accepted due to a good visual fit. Metribuzin was applied three times, which probably lead to the variation. The repeated application led to high starting concentrations (t=0) of metribuzin-desamino (Fig. 24). For boscalidmet. high starting concentrations were observed as well, resulting from its detection before boscalid was sprayed at HØ.

Metribuzin was the pesticide with the fastest dissipation of 19.6 days (HV) and 12.4 days (HØ). Metribuzin is a mobile pesticides and leaching might have contributed to the fast dissipation. Both metribuzin and metribuzin-desamino were classified as non-persistent according to the half-lives calculated in this study. The classification from the PPDB (2020) divides the pesticides according to their half-life in non-persistent (DT50 < 30 days), moderately persistent (DT50: 30-100 days), persistent (DT50: 100-365 days) and very persistent (DT50 > 365 days). The remaining pesticides were classified as moderately persistent and boscalid as persistent.

Table 16: Model information for pesticides sprayed in 2019 at HV and HØ. Estimated values for the M0 and k. Standard deviation (σ) for k. chi-square (X^2), r^2 and residues for the modes are shown as well as DT50 and DT90. X^2 values > 15 % are marked red.

Pesticide	Model	M0	k	σ	χ^2	r^2	RSS	DT50	DT90
HV									
Metribuzin	SFO	11.07	0.035	0.005	16.2	0.934	36	19.6	65.2
Metribuzin-desamino	SFO	1.904	0.257	0.073	18.3	0.909	36	2.69	8.95
HØ									
Boscalid	SFO	178.1	0.006	0.002	7.74	0.604	30	122	405
Boscalidmet	SFO	7.844	0.01	0.012	12.8	0.838	30	68.9	229
Cyprodinil	SFO	214.7	0.013	0.007	12.5	0.560	24	53.4	177
Cyprodinilmet	SFO	0.988	0.019	0.012	11.1	0.866	24	36.8	122
Fludioxonil	SFO	147.2	0.01	0.004	4.27	0.723	11190	70.5	234
Metribuzin	SFO	20.72	0.056	0.015	6.81	0.932	36	12.4	41.2
Metribuzin-desamino	SFO	1.868	0.036	0.072	14.1	0.901	36	18.8	62.3
Pyraclostrobin	SFO	27.29	0.01	0.004	10.8	0.627	647	71.4	237

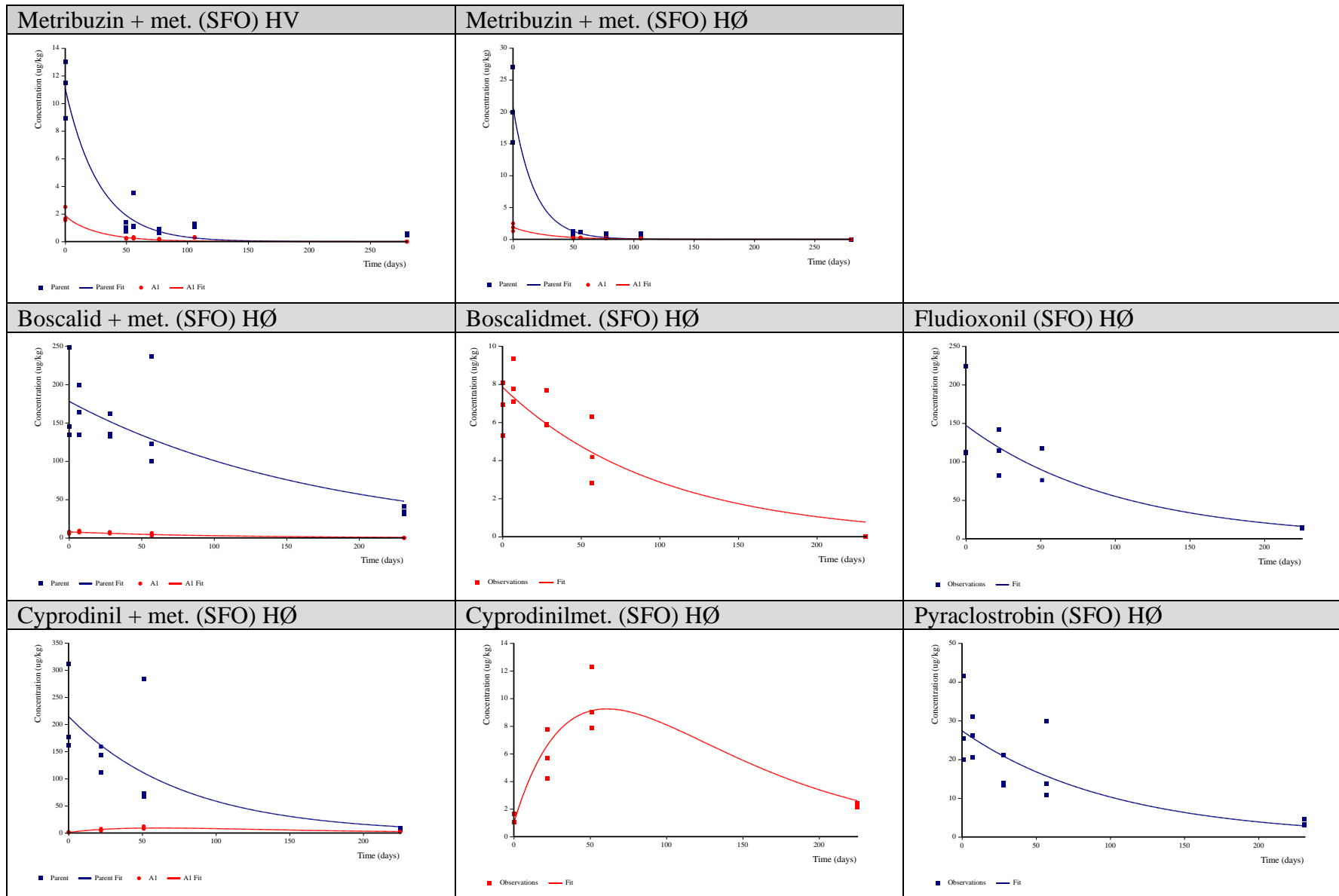


Figure 23: Dissipation curves for residues of pesticides sprayed at HV and HØ in 2019. On the y-axis is the concentration in µg/kg and on the x-axis the time. The blue curves represent the model for the parent component and the red curves for metabolites. Dots are the observed values in the field.

Soil background pesticides

Of the soil background pesticides (not sprayed in 2019) at HV, most had calculated DT50 values over 1000 years, namely azoxystrobin, fludioxonil, boscalid and its metabolite, cyprodinil, and imidacloprid (Table 17). The concentration of those pesticides did not change much over the study period and the dissipation curves were almost horizontal (Fig. 24). Most detected substances at HV were classified as very persistent according to their calculated half-life. Exceptions were penconazole (persistent) and pyraclostrobin (moderately persistent).

Models for HØ are presented in Fig 25. At HØ, penconazole was only detected on 21.05.19 and therefore no dissipation was calculated. Imidacloprid was the pesticide with the longest DT50 (>1000 days). Thiacloprid, thiacloprid-amide and pyridafol were classified as persistent with DT50 values between 100 – 365 days. For background pesticides detected on both sites, the dissipation appeared to be faster at HØ compared to HV with shorter half-life and a more visible decline in concentration.

For thiacloprid the error % of the χ^2 test was 72.6. The model was still accepted due to a visually acceptable fit. Thiacloprid had not been sprayed in 2019 and the heterogeneity of the soil might account for the high error value.

Table 17: Model information for background pesticides at HV and HØ. Estimated values for the M0 and k and α and β for FOMC models. Standard deviation (σ) for k, chi-square (χ^2), r^2 and residues for the modes are shown as well as DT50 and DT90. χ^2 values > 15 % are marked red.

Pesticide	Model	M0	k	σ	χ^2	r^2	RSS	DT50	DT90
HV									
Azoxystrobin	SFO	1.15	<0.001	<0.001	10.2	0.075	1.262	1130	3750
Boscalid	SFO	9.658	<0.001	<0.001	6.97	<0.001	54	>10000	>10000
Boscalid met	SFO	5.868	<0.001	0.005	9.52	0.045	54	2120	7030
Cyprodinil	SFO	0.584	<0.001	<0.001	14.1	0.01	54	>10000	>10000
Cyprodinil met	SFO	3.142	<0.001	<0.001	12.3	0.084	54	870	2890
Fludioxonil	SFO	2.744	<0.001	0.001	24.6	0.019	36.31	1020	3400
Imidacloprid	SFO	4.729	<0.001	<0.001	6.72	0.137	10.16	1240	4130
Penconazole	SFO	1.914	0.005	<0.001	13.9	0.605	3.591	147	488
Pyridafol	FOMC	2.32	α : 0.150 β : 4.229	α : 0.06 β : 6.056	7.27	0.661	1.355	422	>10000
Thiacloprid	SFO	0.311	0.013	<0.001	10.3	0.313	54	537	1780
Thiacloprid-amide	SFO	6.927	0.001	0.071	8.97	0.335	54	519	1720
HØ									
Imidacloprid	SFO	2.156	<0.001	<0.001	13.3	0.073	4.897	1070	3560
Pyridafol	FOMC	1.591	α : 0.537 β : 64.75	α : 0.407 β : 88.98	13.4	0.624	1.391	171	4640
Thiacloprid	SFO	0.267	0.003	0.005	72.2	0.023	54	247	822
Thiacloprid-amide	SFO	3.417	0.003	0.041	13	0.391	54	276	916

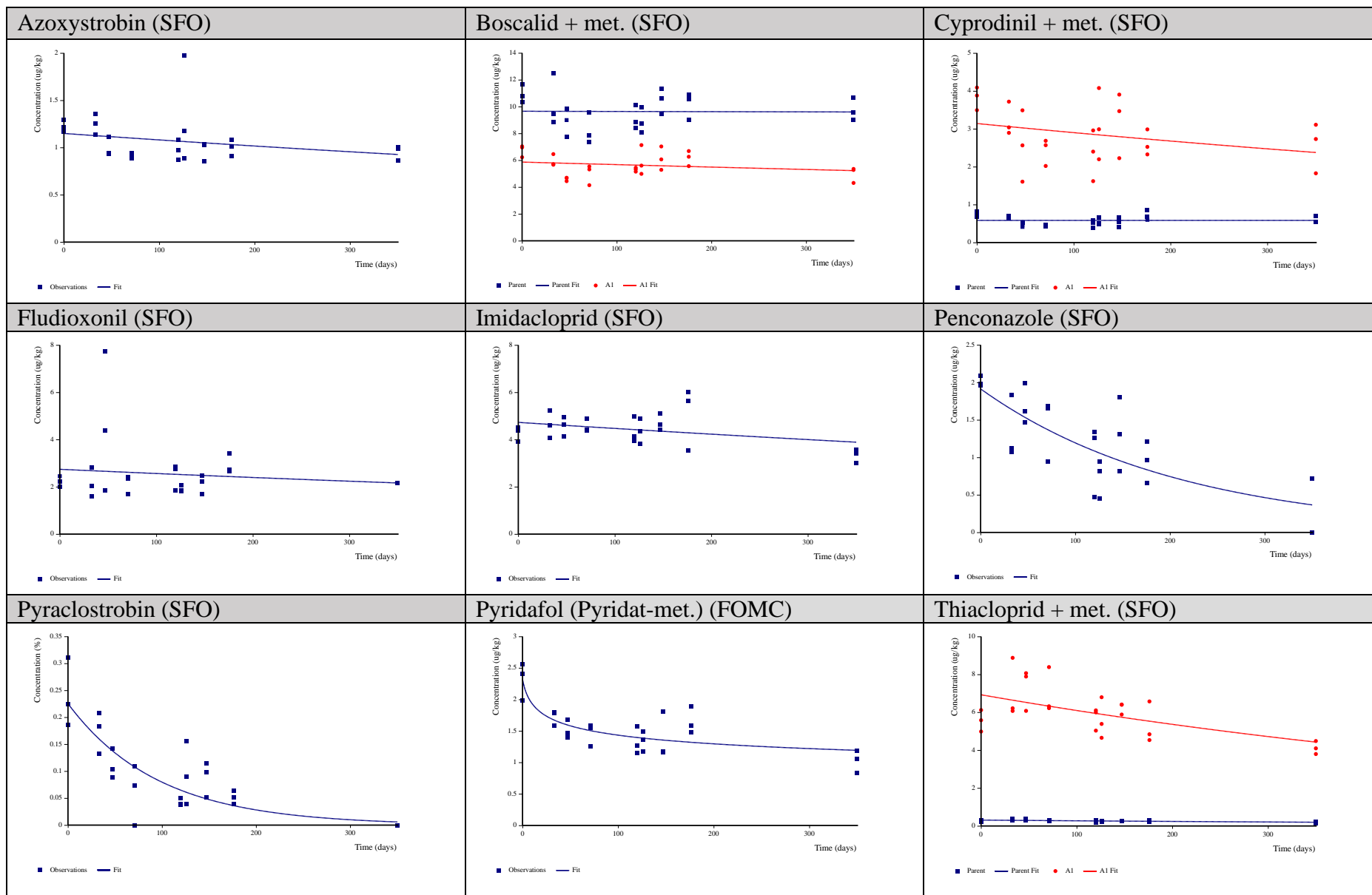


Figure 24: Dissipation curves for residues of background pesticides detected at HV in 2019. On the y-axis is the concentration in $\mu\text{g}/\text{kg}$ and on the x-axis the time. The blue curves are the model for the parent component and the red curves for metabolites. Dots are the observed values in the field.

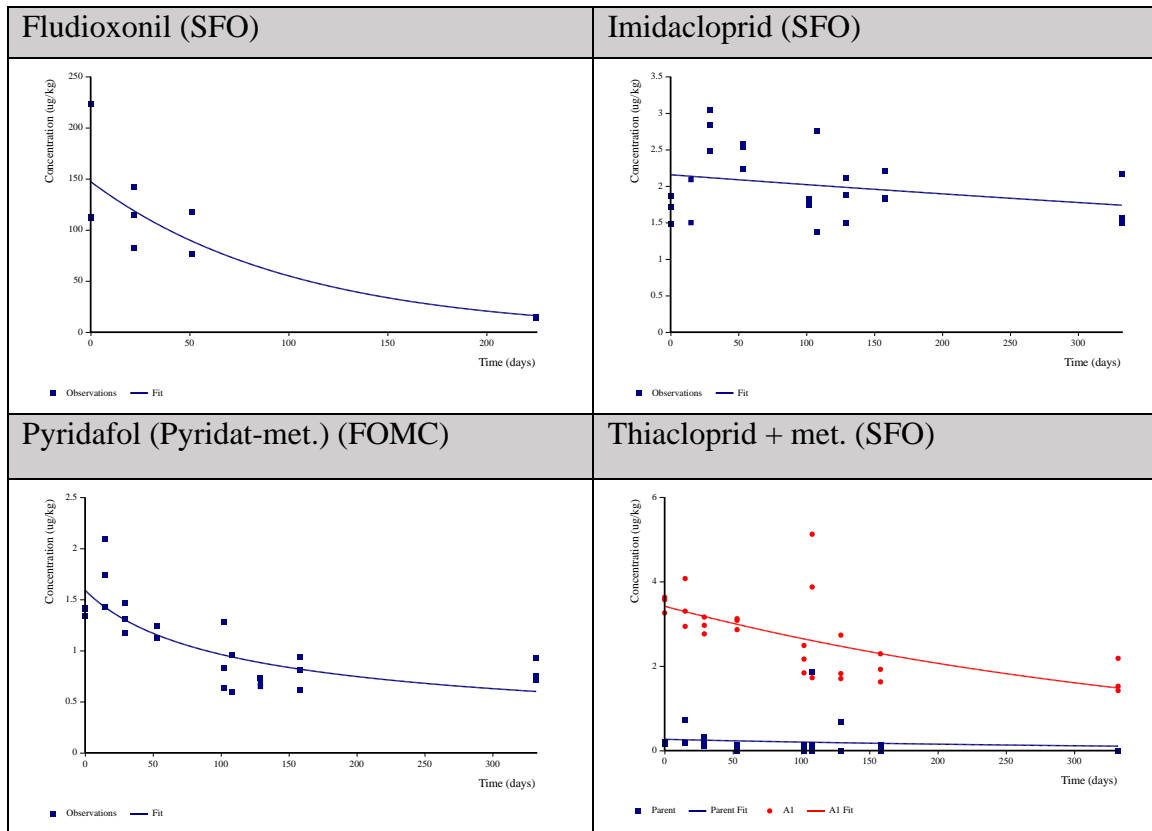


Figure 25: Dissipation curves for HØ with the concentration in µg/kg on the y-axis and the time on the x-axis. The blue curves are for parent components and red curves for metabolites. Dots are the observed values in the field.

5. Discussion

5.1 Comparison of pesticide residues in carrots and vegetables

Differences among the regions in the level of residues can be pointed out. A similar trend was observed in carrot samples analysed in Norway and Germany, where the number of samples without residues decreased over time (Fig. 9, Fig. 15). In Norway, 59.1 % of Norwegian carrots were without residue in 2008, and in 2018 it was 37.0 %. The decrease might be explained by changes in agricultural practices and pesticides used. Increased number of applications but lower concentrations could be mentioned as an example, but no conclusion can be drawn from the data here.

The average over the years for samples without residues was around 50 % for carrots analysed in Norway and Germany. In the EU, a higher percentage of carrot samples was without pesticide residues (64.5 %, Fig. 13). In one study from China, pesticide residue levels in over 800 carrot samples were reported, with 14.3 % having residues below the MRL and 0.24 % showing residues over the MRL (Wu et al., 2017). This might indicate lower amounts of residues in carrots in China compared to the EU, where 1.9 % of samples exceeded the MRL. The percentage of MRL exceedances in carrot samples analysed in Germany (0.2 %) and Norway (0 %) was similar to exceedances of MRL in carrots samples from China (0.24 %). However, the carrot samples were from a north western region in China and in another study it was pointed out that there is a great intercity variability of residues levels in China (Liu et al., 2020), which could lead to a different outcome if more regions were included.

Since little data on pesticide residues in carrots in China was available, vegetables in China and the EU were compared. The percentage of samples with residues were similar with around 40.6 % in the EU and 34.7 % in China. When MRL exceedances of vegetables were compared, China had a higher average (5.9 %) compared to the EU average (3.9 %). It should be kept in mind, that the number of active ingredients with MRL is lower in China than in the EU (Handford et al., 2015). When EU MRLs were applied to findings in the study by Liu et al. (2020), the compliance rate dropped from 97.5 % to 86.6 %, indicating that the residue levels might be higher in China compared to the EU, but more data is necessary to come to a conclusion.

Fungicides were the main type of pesticides detected in carrots and vegetables in Norway, the EU, and Germany. Information available for China pointed towards insecticides being the main class detected in vegetable samples. Commonly used pesticides in China were often studied, these being mainly insecticides. Pesticides approved in carrots in China were also mainly insecticides (Table 12), which would support the assumption that insecticides are the main class found in vegetables from China. This is supported by results from Germany and Norway where fungicides were the main class of pesticides approved in carrots and the main class detected in carrots. If more pesticides were included in the residue analysis in China, results might be different, and as reported in Lin et al. (2020), 35.6 % of analysed samples showed residues of three fungicides. This might indicate that residues of fungicides are more common in vegetables from China than reported in the studies that focused mainly on insecticides.

In carrots grown in Norway, eight different pesticides were detected (Fig. 10). The average number of different pesticides detected per year was 47.3 in the EU, 42.5 in Germany, and 7.7 in Norway. A lower amount of different residues in carrots in Norway might be surprising, since the degradation of pesticides is expected to be slower in colder climates (VKM, 2015), but differences in number of approved pesticides might explain the lower number of pesticides detected. In Norway, around 120 pesticides are approved of which 16 are approved in carrots (Table 10). In Germany, 43 active ingredients are approved in carrots out of a total of 285 in 2018 (BVL, 2020a). Therefore, it could be argued that there is a relationship between the number of approved pesticides and the number of pesticide residues found in food commodities. Consequently, regulations and their implementations are important factors affecting residues in the environment. This was also found in a study from China where residue levels in vegetables were dependent on policymaking (Liu et al., 2020). The differences could also stem from application techniques, agricultural practices, and climatic conditions. In Norway, the cold climate conditions were found to play an important role in degradation of pesticides and especially fungicides were reported to degrade slower in colder climates (Almvik et al., 2014).

Boscalid was the most detected pesticides in Norway, the EU, and Germany. The pesticide residues detected in carrots were similar with aconitine, iprodione, difenoconazole, and linuron being often being detected in Norway, the EU, and Germany. All of them are or were approved in carrots, but linuron and iprodione lost their approval in the EU in 2017 and 2018 respectively. Data from the Norway, the EU, and Germany point towards pesticides that are approved in carrots are mainly being the ones found in carrot samples.

Little overlap between residues in vegetables in China and the EU was observed. Organophosphates were frequently detected in China (Chen et al., 2011; Qin et al., 2015; Wang et al., 2013; Wu et al., 2017) . Several of them being neurotoxicants or having endocrine disrupting properties (PPDB, 2007). Many of the frequently detected pesticides such as acephate, chlorpyrifos, omethoate and phorate are banned in the EU (EU Pesticides Database, 2020). China is trying to improve their legislation system but has met some difficulties with enforcing it (Yang et al., 2014).

Despite their differences some similarities among the countries and regions can be mentioned. In none of the regions a health risk for the consumer due to pesticides was identified (Chen et al., 2011; Lin et al., 2020; Liu et al., 2020). Additionally, the cooccurrence of two pesticides was most common among multiple residues in vegetables and carrots. In all regions, residues of non-approved pesticides were detected in carrots and vegetables. DDT for example has been banned in the EU since 1972 (PPDB, 2020), but is frequently detected in carrots in the EU and Germany. Other banned pesticides included ethirimol, diazinon, and dieldrin. In a study from northwest China, selected banned and restricted pesticides and their residues in vegetables were investigated (Yu et al., 2017). In 7.7 % of samples exceedances of MRL of banned and restricted pesticides were observed. Banned pesticides included methylbenzene, parathion, omethoate, and carbofuran. Among the restricted pesticides was phoxim which is officially approved for use in carrots. Some of the banned pesticides detected in the study were also found in other studies from China (Chen et al., 2011; Liu et al., 2020; Qin et al., 2015), for example fenvalerate, and omethoate. This suggests that residues of pesticides that had been banned, are still present in soils and can be taken up by vegetables. Data on pesticide residues in soils in the EU supports this. In Silva et al. (2019) residues of DDT, dieldrin and twelve other residues of no longer approved pesticides were detected.

5.2 Pesticide residues in soil at HV and HØ

5.2.1 Cooccurrence of pesticides in soil

In the soils from HV and HØ, 14 and 13 different pesticides, including metabolites were detected. In 2019, a study about pesticide residues in European agricultural soils was published (Silva et al., 2019), where 83 % of the soils analysed had residues. Multiple residues were found in 58 % and predominantly with 2-5 different components. Soils from northern Europe showed high numbers of cooccurring pesticides with 10 or more being common, while soil samples from southern Europe had no samples with more than 10 pesticide residues (Silva et al., 2019). Northern Europe included the United Kingdom and Denmark, where the climatic conditions are quite different compared to Norway, but in the study by Silva et al. (2019), no significant relationship was found between detected amounts and the climate. Nevertheless, the general influence of climate on pesticide degradation had been described, with the potential of increased leaching during winter in cold climate conditions (Stenrød et al., 2008) and slower degradation of fungicides under Norwegian conditions (Almvik et al., 2014). It is known that no or very little degradation takes place at cold temperatures due to the influence of temperature on the microbial activity (VKM, 2015) which might explain the higher number of different pesticide residues in Norwegian soils compared to soils in the rest of Europe.

The number of different residues may not solely depend on the climate but also on the crop. Soils of root crops were found to have higher amounts of residues with 85 % of them having multiple residues (Silva et al., 2019). A Czech study found, that the application of different pesticides was highest in beet (Kosubová et al., 2020). Additionally, a potato field in Norway showed higher concentrations of pesticides compared to a grain field (Eklo et al., 2019), which would further support that it is not only dependent on the climate but also on other factors like number of applications and crop type. Pesticide and soil properties were also found to have a strong correlation with total pesticides content especially the organic carbon content (Silva et al., 2019). For cationic pesticides, clay minerals are more important (Blume et al., 2016). The amount applied, water solubility, and DT50 values (Kosubová et al., 2020) were also found to play an important role in terms of cooccurrence.

Another aspect could be, that residues depend on the class of pesticide. Most pesticide residues detected in soils from HV and HØ were fungicides or metabolites of fungicides. In the previous years (2016-2018), mainly insecticides and herbicides were sprayed, yet fungicides were the dominating class among the residues in the soil. Even though fungicides were applied at HØ in 2019, residues of boscalid, cyprodinil and fludioxonil were detected prior to spraying.

In a Norwegian study, fungicides were reported to degrade slower under cold conditions compared to warmer climates (Almvik et al., 2014). The frequent detection of fungicides in soils was also reported in other studies, where in total seven compounds were quantified in >10 % of soil samples of which four were fungicides (boscalid, epoxiconazole, tebuconazole, and phthalimide which is metabolite of folpet, (Silva et al., 2019)). A Czech study also found fungicides to be the dominating class among the compounds detected in the soil (Kosubová et al., 2020). The presence of residues of fungicides seems to be a widespread phenomenon, in Europe and especially in colder climates.

5.2.2 Pesticides sprayed in 2019 and their concentration in the soil

Metribuzin was the first active ingredient to be sprayed and detected on both sites, which makes it possible to compare the concentrations in the soils at the two sites. The amounts detected varied between HV and HØ (Fig. 19). When metribuzin was sprayed the first time, the plants were still very small and higher amounts could have reached the soil. The first dose applied was also the largest dosage, explaining the higher amount of metribuzin detected. On the two later dates, an insect net was covering the rows and metribuzin and aclonifen were sprayed on top of the net, possibly influencing the amount reaching the soil. In addition, the carrots at HV were irrigated three times after the last application of metribuzin, which could have led to increased leaching of metribuzin at HV compared to HØ. Metribuzin is a mobile pesticides and was frequently detected in high concentrations in catchments in Norway (Stenrød, 2015). In previous studies the influence of application technique, crop stand, and agricultural practices on dissipation of pesticides in the soil had been described (Kosubová et al., 2020). The concentration of metribuzin-desamino was very similar at both sites, pointing more towards factors other than microbial degradation causing the faster dissipation at HØ. Preferential flows could also result in the differences, which will be discussed later.

In August, after the removal of the insect net, fungicides were sprayed at HØ and a steep increase in the concentration of boscalid, cyprodinil, and fludioxonil was observed (Fig. 20). Over winter, the concentrations of the three fungicides decreased steeply. Dissipation of pesticides is a result of degradation, sorption, and transport processes. Soil temperature is an important factor for degradation due to its influence on microbial activity, and with low temperatures near or below zero no degradation is expected to take place (VKM, 2015).

Looking at the soil temperature in the period between October 2019 and April 2020, the soil temperature from January until March was 0°C (Fig. 2a) and no or little degradation should have taken place. Yet the concentration of boscalid, cyprodinil and fludioxonil decreased sharply. In previous studies from Norway a higher mobility of pesticides in frozen soil compared to unfrozen soil had been observed for some pesticides (Holten et al., 2019; Stenrød et al., 2008). This has been documented for boscalid (Holten et al., 2019), but not for the other two pesticides. Increased mobility and leaching could be a possible explanation for the decrease in concentration over the winter, but further investigation is needed.

5.3 Field dissipation and DT50 values of pesticides sprayed in 2019

The modelled DT50 values of pesticide sprayed at HV and HØ were compared to EU endpoints reported in the PPDB (2020) as well as other studies reporting on field dissipation of those pesticides. Measured residues of pesticides before spraying were not included in the modelling process which could influence the calculated dissipation in the field. Additionally, no data on field dissipation of pesticides under a carrot cropping system were available which might lead to further differences in the dissipation rate.

For boscalid the calculated half-life at HØ was shorter than reported in another study from Norway (Almvik et al., 2014), with 96.2 days compared to 157- >700 days. However, in that field experiment, pesticides were applied to the soil without crops. At HØ, carrots were planted and could potentially have taken up some of the active ingredient which would reduce the dissipation time for boscalid. This could be supported by findings of residues in carrots where boscalid was the most detected pesticide in carrots from Norway and Germany (Fig. 10, Fig. 16). The DT50 value of boscalid was generally lower than EU endpoints and values reported from a field study of leek conducted in Germany (Karlsson et al., 2016). Another explanation for the faster dissipation could be a reduction in concentration over winter due to leaching processes. Reduction in concentration is unlikely to be a result of microbial activity due to low temperatures being known to limit the activity of microbes (Blume et al., 2016). Cold climate conditions were reported to influence the leaching of some pesticides. Increased leaching for boscalid as a result of freeze-thaw cycles has been demonstrated by Holten et al. (2019).

Table 18: Half-lives for pesticides sprayed at HV and HØ in 2019 compared half-lives from other studies.

	HØ [days]	Range EU field studies [days]	Range EU lab study [days]	Other field study [days]
Boscalid	96.2	196-312.2	103.3-1214.4	Norway: 157- >700 (Almvik et al., 2014) Germany: 104-132, leek (Karlsson et al., 2016) China: 6.1, 8.0, strawberries (Chen & Zhang, 2010) China: 17.33, cucumber (He et al., 2020)
Boscalidmet M510F49	68.9		356	
Cyprodinil	128	11-98	31-41	China: 5.8–15.6, grapes (Zhang et al., 2015) China: 14.5, 12.5, strawberries (Liu et al., 2011)
Cyprodinilmet CGA 249287	36.8	35-325	76-153	
Fludioxonil	70.5	8-43	119-365	China. 6-12.1, grapes (Zhang et al., 2015)
Metribuzin	12.4 19.6 (HV)	19	5.98-7.76	Norway: 27-48 (Stenrød et al., 2008) England: 9.3-49.4 (Kah et al., 2007)
Metribuzin- desamino	18.8 2.69 (HV)		2.15-6.83	Denmark: 29 (Henriksen et al., 2004) (lab)
Pyraclostrobin	71.4	5.2-181	4.2-95.4	Norway: 55-329 (Eklo, 2019) China: 13.1-16.5, peanuts (Zhang et al., 2012)

Prolonged DT50 values under Norwegian conditions were observed for cyprodinil and fludioxonil. The DT50 value for cyprodinil was 128 days at HØ, which is longer than the EU endpoints in field and lab studies. Its metabolite (CGA 249287) was within the range for EU field studies (35-325 days) and lower than EU lab studies (76-153 days). In contrast to boscalid, cyprodinil is found very seldomly in carrot samples analysed in Norway, the EU, and Germany, suggesting poor uptake of cyprodinil. The dissipation of fludioxonil showed a similar tendency to cyprodinil. Calculated dissipation was slower compared to dissipation reported by EU field studies and faster than laboratory studies stated by the EU. Fludioxonil was also not reported in carrot samples from Norway and in very few samples from the EU and Germany. However, both pesticides were sprayed at the end of the growing season (03.09.2019) and slower degradation could be related to decreasing temperatures.

For metribuzin, a slower degradation due to colder climate was expected (Benoit et al., 2007), but metribuzin was sprayed in June and July, when the temperatures in the soil and air were high. A Danish study showed a high dependency of metribuzin dissipation on temperature (Henriksen et al., 2004). Additionally, metribuzin was sprayed three times, but the degradation was calculated using concentrations from the last application until the end of the study period, resulting in lower starting values for metribuzin and higher starting values for metribuzin-desmino, which also influenced the calculated dissipation. Differences in DT50 values were observed at HV (19.6 days) and HØ (12.4 days), with slightly slower degradation at HV. Metribuzin-desamino was degraded slower at HØ (18.8 days) compared to HV (2.69), even though the concentrations of the metabolite in the soil showed little differences between the sites. Repeated application of metribuzin had an influence on the starting value for the modelling, potentially leading to errors in the models for metribuzin.

The calculated half-life of pyraclostrobin (71.4 days) was both within the range for field and lab studies in the EU and very close to a value reported in an earlier study from Norway (Eklo, 2019). Pyraclostrobin is classified as moderately persistent, non-volatile and with a low leaching potential (PPDB, 2007). The dissipation of pyraclostrobin was found to be faster under anaerobic conditions due to a higher efficiency of anaerobic bacteria in degrading pyraclostrobin (Reddy et al., 2013). Pyraclostrobin was sprayed in August and a lot of precipitation occurred from August-November (Fig. 2), which might explain the fast dissipation.

Compared to the dissipation of the pesticides sprayed at HV and HØ, the dissipation in China appears to be much faster than in Norway. However, the field trials in China covered a very short time (21-28 days) compared to the study at HV and HØ and pesticides were applied multiple times in high dosages (Liu et al., 2011; Zhang et al., 2015). In addition, different crops were used which can further influence the dissipation, making it hard to compare the dissipation, and no conclusion can be drawn. Dissipation is dependent on different process, namely degradation, sorption and transport, all of whom display great variability across an agricultural field as well as different climates, making interpretation and comparison between sites difficult (Kah et al., 2007; Stenrød et al., 2008).

5.4 Persistency of pesticides sprayed in previous years (before 2019)

Of the detected pesticides and metabolites at HV and HØ, several of them had been sprayed prior to 2019 and some prior to 2016, indicating long persistence in the soil. The concentrations were low, ranging from 0.02 µg/kg up to 11 µg/kg, with HØ showing lower concentrations than HV (Fig. 21, Fig. 22).

Of background pesticides that were detected in the soils in both sites, HØ showed lower concentrations as well as shorter half-lives. The DT50 value of pyridafof for example was 250 days longer at HV compared to HØ. A similar trend was observed for thiacloprid with the dissipation being 270 days longer at HV than at HØ. On both sites the TOC content was similar (2.4 % and 2.2 %), as well as the clay percentage (16 and 18 %), which both play an important role in the sorption of pesticides (Blume et al., 2016). Soil analyses for HV and HØ were performed with soil samples taken from the top layer only (0-10 cm). According to kilden.no, there is a difference between the two sites with HV belonging to the texture group silt and HØ to silty loam. Preferential flow through macropores were found to play an important role in the transportation of pesticides (Holten et al., 2018), with increased mean macropore flow in silty loam compared to silty soils. The classification from kilden.no included soil analyses from deeper layers which might give different result compared to the topsoil analysis in this study. Therefore, an overall faster dissipation of pesticides at HØ could be explained by increased macropore flow.

For many of the long-term residues in the soil, the concentration over the study period changed very little, and consequently the calculated half live for many of them was over 10 000 years. In Blume et al. (2016), the phenomenon of aging is described, where the bioavailability of pesticides decrease over time and non-extractable residues can be formed, reducing the availability and exposure of soil organisms and aquatic organisms. However, small fractions of non-extractable residues can be liberated by physiochemical and biochemical reactions (Blume et al., 2016), making them bioavailable again.

Other studies also report long persistence of pesticides in soil. In a study from Switzerland, it was found that 38 % of detected pesticides were not sprayed between 1995 and 2008 which implies that they have been sprayed before 1995 (Chiaia-Hernandez et al., 2017). Transport from neighbouring fields or incomplete spraying journals were given as an alternative explanation, but other studies reported long term residues as well. In a study from Czech it was

suggested that up to 69 % of pesticide residues were inherited from previous growing seasons (Kosubová et al., 2020). For HV only two compounds were residues from a substance sprayed in 2019, with the rest being inherited from previous growing seasons. Even the pesticides sprayed (metribuzin) showed residues before spraying in June 2019.

Pesticides with basic properties are known to sorb much stronger to soils than acidic pesticides (Kah et al., 2007). Of the background pesticides, none had acidic properties and three showed basic properties, namely cyprodinil, metribuzin and penconazole (PPDB, 2007). All three of them showed residues at HV and HØ in the beginning of the study period. Additionally, most of the persistent pesticides had a low water solubility (azoxystrobin, boscalid, cyprodinil, fludioxonil, pyraclostrobin) which could further explain their persistence in soil.

AT both sites, residues of the neonicotinoids imidacloprid and thiacloprid were found with calculated half-lives of over 1000 days for imidacloprid and between 200–600 days for thiacloprid. The degradation of neonicotinoids is fast, but some soil conditions have been identified that can lead to long persistence. The sorption of imidacloprid was found to be positively related to OM and mineral clay content as well as lower desorption at low temperatures leading potentially to accumulation in soils having those properties (Bonmatin et al., 2015). In the field study at HV and HØ, these factors could explain the long-term residues of imidacloprid.

Residues of neonicotinoid seem to be common, in the EU. In a study from France, 91 % of soil samples showed residues of imidacloprid ($>0.1 \mu\text{g}/\text{kg}$), even though only 15 % of sites used seeds with imidacloprid coating (Bonmatin et al., 2005). A study from Switzerland found residues of imidacloprid in 87 % of soil samples and thiacloprid in 43 % of soil samples, leading to potentially chronic exposure of non-target organisms (Humann-Guillemot et al., 2019). Potential negative effects of neonicotinoids on biodiversity were reported (Goulson, 2013). Most neonicotinoids are phased out and thiacloprid is no longer approved in the EU whereas imidacloprid is banned for open field use but still allowed for use in greenhouses (EU Pesticides Database, 2020).

5.5 Consequences and significance of pesticide residues in food crops and soil

Fungicides were the dominating class of pesticide residues found in carrots and soils in Norway. Especially under cold climate conditions the degradation of fungicides seems to be slower (Almvik et al., 2014) as shown for some pesticides in this study as well. Fungicides in general were found to have greater negative effects on non-target soil organisms than herbicides or insecticides (Bünemann et al., 2006), making further studies on persistence of fungicides and uptake into plants important.

Furthermore it is unknown how climate change will affect the degradation of pesticides and increased temperatures might lead to faster degradation (Babut et al., 2013). Increased precipitation due to climate change could also lead to increased surface runoff and lead to an increased risk of pesticide exposure for the aquatic biota (Babut et al., 2013). Additionally, higher application rates as a result of increased pest pressure could lead to further environmental exposure to pesticides (Stenrød et al., 2016).

In this study, long-term residues of pesticides in the soil occurred. With DT50 values being a main part of the risk assessment and approval of pesticides, more field dissipation studies of pesticides under real world scenarios are needed to further minimize the exposure of non-target organisms and to reduce residues in the environment.

5.6 Uncertainties and limitations

Differences in pesticide residues in carrots between the differences in Norway, the EU, and Germany were difficult to quantify, due to differences in reporting. It would have been interesting to compare which pesticides are found in carrots in Norway, the EU, and Germany and non-EU countries, but in Germany for example, pesticide that were detected were not reported according to the origin of the samples, making the differentiation between samples from Germany, the EU and from non-EU countries impossible (BVL, 2020b). The same occurred in the EFSA reports, where pesticide occurring in a commodity were not reported according to their origin. For China, only one study reported data on pesticide residues in carrots. Some studies reported on residues in vegetable, but mainly commonly used pesticides were mainly the ones analysed (Chen et al., 2011; Qin et al., 2015; Wang et al., 2013; Wu et al., 2017). Therefore, more data on pesticide residues would be necessary to gain a better overview of differences between countries.

The field study was limited to carrots on two sites in Norway, which is not sufficient to give an indication on residue levels and field dissipation. Soils from root crops were generally found to have significantly more pesticide residues compared to soils from other crops (Silva et al., 2019). The residue level and the field dissipation of pesticides detected at HV and HØ might be very different in other types of crops. In addition, no other field studies on field dissipation in a carrot cropping system were available. Field experiments where current agricultural practices are observed could be a valuable addition to current field studies but are challenging at the same time. It was not always possible to take soil samples on the date the pesticides were sprayed, resulting in variation between the starting points for the modelling of the field dissipation.

Neither soil properties, pesticide properties, or the influence on dissipation and cooccurrence of residues was analysed statistically. Even though the fields were next to each other, many factors were different, including the soil type, number of applied pesticides, crop cover, cultivation method and cropping history. This made comparing the fields difficult. More studies of factors and properties influencing field dissipation are necessary for a better understanding of factors influencing the dissipation under different climatic conditions and current agricultural practices. Additionally, more parameters could have been measured to further investigate the differences between the sites, for example the microbial biomass or activity, to further explain differences in dissipation time. No residues were detected in carrots from HV, but metribuzin was sprayed at HV field early in the season, therefore it would have been interesting to have carrots from HØ as well since fungicides were sprayed there. Especially with regard to fungicides being the most detected class in carrot samples from Norway.

6. Conclusion

Fungicides are the most common class of pesticides approved and detected in carrots in Norway, the EU and Germany. In China insecticides are the main class approved in carrots and found in vegetables. Levels of pesticide residues were lower in China compared to the EU with a higher number of pesticides being residue free. A higher number of samples with residues over the MRL were observed in China compared to the EU. Among detected pesticides in carrots and vegetables, almost no overlap between the EU and China occurred, with pesticides detected in China, often being banned in the EU. However, in all countries, residues of banned pesticides were detected in carrots and vegetables. It was hypothesised, that more residues will be found in China compared to countries in the EU due to usage of more toxic substances, higher application rates and differences in food safety regulations, which cannot be confirmed here. However, pesticide residues detected in China were more toxic compared to residues detected in the EU.

Fungicides were the most detected class in carrot fields in Norway, with cyprodinil and fludioxonil showing slower degradation under Norwegian conditions compared to values reported in the EU. The remaining sprayed pesticides were in the range of reported DT50 values or below, highlighting the influence not only of climate but also of the crop planted. Long-term residues of imidacloprid, thiacloprid and azoxystrobin with DT50 values over 1000 years were reported and suggest a long persistency of pesticide residues under Norwegian conditions. Therefore, the second hypothesis was partly validated due to faster dissipation of boscalid at HØ, but other fungicides (fludioxonil, cyprodinil) showed a slower dissipation.

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Appendix

Summary of appendixes

No.	Subject
1	Table of residue levels of carrot samples analysed in Norway from 2008-2018
2	Table of residue levels in carrot samples analysed in Germany from 2013-2018
3	Table of pesticides detected in carrots analysed in Germany (2013-2018)
4	Detected pesticides in China
5	Average concentration of pesticides detected at HV per sampling date
6	Average concentrations of pesticide residues per sampling date at HØ.

APPENDIX 1: Carrots analysed in Norway with and without pesticide residues from 2008 to 2018 (Mattilsynet, 2019)

	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	average
Samples from Norway [%]	91.8	87.3	89.6	85.3	80.3	73.6	84.1	78.0	78.6	84.6	73.3	82.4
Samples that were imported [%]	8.2	12.7	10.4	14.7	19.7	26.4	15.9	22.0	21.4	15.4	26.7	17.6
Total carrots without residues [%]	64.7	56.3	53.7	52.9	54.1	50.9	41.3	44	42.9	44.2	43.3	49.9
Samples without residues (Norway) [%]	62.8	50	56.7	50	46.9	56.4	37.7	38.5	39.4	47.7	40.9	47.9
Samples without residues (imported) [%]	85.7	100	28.6	70	83.3	35.7	60	63.6	55.6	25	50	59.8
Total with residues [%]	35.3	43.7	46.3	47.1	45.9	49.1	58.7	56	57.1	55.8	56.7	50.1
Samples with residues (Norway) [%]	37.2	50	43.3	50	53.1	43.6	62.3	61.5	60.6	52.3	59.1	52.1
Samples with residues (imported) [%]	14.3	0	71.4	30	16.7	64.3	40	36.4	44.4	75	50	40.2

APPENDIX 2: Carrot samples analysed in Germany from 2013-2018 (BVL, 2020b)

	2013	2014	2015	2016	2017	2018	average
Samples from Germany [%]	68.06	69.22	62.93	73.76	72.21	69.68	69.31
Samples that were imported [%]	31.94	30.78	37.07	26.24	27.79	30.32	30.69
Total carrots without residues [%]	56.65	53.92	49.14	49.17	43.10	51.06	50.51
Samples without residues (Germany) [%]	56.42	49.71	45.21	45.69	39.01	44.27	46.72
Samples without residues (imported) [%]	57.14	63.40	55.81	58.95	53.74	66.67	59.29
Total with residues [%]	43.35	46.08	50.86	50.83	56.90	47.87	49.31
Samples with residues (Germany) [%]	43.58	50.29	54.79	54.31	60.99	55.73	53.28
Samples with residues (imported) [%]	42.86	36.60	44.19	41.05	46.26	29.82	40.13
Total samples >MRL [%]	0.38	0.60	0.00	0.00	0.38	0.00	0.23
Samples >MRL (Germany) [%]	0.00	0.87	0.00	0.00	0.52	0.00	0.23
Samples >MRL (imported) [%]	1.19	0.00	0.00	0.00	0.00	0.00	0.20

APPENDIX 3: Pesticides that had residues in carrots analysed in Germany (BVL, 2020b)

Pesticide	Frequency in [%]	Class
2,6-Dichlorbenzamide	0.3	H
4-Hydroxychlorothalonil; 4-Hydroxy-2,5,6-trichlorisophthalonitril	0.1	F
Acetamiprid	0.0	I
Aclonifen	1.4	H
Azoxystrobin	11.8	F
Benzalkonium chloride	0.1	H
Bifenthrin	0.04	I
Boscalid	18.2	F
Bromid ions	0.9	
Bupirimate	0.4	F
Captan	0.04	F
Carboxin	0.04	F
Chlorantraniliprole	0.04	I
Chlorate	1.6	H
Chloridazondesphenyl; 5-Amino-4-chlor-2,3-dihydro-3-oxo-pyridazin	0.1	H
Chlorpropham	0.2	H, G
Chlorpyrifos	0.5	I
Chlorpyrifos-methyl	0.04	I
Chlorothalonil	0.04	F
Clomazone	0.5	H
Clothianidin	0.3	I
Cyproconazole	0.04	F
Cyprodinil	0.4	F
Cyromazine	0.04	I
DDT, sum DDT, DDE, DDD	0.5	I
Deltamethrin	0.04	I
Dicofol	0.04	A
Didecyldimethylammoniumchlorid (DDAC-C10)	0.2	F
Dieldrin, sum dieldrin and aldrin	0.3	I
Difenoconazole	11.3	F
Diflufenican	0.04	H
Dimethoate	0.5	I
Dimethomorph	0.2	F
Diniconazole	0.04	F
Endosulfan	0.1	I
Epoxiconazole	0.1	F
Fenbuconazole	0.04	F
Fenbutatin oxide	0.04	A
Fenpropidin	0.1	F
Fenpropimorph	0.04	F
Fipronil	0.1	I
Fipronil-desulfinyl	0.04	M

Fluazifop	0.2	H
Fludioxonil	0.7	F
Fluopicolide	1.0	F
Fluopyram	5.3	F
Flurochloridone	0.04	H
Flutriafol	0.1	F
Fluxapyroxad	0.1	F
Fosetyl	0.3	F
Glyphosate	0.1	H
Haloxifop	0.2	H
HCH	0.1	
Imidacloprid	0.2	I
Iprodione	1.2	F
Isopyrazam	0.04	F
Copper	8.8	F
Lambda-cyhalothrin	0.1	I
Linuron	2.7	H
Metalaxyl and Metalaxyl M	0.3	F
Methiocarb	0.04	I
Methoxyfenozide	0.04	I
Metribuzin	0.3	H
Molinate	0.1	H
Omethoate	0.1	I
Pendimethalin	7.9	H
Phosphorous acid	0.2	
Pirimicarb	0.04	I
pp-DDE	0.04	M
Prochloraz	0.04	F
Procymidone	0.04	F
Propamocarb	0.13	F
Propiconazole	0.04	F
Prosulfocarb	2.1	H
Prothioconazole-desthio	1.5	F
Pyraclostrobin	2.7	F
Pyrimethanil	0.3	F
Quicksilver Hg	0.04	
Quintozene	0.1	F
Quizalofop	0.2	H
Spinosad	0.04	I
Tebuconazole	8.2	F
Tefluthrin	0.1	I
Tepraloxydim	0.3	H
Terbutylazin-desethyl	0.04	M
Thiabendazole	0.04	F
Thiacloprid	0.04	I

Thiamethoxam	0.3	I
Triadimefon and Triadimenol	0.2	F
Triazolyl-Alanine	1.1	M
Triazolyl acetic acid	0.04	M
Triazolyl lactic acid	0.2	M
Trifloxystrobin	0.8	F
Trifloxysulfuron	0.04	H
Triticonazole	0.04	F

APPENDIX 4: Detected pesticides in vegetable samples from China

Chen et al., 2011	Liu et al., 2020	Qin et al., 2015	Wang et al., 2013	Wu et al., 2017
Acephate	Acetamiprid (17.7 %)	Acephate	Acephate	Acetamiprid (4.67 %)
Bifenthrin	Carbendazim (29.3 %)	Bifenthrin	Chlorpyrifos	Carbofuran (3.80 %)
Chlorothalonil	Dimethomorph (18.3 %)	Chlorpyrifos	Dichlorvos	Chlorothalonil (4.13 %)
Chlorpyrifos	Imidacloprid (11.7 %)	Cyfluthrin	Dimethoate	Cyhalothrin (2.94 %)
Cyfluthrin	Metalaxyl (10.1 %)	Cyhalothrin	Disulfoton	DDT (2.72 %)
Cyhalothrin		Cypermethrin	Ethoprophos	HCB (4.16 %)
Cypermethrin		Dichlorvos	Methidathion	Imidacloprid (3.43 %)
Deltamethrin		Fenpropathrin	Omethoate	Procymidone (6.43 %)
Dichlorvos		Fenvalerate	Phorate	Pyridaben (3.26 %)
Dimethoate		Isocarbophos	Tolclofos-methyl	Pyrimethanil (4.15 %)
Fenitrothion		Omethoate		
Fenpropathrin		Parathion		
Fenvalerate		Parathion-methyl		
Isocarbophos		Permethrin		
Methamidophos		Phorate		
Omethoate		Profenofos		
Parathion		Triazophos		
Parathion-methyl				
Phorate				
Triadimefon				
Triazophos				

APPENDIX 5: Average concentration of three measurements per sampling date of pesticides detected at HV

	Azo.	Bosc.	Bosc met	Cypro.	Cypro. met.	Flu.	Imida.	Metri.	Metri. met	Penco.	Pyracl.	Pyrid.	Thiacl.	Thiacl. met	average	sum
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
03.05.19	1.227	10.938	6.744	0.738	3.822	2.230	4.286	0.261	0.062	2.010	0.240	2.321	0.253	5.562	14.159	40.69
04.06.19	1.248	10.270	5.949	0.660	3.218	2.156	4.636	33.822	0.746	1.342	0.174	1.719	0.340	7.054	4.889	73.34
18.06.19	0.997	8.867	4.608	0.466	2.554	4.655	4.573	7.223	0.989	1.691	0.111	1.516	0.347	7.344	37.007	45.94
12.07.19	0.915	8.254	5.002	0.445	2.425	2.158	4.560	11.137	1.904	1.429	0.061	1.466	0.288	6.976	18.900	47.02
30.08.19	0.976	9.125	5.311	0.496	2.327	2.480	4.354	1.060	0.231	1.025	0.043	1.328	0.223	5.713	38.396	34.69
05.09.19	1.349	8.925	5.911	0.551	3.089	1.917	4.347	1.905	0.263	0.737	0.095	1.340	0.246	5.613	2.419	36.29
26.09.19	0.972	10.465	6.131	0.537	3.200	2.136	4.724	0.786	0.167	1.309	0.088	1.382	0.260	6.232	39.053	38.39
25.10.19	1.000	10.167	6.174	0.716	2.613	2.933	5.066	1.158	0.295	0.946	0.052	1.650	0.268	5.317	2.557	38.36
16.04.19	0.951	9.754	4.985	0.649	2.556	2.168	3.342	0.506	0.000	0.239	0.000	1.023	0.195	4.127	2.033	30.50
average	1.071	9.641	5.646	0.584	2.867	2.537	4.432	6.429	0.517	1.192	0.096	1.527	0.269	5.993		

APPENDIX 6: Average concentrations of three measurements per sampling date of pesticide residues at HØ

	Bosc.	Bosc met	Cypr.	Cypro. met.	Flu.	Imida.	Metri.	Metri. met	Penco.	Pyracl.	Pyrid.	Thiacl.	Thiacl. met	average	sum
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
21.05.19	20.403	9.662	0.242	0.796	0.841	1.686	0.029	0.000	0.660	0.000	1.386	0.172	3.486	3.028	39.36
04.06.19	18.178	4.742	0.000	0.520	0.488	7.613	29.669	0.863	0.000	0.000	1.754	0.359	3.439	4.650	67.62
18.06.19	10.204	4.873	0.000	0.264	0.429	2.787	11.363	0.915	0.000	0.000	1.315	0.210	2.964	2.717	35.32
12.07.19	10.138	4.020	0.000	0.401	0.357	2.450	20.725	1.874	0.000	0.000	1.203	0.083	3.024	3.406	44.27
30.08.19	175.808	6.779	0.000	0.266	0.143	1.785	0.895	0.191	0.000	29.027	0.914	0.043	2.165	16.770	218.02
05.09.19	166.047	8.070	216.731	1.245	149.052	3.265	1.126	0.260	0.000	25.875	1.564	0.666	3.574	44.421	577.47
26.09.19	143.378	6.480	138.063	5.887	112.940	1.826	0.686	0.205	0.000	16.111	0.689	0.225	2.087	32.967	428.58
25.10.19	153.094	4.438	141.132	9.722	139.699	1.956	0.653	0.138	0.000	18.122	0.790	0.086	1.948	36.290	471.78
16.04.19	35.414	0.000	8.091	2.249	14.005	1.746	0.000	0.000	0.000	3.618	0.800	0.000	1.710	5.203	67.63
average	80.61	5.451	56.029	2.372	46.439	2.790	7.238	0.494	0.073	10.306	1.157	0.205	2.711		



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