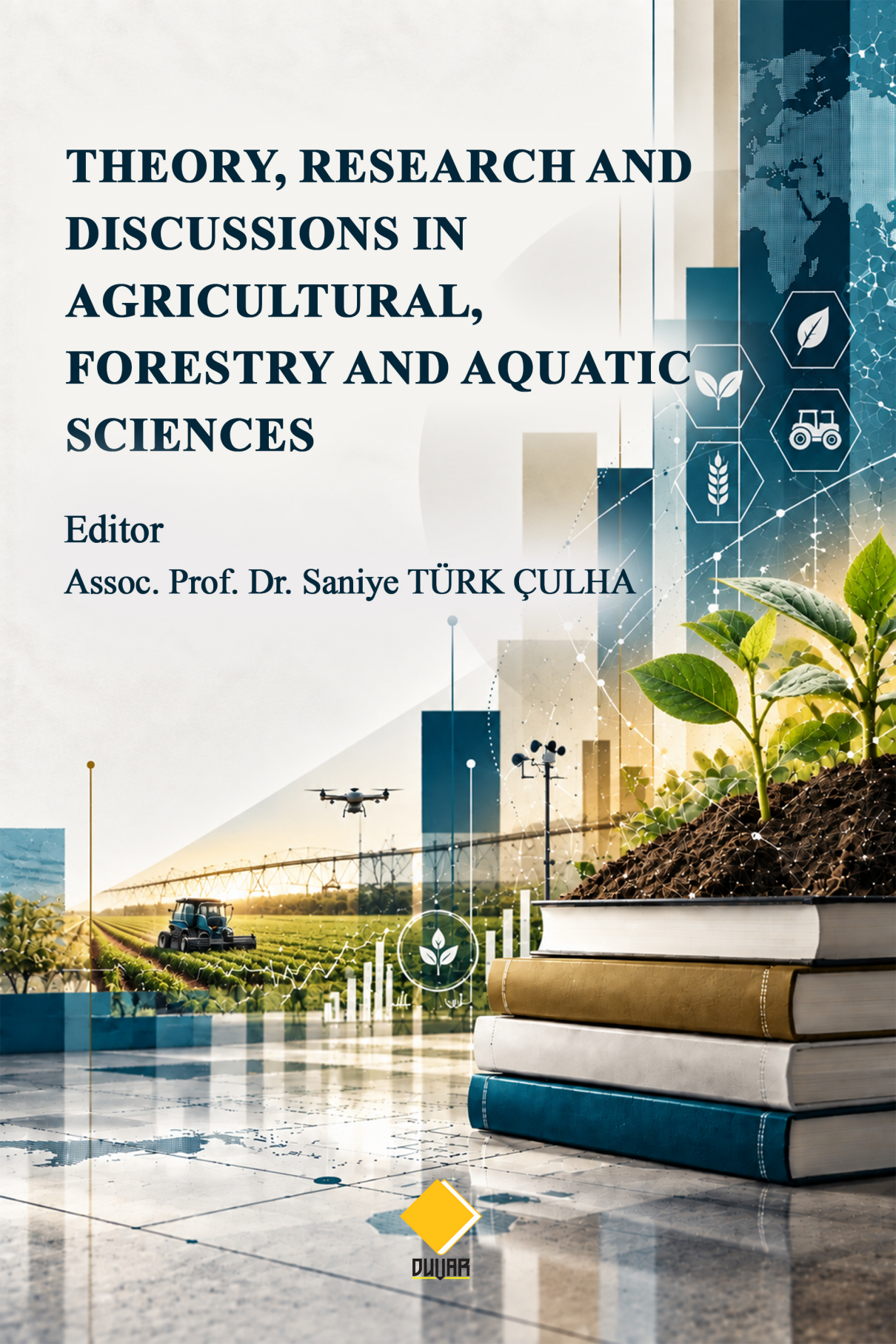


THEORY, RESEARCH AND DISCUSSIONS IN AGRICULTURAL, FORESTRY AND AQUATIC SCIENCES

Editor

Assoc. Prof. Dr. Saniye TRK ULHA



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***Theory, Research and Discussions in Agricultural, Forestry and
Aquatic Sciences***

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Assoc. Prof. Dr. Saniye TRK ULHA

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853 Sokak No:13 P.10 Kemeraltı-Konak/İzmir

Tel: 0 232 484 88 68

www.duvar yayinlari.com

duvarkitabevi@gmail.com

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Chapter 1

WOOD ODOR: Chemistry and Characterization Techniques

Ayben KILIÇ PEKGÖZLÜ¹

Introduction

Wood has been one of the most versatile renewable materials used by humans for thousands of years due to its mechanical properties, aesthetic appearance, and sustainability. Beyond its structural performance, wood possesses a distinctive natural odor that significantly contributes to its sensory quality and influences consumer perceptions. The characteristic aroma of wood is often associated with freshness, naturalness, and authenticity, making odor an important quality attribute in furniture, interior construction, flooring, packaging, musical instruments, and specialty wood products.

Wood odor arises from a mixture of volatile organic compounds (VOCs), which are largely derived from wood extractives and, to a lesser extent, from the thermal or biological degradation of structural polymers such as cellulose, hemicelluloses, and lignin (Schreiner et al., 2017; Rabinovich et al., 2004). Their composition and relative abundance vary considerably depending on numerous factors, including tree species, geographic origin, growth conditions, wood anatomy, age, storage conditions, drying processes, heat treatment, microbial degradation, and finishing materials (Adamová et al., 2020; Wang et al., 2026).

The development of modern analytical techniques has substantially improved our understanding of wood odor chemistry. Headspace solid-phase microextraction (HS-SPME) and thermal desorption (TD), particularly when coupled with gas chromatography–mass spectrometry (GC–MS), provide sensitive approaches for the extraction, separation, identification, and quantification of volatile compounds in complex wood matrices (Schierer et al., 2023; Wei et al., 2023). More advanced techniques, including gas chromatography–olfactometry (GC–O), GC–MS/O, and comprehensive two-dimensional gas chromatography (GC×GC–MS/O), further enable the identification of odor-active compounds and establish a direct link between chemical composition and human sensory perception (Ghadiriasli et al., 2018; Mahmoud and Zhang, 2024). Electronic nose systems complement these chromatographic approaches by generating characteristic odor fingerprints from

¹ Prof.Dr., Bartın Üniversitesi, Bartın Orman Fakültesi, Türkiye. ORCID:0000-0002-3640-6198.

sensor-array responses, thereby enabling rapid discrimination and classification of samples according to differences in their overall volatile profiles.

In this review it was aimed to discuss the wood odor with its chemical origins, major odor-active compounds, factors influencing odor formation and analytical methodologies used for characterization.

2. Chemical Constituents Responsible for Wood Odor

Wood odor arises from a complex mixture of volatile organic compounds, predominantly originating from low-molecular-weight extractives naturally present in wood. These volatile constituents comprise a wide range of chemical classes, including terpenes and terpenoids, aldehydes, ketones, alcohols, organic acids, phenolic compounds, and other oxygenated compounds. In particular, terpenes are strongly associated with resinous components, and mono-, sesqui-, and diterpenes constitute major volatile constituents of coniferous woods, whereas deciduous species generally exhibit different extractive and VOC profiles (Adamová et al., 2020).

The characteristic odor of wood, however, cannot be attributed to a single compound or chemical class but rather results from the combined contributions of multiple VOCs that differ in concentration, volatility, and sensory relevance. Their composition is influenced not only by species but also by factors such as heartwood–sapwood distribution, tree age, growth location, and subsequent technological processing. For example, pine emissions are typically characterized by terpenes such as α -pinene, β -pinene, Δ^3 -carene, and limonene, however hardwoods such as oak and beech may release comparatively larger acetic and formic acids amounts and low terpenes. Aldehydes, including hexanal, pentanal, nonanal, and benzaldehyde, also constitute an important fraction of wood volatiles (Adamová et al., 2020)

In addition to naturally occurring extractives, some odor-active compounds may be generated through chemical and thermal transformations of the major structural components of wood during drying and processing. For instance, deacetylation of hemicelluloses can result in the formation of acetic acid, while degradation of wood polysaccharides may generate furan and pyran derivatives. Thermal degradation of lignin, on the other hand, primarily produces phenolic odorants such as vanillin, guaiacol, and eugenol, which may contribute substantially to the characteristic aroma of thermally processed wood (Schierer et al., 2023).

2.1. Terpenes

The term “terpene” originates from turpentine oil and was coined by Kekulé to describe the hydrocarbons ($C_{10}H_{16}$) occurring naturally in this resin-derived product. Terpenes and terpenoids are the largest and most structurally diverse classes of natural products, with tens of thousands of compounds identified to date. Terpenes are commonly classified according to the number of isoprene (C_5) units incorporated into their carbon skeleton, giving rise to several major groups, including monoterpenes (C_{10}), sesquiterpenes (C_{15}), diterpenes (C_{20}) and triterpenes (C_{30}) (Rowe, 1989). Among these groups, **monoterpenes and sesquiterpenes** are particularly relevant to wood odor because of their comparatively high volatility and widespread occurrence in wood extractives and resinous parts.

Terpenes constitute a major fraction of the volatile extractives of many wood species and are particularly abundant in coniferous woods. In softwoods, they are closely associated with oleoresin stored in resin canals and related secretory structures, from which volatile terpenes can readily be released into the surrounding atmosphere. **α -pinene** is among the most prominent representatives and occurs at high concentrations in the oleoresin of many coniferous species. In incense cedar, for example, α -pinene was identified as an odor-active constituent with a characteristic **woody and resinous** odor and exhibited a high flavor dilution factor, indicating its substantial sensory relevance to the odor of untreated wood (Schreiner et al., 2017). Among the monoterpenes commonly associated with wood, **α -pinene** is generally characterized by resinous or woody notes, while **β -pinene** has terpene- and eucalyptus-like characteristics. **β -Myrcene** contributes earthy, green, and geranium-like notes, whereas **limonene** is associated with a characteristic citrus or orange-peel-like odor. Other monoterpenes and oxygenated derivatives further broaden the sensory profile: camphene exhibits green and camphoraceous notes, ocimene is citrus-like, green and fruity, p-cymene possesses woody and herbal characteristics, linalool is floral and fresh, and fenchol is typically described as earthy and musty (Mahmoud and Zhang, 2024).

Terpene abundance alone does not determine its contribution to wood odor. Gas chromatography–olfactometry studies of natural wood have demonstrated that several terpenes are odor-active even when they are not necessarily the quantitatively dominant VOCs. In natural oak wood, numerous mono- and sesquiterpenoid odorants were identified, including **limonene, ocimene, δ -3-carene, camphene, 1,8-cineole, linalool, β -caryophyllene, α -terpineol, borneol, germacrene, α -bisabolol, and other oxygenated terpenoids**. The resulting odor spectrum ranged from resinous and woody to green, citrus-like,

floral, earthy, and camphoraceous notes, illustrating the important role of terpene diversity in shaping the complex sensory character of wood (Ghadiriasli et al., 2018).

2.2. Aldehydes and Ketones

Aldehydes and ketones are carbonyl-containing organic compounds characterized by the presence of a C=O functional group. In aldehydes, the carbonyl carbon is bonded to at least one hydrogen atom, whereas in ketones it is bonded to two carbon-containing substituents, which may be alkyl or aryl groups (Noller, 1965).

Aldehydes encompass chemically diverse groups, including aliphatic, aromatic, and terpenoid derivatives, each of which may impart distinct odor characteristics. For example, hexanal, octanal, and decanal have been associated with fresh apple, citrus, and orange-peel-like notes, respectively, whereas benzaldehyde is characterized by a distinctive bitter almond-like aroma. Terpenoid aldehydes, such as citral, may further contribute citrus- or lemon-like notes. Thus, even within a single chemical class, aldehydes can produce markedly different sensory impressions depending on their molecular structure (Catalano et al., 2024).

In wood, aldehydes may occur as naturally present volatile constituents but can also arise as secondary products of oxidative and thermal transformations. Of particular relevance is the formation of aliphatic aldehydes via heat-induced oxidation of fatty acids, especially unsaturated ones. This pathway can generate saturated aldehydes such as **hexanal, heptanal, and decanal**, as well as unsaturated derivatives including hexenal, heptenal, and decenal. Consequently, changes in wood during drying, heating, storage, or oxidative aging may alter the aldehyde composition, thereby modifying its overall odor profile (Catalano et al., 2024).

Ketones may exhibit fruity, floral, camphoraceous, earthy, or woody odor characteristics. Methyl ketones, for example, may display fruity notes similar to those of the corresponding acetate esters, whereas cyclic ketones are frequently associated with more specific odor characteristics, including camphoraceous and floral notes. The contribution of ketones to odor may also depend strongly on the three-dimensional environment of the carbonyl group. Studies on bicyclic ketones have demonstrated that changes in steric shielding around the functional group can shift the odor character from strongly camphoraceous toward more balanced notes of woody, earthy, and camphoraceous character (Rossiter, 1996). Ketones have also been identified among the odor-active constituents of natural wood. For example, 1-octen-3-one was detected as a fatty-acid degradation

product in natural oak wood, demonstrating that oxidative degradation of wood lipids may contribute not only aldehydes but also odor-active ketones to the volatile profile (Ghadiriasli et al., 2018).

2.3. Alcohols

Alcohols constitute another diverse group of volatile compounds contributing to the overall odor profile of wood. Their sensory characteristics vary considerably depending on molecular structure and may range from green, fresh, and floral to citrus-like, earthy, musty, and woody notes. In particular, several oxygenated monoterpenes containing hydroxyl groups have been identified as potent odor-active constituents of natural wood. In oak wood, **nerol, citronellol, α -terpineol, geraniol, linalool, fenchol, and borneol** were detected among the odor-active compounds; nerol and geraniol were associated with citrusy and floral notes, citronellol with a lemon-like odor, and α -terpineol with musty, citrusy, and fruity characteristics. In contrast, borneol exhibited an earthy, moldy, and woody odor, illustrating the broad sensory diversity of alcohol-containing wood volatiles. The odor characteristics of aliphatic alcohols are likewise strongly influenced by molecular structure and unsaturation. For example, *cis*-3-hexen-1-ol, commonly known as *leaf alcohol*, is characterized by a pronounced green odor, while changes in the position and configuration of the double bond in hexenols can shift their sensory character from fresh, fruity, and grassy-green toward fatty, oily, or herbal notes. Thus, although alcohols may not always represent the quantitatively dominant VOC class in wood, specific alcohols can contribute significantly to its fresh, green, floral, citrus-like, earthy, and woody odor nuances (Ghadiriasli et al., 2018).

2.4. Phenolic compounds

Volatile phenolic compounds represent an important group of odor-active constituents characterized by an aromatic ring bearing one or more oxygen-containing substituents. Their contribution to aroma depends strongly on molecular volatility; highly hydroxylated polyphenols generally exhibit limited volatility and therefore contribute little directly to odor, whereas lower-molecular-weight volatile phenols may exert a pronounced sensory effect. Among the most prominent odor-active phenolic compounds are **guaiacol, 4-vinylguaiacol, vanillin, eugenol, isoeugenol, cresols, and syringol-type derivatives**. In broader aroma studies, guaiacol, vanillin, and 4-vinylguaiacol have been identified among the most recurrent odor-active phenolic compounds, illustrating the substantial contribution of relatively simple methoxylated phenols to characteristic aroma profiles (Schieber and Wüst, 2020).

For wood odor, volatile phenols are particularly relevant because they may originate from both naturally occurring phenylpropanoid-related pathways and the thermal transformation of structural and extractive components. The biosynthetic pathway leading to volatile phenylpropanoids shares intermediates with lignin biosynthesis, including coumaryl- and coniferyl-alcohol-related pathways. Moreover, thermal treatment can generate volatile phenols from labile precursors; importantly for wood, pyrolysis has been reported to produce a variety of volatile phenols that contribute to characteristic aromatic and smoky properties. Compounds such as guaiacol and related methoxyphenols are therefore especially significant in thermally treated, toasted, or degraded wood matrices (Schieber and Wüst, 2020).

The sensory contribution of individual phenolic compounds may be substantial even when they occur at relatively low concentrations. Guaiacol and 4-vinylguaiacol are commonly associated with phenolic, clove-like, and smoky notes, whereas vanillin contributes characteristic vanilla-like and sweet aromas (Angeloni et al., 2020).

2.5. Organic acids

Compared with other plant organs, relatively few organic acids have been reported in woody tissues such as bark and heartwood. Greater diversity occurs in leaves, fruits, and other non-woody tissues. Nevertheless, organic acids can be important odor-active constituents of wood, among which acetic acid is particularly relevant for its characteristic vinegar-like odor and high odor potency. In natural oak wood, acetic acid has been identified as one of the most potent odorants. Its presence in wood is also associated with hydrolytic and thermal reactions involving acetyl groups in hemicelluloses, and its release may therefore increase during wood processing (Fernandez de Simon et al., 2009). Other organic acids, including butanoic acid, 3-methylbutanoic acid, decanoic acid, and 3-phenylpropanoic acid, may contribute cheesy, rancid, animalic, fatty, or waxy notes to the overall odor profile. Thus, despite their relatively limited diversity and abundance in woody tissues, organic acids can make an important contribution to characteristic wood odor because of their distinctive sensory properties and, in some cases, high odor potency (Ghadiriasli et al., 2018).

3. Factors Affecting Wood Odor

Wood species represents a major source of variation in odor composition because individual species possess distinct extractive profiles and consequently emit different combinations of odor-active volatile compounds. Comparative studies of different wood species have demonstrated that both the identity and

relative importance of key odorants vary considerably among species, supporting the use of volatile odor profiles as species-specific sensory fingerprints (Wang et al., 2018).

3.1 Geographical Origin and Tree Species

Tree species are one of the primary determinants of wood odor because both the amount and composition of volatile extractives differ substantially among species. In general, resin-rich softwoods such as pine and spruce are characterized by relatively high emissions of monoterpenes and sesquiterpenes, whereas hardwoods generally exhibit lower terpene emissions and a greater contribution from oxygenated compounds such as aldehydes and organic acids. Considerable variation may also occur among individual species within these broad groups. For example, *Cinnamomum camphora* is characterized by **linalool** and **safrrole**, while **α -pinene** is particularly abundant in *Pinus massoniana* and contributes to its characteristic resinous and turpentine-like odor. In contrast, **α -cedrene** and **β -cadinene** predominate in the heartwood of *Cunninghamia lanceolata*, whereas **δ -cadinene** and **α -muurolene** are major constituents of *Eucalyptus* heartwood. *Populus cathayana*, on the other hand, contains comparatively low levels of VOCs in both heartwood and sapwood. Such interspecific differences demonstrate that the characteristic odor of a wood species reflects not simply its total VOC concentration, but rather its specific combination of volatile constituents and their individual odor potencies. Similar species-dependent variation has been reported among oak, chestnut, acacia, ash, cherry, and cedar woods, where aldehydes, lactones, phenolic compounds, terpenes, and other odorants contribute differently to the resulting sensory profiles (Schreiner et al., 2017).

Tree growth locality, together with genetic background, tree age, environmental pollution, and nutrient conditions, has been identified as a factor affecting the composition and emission of wood volatiles (Adamová et al. 2020). Nevertheless, available evidence indicates that genetic and anatomical differences may sometimes exert a stronger influence than growth conditions; particularly in softwoods, substantial differences in **α -pinene** and **Δ^3 -carene** emissions have been observed even among individuals of the same species, with inter-tree variability being attributed largely to genetic factors (Pohleven et al., 2019). Geographic patterns may also partly reflect broader climatic adaptations and associated differences in extractive chemistry. Tropical woods, for example, generally contain a wider spectrum and higher amounts of extractives than woods originating from temperate regions, while differences have also been reported between closely related taxa of different geographic origins, such as Siberian and

European larch. Thus, tree species provides the fundamental chemical framework of wood odor, whereas geographic origin and local growth conditions can further modify this species-specific VOC signature (Adamová et al., 2020).

3.2. Drying

Drying is an important factor affecting wood odor because changes in moisture content and drying temperature can substantially modify both the quantity and composition of emitted volatile organic compounds. During drying, native VOCs may be lost through evaporation, while hydrolytic, oxidative, and thermally induced reactions can simultaneously generate new volatile compounds. Consequently, the odor profile of dried wood depends not only on the final moisture content but also on the drying method and temperature applied. This effect was clearly demonstrated in sugi (*Cryptomeria japonica*), where air-dried, conventionally dried, and high-temperature-dried wood exhibited distinct VOC profiles. Sesquiterpenes were predominant in all treatments, with δ -cadinene, α -muurolene, and β -cadinene among the major compounds, whereas monoterpenes such as α -pinene and D-limonene were more characteristic of air- and conventionally dried samples. High-temperature drying markedly reduced monoterpene emissions, consistent with the loss of these relatively volatile compounds during intensive heating (Ohira et al., 2009).

In addition to removing native volatile constituents, elevated drying temperatures may promote the formation of secondary odor-active compounds through the degradation of wood polymers. A particularly important example is **acetic acid**, which was detected in high-temperature-dried sugi but not in air-dried wood. Its formation was attributed to the hydrolysis of acetyl groups in hemicelluloses, especially xylan, under elevated-temperature conditions. Although its concentration was not particularly high, acetic acid has a low odor threshold and an unpleasant odor, demonstrating that drying-induced changes in wood odor cannot be evaluated solely from total VOC concentrations. The sensory results supported this chemical differentiation: odors from air-dried and conventionally dried sugi were perceived as significantly more soothing than those from high-temperature-dried wood, although the perceived odor intensity of all three treatments remained approximately moderate (Ohira et al., 2009). Similar sensory consequences have been observed for hinoki cypress (*Chamaecyparis obtusa*), where air-dried wood was perceived as significantly more natural than high-temperature-dried material and produced a greater reduction in prefrontal cortex activity during olfactory stimulation (Ikei et al., 2015).

Moisture content itself is another important determinant of odor release during drying. In paper mulberry (*Broussonetia papyrifera*), GC–MS/O analysis showed that decreasing moisture content generally reduced both TVOC emissions and total odor intensity in heartwood and sapwood, with particularly pronounced changes as moisture content decreased from 70 to 50% and from approximately the fiber saturation region (30%) toward 10%. The response was also tissue-dependent: sapwood consistently exhibited higher VOC concentrations and odor intensity than heartwood at comparable moisture contents. Thus, drying modifies wood odor through two interacting mechanisms—the progressive reduction in moisture-controlled VOC release and the temperature-dependent loss or formation of specific odor-active compounds (Wang et al., 2021).

3.3. Heat Treatment

Heat treatment can markedly alter wood odor because it simultaneously promotes the loss of native volatile compounds and the formation of new secondary VOCs via thermal degradation. This effect is particularly evident in terpene-rich softwoods. In Scots pine, for example, heat treatment at approximately 230 °C caused a pronounced reduction in total VOC emissions and shifted the volatile profile from a terpene-dominated profile to one dominated by aldehydes, carboxylic acids, and ketones. **α -Pinene, 3-carene, and hexanal**, which were the major constituents of air-dried wood, decreased substantially after treatment, whereas **furfural, acetic acid, and 2-propanone** became the predominant emissions. Similar trends have been reported more broadly for thermally modified softwoods, where volatile terpenes and hexanal decrease while acetic acid and furfural increase with increasing treatment severity (Manninen et al., 2002; Pohleven et al., 2019).

The formation of these secondary odor-active compounds is closely related to the thermal degradation of structural wood polymers. Hemicelluloses are particularly heat-sensitive, and their deacetylation releases acetic acid, while further degradation of pentoses and hexoses generates **furfural, 5-methylfurfural, and hydroxymethylfurfural**. Cellulose degradation can also produce furan derivatives, whereas thermal breakdown of lignin leads to aromatic and phenolic compounds, including **phenol, vanillin, and guaiacylacetone** under more severe treatment conditions. Consequently, heat treatment does not merely reduce VOC emissions; it can fundamentally change their chemical origin and sensory character. The magnitude of this transformation is species-dependent: total VOC emissions generally decline in softwoods because native terpenes are volatilized, whereas hardwoods may show increased emissions

because heat accelerates the formation of secondary degradation products (Peters et al., 2008; Pohleven et al., 2019).

The odor outcome of thermal modification also depends on treatment temperature, duration, and heating medium. Studies on *Afrormosia* and *Newtonia* showed that hexanal and acetic acid were among the principal contributors to unpleasant odor, and their reduction was influenced differently by temperature and treatment medium; palm-oil treatment was more effective than air treatment in reducing several unpleasant-smelling VOCs (Bessala et al., 2024). Likewise, sensory evaluation of thermally modified bamboo demonstrated that untreated material was generally perceived as more soothing than heat-treated samples and that odor differences associated with temperature, duration, and heating medium could be clearly distinguished (Sun et al., 2018). Overall, heat treatment should therefore be regarded as a major factor in reshaping wood odor, balancing the volatilization of native extractives and the generation of thermally derived odorants.

3.4. Aging

Aging is the gradual physical and chemical transformations that occur in wood over time by the environmental factors such as oxygen, light, temperature, and moisture, resulting in changes in its chemical composition and volatile organic compound (VOC) profile (Sonderegger et al., 2015). These changes arise from several concurrent processes, including the gradual loss of highly volatile constituents, oxidation and transformation of existing compounds, and, when wood is in contact with another matrix, the progressive extraction of wood-derived odorants. Accelerated aging experiments on thermally modified Norway spruce clearly demonstrate this dynamic behavior. Exposure to UV radiation, moisture, temperature, and oxygen promotes photochemical and oxidative reactions that modify both the quantity and composition of emitted VOCs. After 600 h of xenon aging, for example, the total VOC emission of spruce treated at 160 °C decreased by approximately 42%, while compositional changes included the disappearance of **furfural** and **β -pinene** and the appearance or increased contribution of other compounds such as **tricyclene**, **longifolene**, and **toluene**. These findings indicate that aging should not be regarded simply as a gradual depletion of volatiles; rather, secondary oxidation, isomerization, and degradation reactions continuously reshape the VOC profile of wood (Bubeníková et al., 2026).

The influence of aging is also evident in systems in which wood-derived odorants are progressively transferred to another matrix, as extensively demonstrated in beverages matured in wooden barrels. The extraction of wood

volatiles is generally most rapid during the initial period of wood contact and subsequently slows as aging proceeds. In wines matured in American oak, compounds such as **cis- and trans-whiskylactones, vanillin, syringaldehyde, furfural, 5-methylfurfural, eugenol, and guaiacol** were transferred from wood, with the highest extraction rates generally occurring within the first 4–9 months and decreasing after approximately 12 months (Ortega-Heras et al., 2007). Aging also promotes chemical transformations among volatile constituents. Importantly, oak lactones impart characteristic **woody and coconut-like notes**, and their relative contribution depends on oak species, with French and American oak differing in the predominance of trans- and cis-isomers, respectively (Madrera et al., 2003).

Long-term aging, therefore, produces not only quantitative changes in VOCs but also a pronounced sensory transition toward more complex **woody, vanilla, spicy, smoky, caramel-like, coconut, and nutty notes**. In Jerez vinegar, prolonged aging in oak casks was associated with decreases in several esters, organic acids, and aldehydes, while alcohols, ketones, and lactones increased; simultaneously, fruity characteristics declined and woody, nutty, vanilla, clove, and wine-like attributes became more prominent (Uysal, 2024). Likewise, five-year aging of tsipouro in contact with different woods resulted in the emergence of wood-derived odorants absent from the control, including **guaiacol, cis- and trans-whiskylactones, 4-ethylguaiacol, 4-vinylguaiacol, furaneol, and vanillin derivatives**. These compounds contributed smoky, roasted/spicy, clove-like, caramel, coconut, and vanilla characteristics, while the wood's botanical origin strongly affected their relative sensory impact (Karathanos et al., 2024). Overall, the available studies indicate that aging progressively transforms wood odor through a balance between **volatile loss, oxidation and secondary reactions, and the formation or release of wood-derived odor-active compounds**, making aging time and conditions important determinants of the final odor profile.

3.5. Fungal Degradation

Fungal degradation is an important biological factor affecting wood odor because fungal colonization alters the chemical composition of wood and introduces fungal-derived volatile metabolites. Wood-decaying fungi utilize structural components such as cellulose, hemicelluloses, and lignin via enzymatic and oxidative mechanisms, and the resulting degradation may release compounds originally bound within the wood matrix and generate new volatile products. The extent and nature of these changes depend strongly on the type of decay. Brown-rot fungi preferentially degrade cellulose and hemicelluloses while causing

relatively limited modification of lignin, whereas white-rot fungi can degrade all major cell-wall components, including lignin. Fungal development itself is also influenced by moisture content, oxygen availability, temperature, and pH; therefore, environmental conditions indirectly contribute to changes in the volatile and odor profile of decaying wood (Mariano et al., 2020; Rabinovich et al., 2004).

The modification of wood odor during fungal decay results from a complex combination of wood-derived VOCs and microbial volatile organic compounds (MVOCs) produced by fungal metabolism. These fungal volatiles encompass a chemically diverse range of compounds, including short-chain alcohols and ketones, organic acids, methyl ketones, terpenes, sesquiterpenes, and aromatic secondary metabolites. Lignin degradation is particularly relevant to odor development because ligninolytic fungi possess oxidative enzyme systems—including lignin peroxidases, manganese peroxidases, and laccases—that transform aromatic structures and can promote the formation of odoriferous aromatic metabolites. The diversity and quantity of these metabolites vary with fungal species and their enzymatic systems. Accordingly, the odor of decayed wood cannot be attributed solely to decomposition products of the wood itself but reflects the combined contribution of compounds released from the substrate and metabolites synthesized by the colonizing fungi (Rabinovich et al., 2004; Mali et al., 2019).

Experimental studies confirm that fungal species, decay type, fungal interactions, and stage of decomposition can produce distinct VOC patterns. In Norway spruce, Mali et al. (2019) demonstrated that brown- and white-rot fungal combinations produced distinct VOC emission profiles over three months of decomposition, with terpinolene, α -humulene, methyl 3-furoate, and 6-methyl-5-heptenone as signature volatiles associated with fungal activity and interactions. Consistent with these chemical differences, Baietto et al. (2010) showed that the volatile fingerprints of healthy and fungal-decayed wood could be clearly distinguished using electronic nose systems, indicating that fungal degradation produces detectable changes in the overall aroma profile of wood. In living trees, the resulting volatile mixture may be particularly complex because it can contain fungal metabolites, host-tree metabolites, and fungus-induced defensive compounds, including phenolics and terpenoids (Baietto et al., 2010).

3.6. Storage

Storage is an important factor governing both the concentration and composition of volatile compounds released from wood and wood-derived materials. In general, freshly produced or processed wood exhibits relatively high

VOC emissions, which then gradually decline as readily volatile compounds are depleted. However, storage is not simply a passive volatilization process; chemical and biological transformations occurring during this period can generate new odor-active compounds and modify the overall odor profile. In woody biomass, terpenes are particularly important during the early stages of storage, with most being released within the first few weeks. At the same time, oxidation of unsaturated fatty acids promotes the formation of aldehydes and ketones. Consequently, compounds such as **hexanal** may increase after storage begins, reach maximum emission levels after several weeks, and subsequently decline over longer periods. The age and initial condition of the raw material are also important, since pellets produced from aged sawdust may maintain elevated hexanal emissions for considerably longer periods than those produced from fresh material (Alakoski et al., 2016; Rahman et al., 2017).

The temporal evolution of odor during storage, therefore, reflects the different formation and release mechanisms of individual VOC classes. Tagliaferri et al. (2024) reported high odor concentrations during the first days of lignocellulosic biomass storage, associated primarily with the rapid release of light terpenes and oxidation products of fatty acids. Terpene emissions became negligible after approximately 40–50 days, whereas aldehydes and ketones showed high concentrations during the initial period and could increase again after 30–80 days as rising pile temperature accelerated oxidation and mass transfer. **Acetic acid** exhibited a different temporal pattern, reaching maximum concentrations after approximately 60–120 days, which was attributed mainly to hydrolysis of acetyl groups in lignocellulosic components rather than lipid oxidation. Accordingly, odor concentration may show a secondary increase after approximately 50–80 days, then become negligible after prolonged storage of about 100–150 days. These findings demonstrate that changes in wood odor during storage arise not only from progressive depletion of existing volatiles but also from the formation of secondary odorants through oxidation, hydrolysis, and biological processes.

Storage conditions further determine the rate at which these changes occur. Temperature, relative humidity, moisture content, and ventilation influence VOC diffusion, chemical reactions, and microbial activity. Studies on *Fraxinus mandshurica* demonstrate that rising temperatures accelerate VOC release, particularly during the early emission period, whereas higher air exchange rates reduce both VOC concentration and perceived odor intensity by enhancing the removal and dilution of emitted compounds (Wang et al., 2022). More recent work on waterborne-coated *F. mandshurica* similarly showed that temperature accelerates VOC release and shortens the time required to reach equilibrium, whereas relative humidity and ventilation modify both emission kinetics and

sensory odor characteristics (Wang et al., 2026). Thus, storage time and storage environment should be considered together, since aging progressively alters the available pool of wood volatiles while environmental conditions determine their release and transformation rates

4. Sample Preparation Techniques for Odor Analysis

Wood contains a complex mixture of VOCs, including terpenes, aldehydes, ketones, alcohols, esters, phenols, acids, lactones, and other oxygenated compounds, which differ considerably in volatility, polarity, and concentration. Different techniques have been developed according to the physicochemical properties of the target analytes and the objectives of the study

Traditionally, volatile compounds have been isolated using solvent-based extraction methods such as steam distillation (SD), simultaneous distillation–extraction (SDE), solvent-assisted flavor evaporation (SAFE), liquid–liquid extraction (LLE), ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), and supercritical fluid extraction (SFE) (Louw, 2021). Solvent-based methods often require extensive sample preparation, relatively large sample amounts, prolonged extraction times, and significant solvent consumption. In addition, concentration and solvent evaporation steps may result in the loss of highly volatile constituents, thereby altering the original odor profile of wood samples (Mahmoud and Zhang., 2024).

In the last years, utilization of solvent-free or solvent-minimized extraction techniques become more popular due to their simplicity, environmental friendliness, and compatibility with contemporary chromatographic systems. Headspace sampling is one such technique. It enables the collection of volatile compounds directly from the gaseous phase above a sample without direct contact with the sample matrix (Schierer et al., 2023). Headspace techniques are generally classified into two categories: static headspace (SHS) and dynamic headspace (DHS) (Helin et al., 2020, Weit et al., 2023). In static headspace analysis, volatile compounds are allowed to equilibrate between the sample and the headspace within a sealed container before an aliquot of the vapor phase is introduced into the analytical instrument. SHS is simple, reproducible, inexpensive, and requires minimal sample preparation. However, its sensitivity is relatively limited, particularly for trace odorants with low vapor pressures. Dynamic headspace sampling overcomes this limitation by continuously purging the sample with an inert gas, which transports volatile compounds onto an adsorbent material for subsequent thermal desorption or solvent elution.

Among solvent-free extraction methods, headspace solid-phase microextraction (**HS-SPME**) has become the preferred technique for volatile

analysis in wood, food, environmental, and plant science applications. HS-SPME integrates sampling, extraction, concentration, and sample introduction into a single procedure by exposing a coated fused-silica fiber to the headspace above the sample (Díaz-Maroto, et al., 2023). The choice of fiber coating plays a decisive role in determining extraction efficiency and the composition of the detected volatile profile (González-Mas et al., 2019). Polydimethylsiloxane (PDMS), carboxen/polydimethylsiloxane (CAR/PDMS), divinylbenzene/polydimethylsiloxane (DVB/PDMS), and DVB/CAR/PDMS are the fiber types with different polarities and molecular weights (Wei et al., 2023). For semi-volatile constituents at low concentrations, the **SPME Arrow** technology has been demonstrated to be superior in terms of recovery. Likewise, vacuum-assisted HS-SPME has emerged as another promising innovation, enabling improved extraction of semi-volatile compounds while reducing extraction time and increasing analytical sensitivity ((Mahmoud and Zhang, 2024).

Thermal desorption (TD), particularly when coupled with dynamic headspace sampling, represents another highly effective approach for odor analysis. In this technique, volatile compounds are first trapped on sorbent tubes packed with materials such as Tenax TA and subsequently released by controlled thermal desorption directly into the gas chromatograph. Compared with solvent extraction, TD minimizes sample manipulation, eliminates solvent contamination, and provides excellent recovery of highly volatile compounds. Also, it can be fully automated, allowing high-throughput analysis with excellent repeatability (Helin et al., 2020).

5. Instrumental Analysis of Odor Compounds

Instrumental analysis is an important tool in the comprehensive characterization of odor compounds. Among these techniques, gas chromatography–mass spectrometry (GC–MS), gas chromatography–olfactometry (GC–O), multidimensional gas chromatography (GC×GC–MS/O), and electronic nose (E-nose) systems have become the principal analytical tools to characterize complex volatile mixtures (Bax et al., 2020).

5.1. Gas Chromatography–Mass Spectrometry (GC–MS)

Gas chromatography–mass spectrometry (GC–MS) remains the most widely used analytical technique for investigating volatile compounds. They are first separated within the gas chromatographic column according to differences in their volatility and interactions with the stationary phase. Each eluting compound enters the mass spectrometer, where it is ionized, fragmented, and identified by comparison with reference spectral libraries. This combination of

chromatographic separation and molecular identification enables qualitative and quantitative characterization of hundreds of VOCs within a single analysis.

Coupling GC–MS with efficient extraction techniques such as HS-SPME, dynamic headspace, and thermal desorption substantially improves analytical sensitivity while minimizing sample preparation. Nevertheless, despite its outstanding capability for chemical identification, GC–MS alone cannot determine the sensory relevance of detected compounds (Schierer et al., 2023)

5.2. Gas Chromatography–Olfactometry (GC–O)

Gas chromatography–olfactometry (GC–O) integrates chromatographic separation with direct human sensory evaluation. During GC–O analysis, the chromatographic effluent is divided between a conventional detector and an olfactory detection port, where trained assessors continuously monitor the eluting compounds. As each odorant reaches the sniffing port, panelists record its odor quality, perceived intensity, and retention time, thereby directly linking chemical separation with human odor perception (Ghadiriasli et al., 2018;

Several sensory evaluation strategies have been developed to improve GC–O analysis, including Aroma Extract Dilution Analysis (AEDA), Detection Frequency (DF), CharmAnalysis™, and Osme methods. Among these, AEDA is particularly valuable because it estimates the relative odor potency of individual compounds through successive dilution of aroma extracts, whereas detection frequency evaluates the consistency of odor perception among multiple assessors. These complementary approaches enable researchers to prioritize key odorants within highly complex volatile mixtures and distinguish compounds with significant sensory impact from those contributing minimally to the overall aroma profile ((Mahmoud and Zhang, 2024).

Human perception is inherently subjective and may vary among assessors according to physiological, psychological, and environmental factors. In addition, olfactory fatigue, assessor training, and limited reproducibility may influence analytical performance (Fu et al., 2025).

5.3. GC–MS/O and Multidimensional Gas Chromatography

Gas chromatography–olfactometry–mass spectrometry (GC–MS/O) combines simultaneous chemical identification and sensory evaluation by coupling mass spectrometric detection with an olfactory port. This integrated approach enables direct correlation between chromatographic peaks, molecular identities, and odor perception, substantially improving the reliability of odor-active compound identification. Such simultaneous analysis reduces uncertainty associated with matching independently acquired GC–MS and GC–O

chromatograms and has become one of the preferred analytical strategies for complex aroma studies (Wei et al., 2023).

Although GC–MS/O provides significant improvements over conventional GC–MS, highly complex matrices such as wood frequently contain numerous co-eluting compounds that cannot be completely resolved using one-dimensional chromatography. To address this challenge, multidimensional chromatographic techniques, including heart-cut multidimensional gas chromatography (MDGC) and comprehensive two-dimensional gas chromatography (GC×GC), have been developed. These techniques employ two columns with different stationary phases, substantially increasing chromatographic resolution and allowing separation of compounds that would otherwise overlap. Consequently, GC×GC–MS/O enables the identification of trace odorants present at extremely low concentrations while reducing matrix interference and improving confidence in compound assignment. Such multidimensional approaches have become increasingly important for the comprehensive characterization of complex natural matrices containing structurally similar volatile compounds (Louw, 2021).

5.4. Electronic Nose Systems

Electronic nose (E-nose) technology represents an alternative analytical strategy that relies on rapid pattern recognition rather than on the identification of individual volatile compounds. An electronic nose typically consists of an array of partially selective gas sensors, a sampling system, signal acquisition electronics, and chemometric or machine learning algorithms. Instead of generating chromatographic peaks, the sensor array produces characteristic response patterns that collectively represent a sample's aroma fingerprint. These fingerprints are subsequently analyzed using multivariate statistical techniques such as Principal Component Analysis (PCA), Linear Discriminant Analysis (LDA), Partial Least Squares (PLS), Artificial Neural Networks (ANN), Support Vector Machines (SVM), and other machine learning algorithms to classify or discriminate samples according to their odor profiles (Rabehi, et al., 2024).

Compared with chromatographic techniques, electronic noses offer several practical advantages, including rapid analysis, minimal sample preparation, portability, low operational costs, and real-time monitoring capability. These characteristics make E-nose systems particularly attractive for routine quality control, monitoring storage conditions, detecting fungal deterioration, and industrial process control. However, electronic noses do not provide structural identification or quantification of individual volatile compounds. They are generally regarded as complementary tools rather than replacements for chromatographic techniques (Prasad et al., 2022).

Conclusison

Wood odor is a complex and dynamic sensory property arising from the combined contribution of numerous volatile and odor-active compounds whose composition and sensory significance are influenced by both the intrinsic characteristics of wood and subsequent environmental and technological processes. Terpenes, aldehydes, ketones, alcohols, phenolic compounds, and organic acids represent major chemical groups contributing to characteristic wood odors, while factors such as tree species, geographical origin, moisture content, drying, heat treatment, aging, fungal degradation, and storage can substantially modify the volatile profile through the loss, formation, or transformation of these compounds. Advances in sampling and analytical techniques, particularly HS-SPME, thermal desorption, GC-MS, GC-O, GC-MS/O, multidimensional chromatography, and electronic nose systems, have considerably improved the characterization of this chemical and sensory complexity. Nevertheless, because the abundance of a volatile compound does not necessarily reflect its sensory importance, an integrated approach that combines chemical analysis with olfactometric and sensory evaluations is essential for a comprehensive understanding of wood odor.

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Chapter 2

From Detection To Precision Intervention: AI-Based Technologies For Sustainable Disease And Weed Management

Emine KAYA ALTOP¹, Sine KAYA²

1. INTRODUCTION

Crop protection is influenced by the demand for a balanced approach regarding productivity, economic sustainability, and eco-friendliness. Disease and weed control especially suit well the principle of "target-only treatment," because of their natural patchy appearance. Disease outbreaks can start small and then gradually develop, whereas the formation of weed patches is affected by various factors such as soil conditions, previous crops, seed dispersal, watering, and field cultivation. It is, therefore, quite clear that a uniform treatment of the whole field will only lead to excessive inputs at places with light problems and ineffective controls at the more serious infestation areas. Thus, in such situations, one might say that both too much use and little use can be the side effects (Anam et al., 2024; Hu et al., 2024; Peña et al., 2013).

The role of crop scouting is essential, given that diagnosing a plant problem involves understanding the interaction of both bio and environmental factors. Crop type, growth phase, weather, field record, cultivar sensitivity, and farming operations are just some of the factors that can influence symptoms interpretation. However, repeated physical checks become impractical with large farms and dwindling labor. Symptoms are not readily detectable in early plant development, which is another hurdle. A picture taken of a diseased and healthy area at the same time would be an objective, but it is the interpretation of this data that will make a decision for the farmer. Digital pictures of plant problems are a reliable source of records, and by use of AI interpretation tools we may get a quick and correct identification based on the picture. Hence, farmers and advisors need not lose their role but to rather be augmented with the capability to cover vast and heterogeneous farm areas (Ferentinos, 2018; Mohanty et al., 2016).

1 Prof. Dr. Ondokuz Mayıs University, Faculty of Agriculture, Department of Plant Protection, Samsun-TÜRKİYE. kayae@omu.edu.tr, ORCID: 0000-0002-0987-9352

2 PhD. Ondokuz Mayıs University, Postgraduate Education Institute, Plant Protection, Samsun-TURKIYE. sinekayaa@gmail.com, ORCID: 0000-0002-4219-9148

Research using pictures for detecting disease has shown the potential of and the pitfalls of deep learning techniques. Mohanty et al (2016) used a convolutional neural network to learn from 54,306 images showing 14 crops, 26 diseases, and healthy states. They obtained 99.35% correctly identified images on a test without the training set. These results show the technical possibility but the dataset was to large extent under controlled condition and the authors clearly indicated further performance in the real world. Ferentinos (2018) also used his big picture set to classify images at high rates. These works are not remarkable for their accuracy but for the message they bring about the difference between detecting things and the whole process leading to their handling after detection.

Weeds and other plant problems that cannot be distinguished just by looking them at also need attention. The capability of recognizing a weed in a picture is only part of what is needed. To get to a practical decision about what to do with the weeds one also has to know the species or the type where needed, how thick they are, where on the farm, what they are doing to the crops, and what the level of their infestation is that needs action. The final step would be that there is a control method suitable for this particular weed.

Therefore, the whole decision chain needs the detection as well as other activities such as sensing, processing, diagnosis, quantification, mapping, prediction, decision support, intervention, and feedback at the end (Hu et al., 2024; Lottes et al., 2017; Peña et al., 2013).

2. CONVENTIONAL CROP PROTECTION AND THE NEED FOR SPATIAL INTELLIGENCE

2.1 Field Inspection and Visual Diagnosis

Visual inspection provides an important biological baseline because experienced observers integrate symptoms with contextual information. In addition to being visual symptoms of disease or pest infestation, such changes in the looks of the leaves like color change, chlorosis or lack thereof, wilting, lesions, necrosis are also very useful in the determination of the disease. Different problems might result in very similar plant symptom expressions. For example, plants affected by water shortage, nutrient deficiencies, injury due to pests, and diseases will all have varying degrees of overlap of these symptoms. Computer vision can be useful in supporting a disease identification decision but it is never going to replace the skills of a plant pathologist (Ferentinos, 2018; Mohanty et al., 2016).

2.2 Weed Identification

In contrast to modern methods, weed identification has traditionally been based on factors like weeds' physical characteristics, growing plants, past experience of weed infestations at the site, and knowledge of indigenous weeds. Unfortunately, weed and crop species may look alike in color and shape, giving rise to the well-known issue of green-on-green. Around such circumstances, a thorough identification might rely on the combination of spatial, spectral, morphological, and temporal factors rather than just simple color thresholding (Hu et al., 2024).

2.3 Uniform Application and Precision Management

Conventional spraying or spraying chemicals evenly throughout a field is done on the basis of the belief the entire area is the same, therefore suitable for single-type management. Such an idea may, however, result in over-application to areas where control is unnecessary if pests or weeds are not homogenous. Instead of conventional uniformity, precision crop protection is based on the principle of "where and how". Where do you perceive the problem from your field of view? How serious is it? Is there a need to intervene and which way? Do the economic and agronomic conditions allow the intervention? (Lottes et al., 2017; Peña et al., 2013).

3. IMAGING TECHNOLOGIES FOR CROP HEALTH MONITORING

The decision to use a sensor should be made after one understands the biological process in the system rather than the default belief that a more complex sensor is always better. The use of RGB imaging, which is a fairly cheap method and has high spatial resolution, makes it possible for the system to identify problems that are visible to the naked eye, spot the weeds, count the plants, and many other features. This is why RGB imaging is used extensively in robotic applications. The addition of a few bands in multispectral imaging beyond the visible range is a way to get to the vegetation characteristics and stress level. In contrast, hyperspectral imaging captures numerous adjacent, narrow bands and thus can reveal minor spectral differences but also generates huge data and needs more sophisticated calibration and processing. Thermal imaging records long-wave infrared emission released by the surface and hence can provide a good measure of canopy temperature and indicate physiological stress, among others like water stress (Ferentinos, 2018; Lottes et al., 2017; Mohanty et al., 2016; Peña et al., 2013).

In fact, interpretation of spectral data as being specifically related to particular disease needs more than the vegetation-index-based explanation. Vegetation index responses can, indeed, be a signal of disease conditions, the effects of a drought, a shortage of nutrients, pests doing damage, and stress from other sources. A combined use of different senses (multimodal sensing) can help since the sensors provide complementary information which when used, it can lead a stronger conclusion. So for instance, the RGB imagery may show symptoms of the disease on the leaf while the thermal data show signs of a higher canopy temperature. This way, by integrating environmental and time aspects into a single decision-making system, the resultant understanding can be made even more robust (Mishra et al., 2020).

Various imaging techniques such as RGB, multispectral, hyperspectral, and thermal imaging have found increasing utility in plant disease and weed detection systems. Different technologies give unique information about the objects' spectral characters and spatial details and they carry corresponding advantages and limitations. The choice of a particular image acquisition technique has an immediate and strong impact on an artificial intelligence model, both as far as the nature and quantity of its input is concerned and its capacity of execution and the type of device on which it operates. Mainly the plant disease and weed detection imaging techniques, their characteristics such as spectral resolution, advantages and weaknesses, and the kinds of artificial intelligence (AI) methods that they have most been applied are summarized in Table 1.

Table 1. Imaging sensors used for plant disease and weed detection (Lottes et al (2017), Peña et al (2013)).

Imaging technology	Spectral information	Advantages	Main limitations	Typical AI applications
RGB imaging	Visible red, green, and blue bands	Low cost; high spatial resolution; easy deployment on UAVs, tractors, robots, and smartphones	Sensitive to illumination; limited information on subtle pre-symptomatic physiological changes	CNN classification; YOLO-type object detection; U-Net segmentation; transformer-based models

Multispectral imaging	Several discrete visible and near-infrared bands	Adds information beyond human vision; well suited to UAV mapping	Higher cost than RGB; limited spectral resolution compared with hyperspectral systems	Random Forest; SVM; CNN; spectral-index models; multimodal learning
Hyperspectral imaging	Many narrow and contiguous spectral bands	High spectral sensitivity; can capture subtle spectral differences	Large data volume; expensive sensors; demanding preprocessing and computation	Spectral-spatial networks; 3D-CNN; transformer models
Thermal imaging	Long-wave infrared radiation related to surface temperature	Provides information not visible in RGB imagery	Strong sensitivity to environmental and measurement conditions; limited disease specificity	CNN; regression; anomaly detection; multimodal AI
RGB + multispectral	Visible + selected near-infrared/spectral bands	Combines spatial detail with spectral information	Sensor synchronization and data fusion can be challenging	Feature fusion; multimodal CNNs; ensemble learning
RGB + thermal	Visible imagery + canopy temperature	Combines structural and physiological information	Requires careful spatial registration and calibration	Multimodal deep learning; CNN fusion; attention-based models
RGB + multispectral + thermal	Visible, spectral, and thermal information	Rich representation of structural and physiological characteristics	High data complexity, cost, synchronization, and computational requirements	Multimodal neural networks; transformer-based fusion; ensemble models

4. COMPUTER VISION, MACHINE LEARNING, AND DEEP LEARNING

4.1 Image Preprocessing

It's important remembering that pictures taken at the farm usually come from a multitude of changes from lighting conditions, shadows, surrounding vegetation, camera height, camera movement, and even the atmosphere at the time of the image. The steps leading up to the data preparation are usually the resizing images, removing noise, adjusting contrast, color normalization, geometric correction, dealing with the background, and correcting for lighting effects. One way of making the number of cases in training a machine to learn bigger is to change these examples by flipping, rotating, scaling, cropping, or flipping them, which would produce different versions of the original. Augmentation of data can, therefore, make it easier to train the neural network if it is presented different images. This is done while ensuring a level of plausibility for the changes, since changes that are too unnatural can end up leading the machine away from what a human can see as the actual problem (Ferentinos, 2018; Too et al., 2019).

4.2 Classification, Detection, and Segmentation

A process of classification by the image will result in it being grouped into one or more predetermined categories. It is mostly useful when a subject is clearly featured in the image. Scenes from fields are typically more complex as crop plants, weeds, soil, residues and symptoms are likely to come across together. Recognition and detection (a combination of identifying the object class and providing its approximate location) would address such a situation since it allows not just identification of a category but also the location of an object to within reasonable proximity. Segmentation gives a fine-grained spatial depiction of the image. Semantic segmentation identifies the category of the pixel whereas instance segmentation picks individual objects belonging to the same category apart. Identification through imaging is a technique by which an image can be linked to one or more predefined categories. This method is very handy if the content of the image is mainly about one subject. Agricultural field scenarios tend to be more complicated for a couple of very good reasons - the different plants including the crop plant, weed plants, exposed soil, and residue particles, the symptom itself, etc., are quite often found together. As such, a combined approach called object detection would provide a solution to this dilemma because through object detection one can find out both the class of and approximate location within the image a particular area, a plant, or an animal.

The task of segmentation is a more detailed description of the spatial structure. At the lowest level a segmentation model identifies each pixel in the image.

When it comes to crop protection, which of these is the best strategy depends on the kind of problem the manager is facing: to discover a disease type one might need classification, to detect individual weeds location might be required, and to segment the area affected one needs estimation (Ferentinos, 2018; Mohanty et al., 2016).

4.3 Classical Machine Learning and Deep Learning

Prior to the emergence of deep learning, the main way of doing image analysis in agriculture was to select various handcrafted features like color, texture, shape, edge, spectral, and morphological and combine them with classifiers such as support vector machines, random forests, k-nearest neighbors, or artificial neural networks. These methods remain effective when datasets are relatively small and the relevant features are well characterized. The advent of deep convolutional neural networks radically changed the workflow by allowing the visual features to be learned in a hierarchical manner directly from the images. Lower layers can capture edges and small color transitions whereas the higher layers can capture lesion-related or leaf morphology, or crop-weed pattern-related complex structures (Ferentinos, 2018; Too et al., 2019).

Transfer learning is especially helpful in agriculture as it is a very expensive thing to do creating extensive, diverse, manually annotated field dataset with a high quality. An agricultural model, for instance, that has been trained on a large general image dataset can be adapted for an agricultural task which means less task-specific training is needed. Comparative study by Too et al (2019) demonstrates that fine-tuning VGG, ResNet, DenseNet like architectures is quite effective for plant disease identification. As is the case with their results and other studies with controlled data, these findings do not constitute proof that the same accuracy can be achieved automatically in every field environment (Too et al., 2019). There are several deep learning systems developed for different purposes such as image classification, object detection, semantic and instance segmentation, and combination or integration between spatial-spectral domain. Traditional convolutional neural networks (CNN) - the models based on these layers are still the most common since they do extremely well as far as feature extraction is concerned. On the other hand, various types of models like ResNet, DenseNet, EfficientNet, and MobileNet are used for very different levels of accuracy, representational capacity, and resource usage. Object detection models, segmentation architectures, and Transformers have significantly

enriched the capabilities of deep learning to provide very accurate crop care solutions that take into account the specific situations where they are deployed.

Table 2 presents a brief description of the main deep-learning architectures that have been applied in AI-based crop protection. The primary capabilities of each, the main strengths, and the biggest difficulties or challenges are given (He et al (2016), Howard et al (2017), Hu et al (2024); Huang et al (2017), Redmon et al (2016), Ronneberger et al (2015), Tan and Le (2019), Too et al (2019)).

Table 2. Deep-learning architectures and their applications in AI-based crop protection

Deep-learning approach	Primary function	Main strengths	Major challenges
Convolutional Neural Network (CNN)	Image classification	Established automatic feature extraction	Needs representative data; basic classification gives limited localization
ResNet	Classification and feature extraction	Residual connections support deep architectures	Computational cost increases with model size
DenseNet	Image classification and feature reuse	Strong feature reuse and information propagation	Can require substantial memory
EfficientNet	Image classification	Useful accuracy–efficiency trade-off	Performance depends on scaling strategy and data quality
MobileNet	Lightweight classification	Low computational requirements	May trade some accuracy for efficiency
YOLO family	Object detection	Fast inference; suitable for real-time applications	Small or visually similar targets remain difficult
Faster R-CNN	Object detection	Strong detection performance	Heavier computation than many one-stage detectors
U-Net	Semantic segmentation	Effective pixel-level segmentation	Performance can decline in complex backgrounds
Mask R-CNN	Instance segmentation	Separates individual objects of the same class	More computationally demanding
3D-CNN	Spatial–spectral analysis	Jointly exploits spatial and spectral information	High data and computational requirements
Vision Transformer (ViT)	Image classification	Captures long-range relationships	Usually needs substantial data and computation
Swin Transformer	Hierarchical visual representation	Multiscale representation with efficient attention	More complex than conventional CNNs

CNN–Transformer hybrid	Local feature extraction + global context	Combines local and global representations	Architecture and computational complexity
Ensemble models	Combination of multiple models	Can improve robustness and reduce dependence on one model	Higher implementation and computation complexity

5. UAV-BASED CROP PROTECTION INTELLIGENCE

UAVs have become a very common tool in different agriculture fields as they take the position midway between ground-based detection systems and satellite imagery. While ground cameras can deliver very high level details at the plant level they have to be moved quite a bit of the time if the area is extensive which may be a big issue. Satellite pictures though being very good on the ground covering area may also be affected by various things like low image resolution, high time between revisiting a point to take another picture, cloud cover, and image availability. UAVs can get high resolution pictures quickly, and besides, they are quite versatile as they can be fitted with such devices as RGB cameras, multispectral, hyperspectral, or thermal sensors (Anam et al., 2024; Lottes et al., 2017; Peña et al., 2013).

Small and medium fields as well as highly detailed surveys are the main application areas for multirotor UAVs. Because of their ability to hover and do vertical take-off and landing (VTOL), multirotor planes are more maneuverable. For a job that requires a long period on the ground or a large area to be covered, then fixed-wing machines are probably the best choice. In the end, the choice of sensors should again be tied to the biological objective. If we can see symptoms through the RGB camera alone, that may be adequate for weed-recognition tasks, although if one wants physiological-level information, one may need multispectral or hyperspectral sensors. In fact, the thermal mode is great as it can tell us how a plant is handling water and what physiological response it has had (Anam et al., 2024; Lottes et al., 2017).

Typically, a UAV's pictures are processed into orthomosaics or else some form of geographic information system outputs. The main benefit of a UAV system is not its ability to produce good images but rather its ability to georeference the images, i.e. to know the exact location of what has been observed. For example, a disease or weed can be plotted onto a map as a hotspot, a place to spray, etc. or as a zone where scouting should be done first.

Lottes et al (2017) gave a practical example of how lightweight drone aerial sensing can be used to carry out classification of crops and weeds while Peña et al (2013) showed early results how UAV multispectral imaging technology was

able to generate very detailed weed maps for maize fields (Lottes et al., 2017; Peña et al., 2013).

Before deep learning became dominant, agricultural image analysis commonly used hand-engineered color, texture, shape, edge, spectral, and morphological features combined with classifiers such as support vector machines, random forests, k-nearest neighbors, and artificial neural networks. These methods remain useful when datasets are relatively small and the relevant features are well characterized. Deep convolutional neural networks changed the workflow by learning hierarchical visual representations directly from images. Early layers can represent edges and local color transitions, while deeper layers can represent more complex structures associated with lesions, leaf morphology, or crop–weed patterns (Ferentinos, 2018; Too et al., 2019).

Transfer learning is particularly useful in agriculture because large, diverse, expertly annotated field datasets are expensive to create. A model pretrained on a large general image dataset can be adapted to an agricultural task, reducing the amount of task-specific training required. Comparative work by Too et al (2019) showed the value of fine-tuning architectures such as VGG, ResNet, and DenseNet for plant disease identification. Their results, like other controlled-dataset studies, should not be interpreted as evidence that the same accuracy will automatically transfer to every field environment (Too et al., 2019).

AI-based disease and weed management in precision agriculture is not limited to detecting problems from images, and it requires a multistep decision-making and intervention process.

It is mainly composed of collecting image and sensor data, preprocessing of data, identification of disease or weed, assessing the severity and spatial distribution of the disease or weed outbreak, determining the intervention measures needed, choosing and implementing effective control measures, post-intervention monitoring and feedback that not only assess the performance of the system but also refine and improve management decisions. Figure 1 outlines this whole detection-to-intervention process. This integrated pipeline shows the most important steps from data collection, AI-based analysis, to the actual intervention in the field and getting feedback for the next round of decisions.

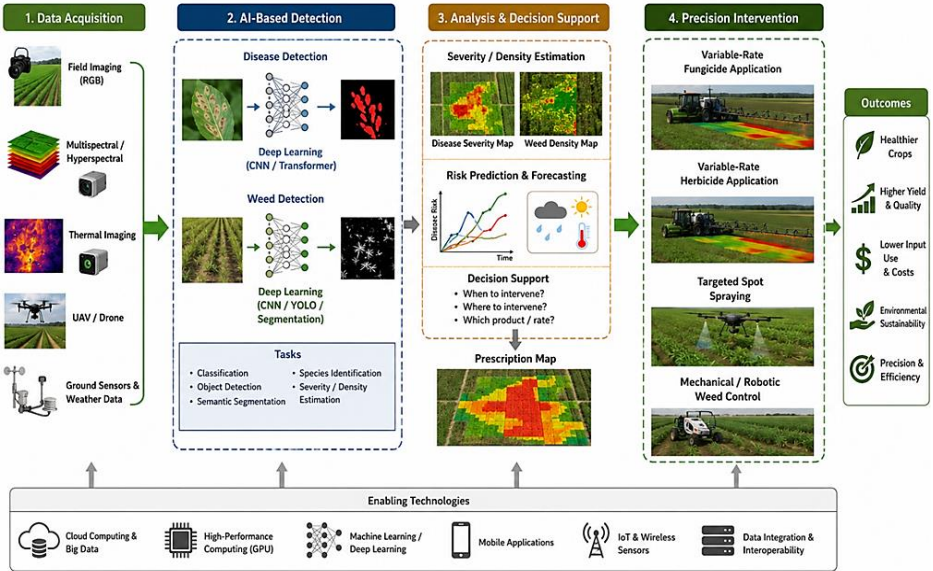


Figure 1. An integrated detection-to-intervention pipeline for AI-based disease and weed management in precision agriculture.

6. FROM DETECTION TO PRECISION INTERVENTION

The usefulness of AI can be enhanced when a model starts not only to detect the presence of a disease or weed but also to answer progressively more detailed questions such as what the problem is, where it occurs, how severe or dense it is, how it is changing, whether intervention is necessary, and what kind of action is suitable (Hu et al., 2024; Wang et al., 2017).

One could represent the process chain that follows as sensing, image processing, detection, diagnosis, quantification, spatial mapping, prediction, decision support, precision intervention, and feedback. Sensing may include UAVs, smartphones, ground cameras, weather stations, soil sensors, satellites, and IoT devices. Data analysis is performed at the processing stage. Detection and diagnosis identify irregular states and give likely classes. Quantification is the approximation of severity, density, infected area, or stress intensity. The outputs are then mapped to geographic coordinates through mapping. Prediction involves adding temporal and environmental data. Agronomic and economic criteria are used in decision support. Intervention translates the prescription into an action of chemical, mechanical, bacterial, or robotic type. Finally, the feedback step measures the result and introduces more data for future decisions (Lottes et al., 2017; Peña et al., 2013; Wang et al., 2017).

This explanation reveals the limitations of using only classification accuracy as a performance measure and highlights why the framework is more comprehensive. A model might be excellent in recognizing the isolated leaf images only for a high classification accuracy but may not still be suitable for the field-level decisions if it is not trustworthy about symptom localization, can't make a disease vs abiotic stress distinction, can't estimate severity, or can't deal with the changing illumination and crop architecture. In addition, the fields should be considered from the crop architecture viewpoint since the symptoms may be easier to detect if they are at the top in some plants but may be hidden in other types of plants. Therefore evaluation of such field-operational AI systems should include field-level generalization, spatial accuracy, temporal stability, uncertainty, latency, economic outcomes, and the effectiveness of ensuing interventions (Hu et al., 2024; Mohanty et al., 2016; Wang et al., 2017).

7. AI-BASED PRECISION DISEASE AND WEED MANAGEMENT

7.1 Precision Disease Management

Disease identification is one of the benefits of using crop management tools to locate disease hotspots when image data together with agronomic interpretation are used. To illustrate, a disease-risk map could identify areas for further inspection or the locations where treatments must be applied. Determination of disease severity is of special interest since the mere fact of the presence does not necessarily show the need of taking any actions. In this manner, Wang et al (2017) did a study to see if machine learning can help in diagnosing apple black rot at different disease stages automatically and they found that a machine learning model with VGG16 transferred features scored 90.4% in terms of holding out test set. In this case, machine learning was able to move the disease detection into the realm of generating quantitative information that supports crop protection activities (Lottes et al., 2017; Peña et al., 2013).

Variable-rate application of fungicide should be considered when both, the distribution of diseases and the decision thresholds, are clearly understood. At the same time, an AI model has to be cautious about proposing pesticide use autonomously without an agronomic check. The agronomic aspects that must be considered along with determining a treatment include the crop type, disease organism, product label, resistance Management principles, weather condition, disease development stage, and also local recommendations. AI may help a person organize and choose which information to follow, but the prescription should still be decided by agronomically valid knowledge (Ferentinos, 2018; Wang et al., 2017).

Table 3 summarizes illustrative disease-risk classes and the corresponding management approaches, from routine monitoring to immediate intervention.

Table 3. Illustrative disease-risk classes and management concepts.

Disease-risk class	Suggested management concept
Very low	Monitor
Low	Continue monitoring
Moderate	Targeted intervention if a validated threshold is exceeded
High	Priority treatment following agronomic verification
Very high	Immediate agronomic assessment and intervention where justified

7.2 Precision Weed Management

Weed control is highly compatible with precise site-specific weed control since different weed species are often found in patches. Detection could be done with cameras mounted on tractors, robots, or drones. A ground-based system may follow the sequence camera → AI detection → weed coordinates → decision → actuator → targeted control.

A UAV-based workflow can also provide a weed distribution map, convert it into management zones or a prescription map, and then transfer that information to a ground sprayer or other control platform (Eyili &Kaya Altop, 2024; Hu et al., 2024; Lottes et al., 2017; Peña et al., 2013).

Identification of the weed species can be a very deciding factor in such cases of weed management where a particular herbicide is effective only against susceptible weed species or where weed resistance to the herbicide is considered. Estimation of weed density can also be of great concern where management is based on herbicide rate of application or whether herbicides are used at all since a few isolated weeds and the whole field densely infested may not necessarily receive similar treatment (Hu et al., 2024; Peña et al., 2013).

AI-based precision crop protection extends beyond disease and weed detection by converting sensing data into actionable, site-specific management decisions. By combining imagery from UAVs and ground-based platforms with environmental and spatial information, AI systems can identify problem areas, assess risks, and guide targeted interventions such as spot spraying, variable-rate application, and mechanical weed removal. Table 4 summarizes representative AI-based pathways from crop sensing and analysis to decision support and precision intervention.

Table 4. Examples of AI-based pathways from crop sensing to precision intervention (Hu et al (2024); Lottes et al (2017), Peña et al (2013)).

Target problem	AI input	AI output	Decision-support function	Precision intervention	Expected benefit
Plant disease	RGB/UAV imagery	Disease presence and location	Identify disease hotspots	Targeted fungicide application	Potential reduction in unnecessary treatment
Plant disease	Multispectral imagery	Crop-stress indicators	Prioritize scouting	Site-specific treatment after verification	Earlier identification of potentially affected areas
Plant disease	Hyperspectral imagery	Spectral disease/stress signatures	Support early risk assessment	Targeted scouting or treatment	Potentially earlier intervention
Plant disease	UAV + weather data	Disease-risk prediction	Estimate short-term risk	Preventive or targeted intervention using validated thresholds	Improved timing of management
Plant disease	UAV time-series imagery	Disease progression	Identify expanding zones	Prioritize rapidly developing hotspots	Faster response to spread
Weed infestation	RGB camera	Weed detection	Identify individual weeds	Spot spraying	Potentially lower herbicide application
Weed infestation	RGB + multispectral UAV imagery	Weed distribution map	Generate management zones	Variable-rate herbicide application	Improved spatial targeting
Weed species	High-resolution RGB imagery	Species-level classification	Select management strategy	Species-specific treatment where validated	Improved weed-control efficiency
Weed infestation	Ground robot + RGB camera	Real-time weed localization	Determine treatment coordinates	Mechanical weed removal	Reduced dependence on herbicide
Mixed crop stress	RGB + thermal + multispectral data	Stress classification	Distinguish possible causes of stress	Agronomic verification and targeted management	Reduced risk of treating the wrong problem
Disease + weed	Multimodal UAV data	Integrated crop-protection map	Establish spatial management zones	Variable-rate or site-specific intervention	More comprehensive field management
Crop protection	AI + IoT + weather data	Risk and treatment recommendation	Combine real-time, historical information	Timely intervention	Improved decision support
Autonomous crop protection	Camera + AI + GPS/RTK	Real-time target coordinates	Control machinery	Autonomous spraying or mechanical removal	High spatial precision, reduced operator workload

8. EARLY WARNING, MULTIMODAL AI, AND REAL-TIME DECISION MAKING

Detection means the identification of the existing problem, but early warning aims at predicting a forthcoming situation based on the available data. Factors like temperature, humidity, rainfall, leaf wetness, crop physiological stage, cultivar vulnerability, pathogen presence and crop past history that together determine the manifestation of diseases are considered in a predictive system. This system is supposed to integrate such factors and repeated imaging data to produce a risk estimate (Anam et al., 2024; Wang et al., 2017).

A spatially resolved early warning is more valuable than a single field-level risk score. For example, it might suggest “risk is mainly located in the northern part of the field” to facilitate scouting and optimize resource allocation. Alternatively, it could mean the whole field is high on risk. Multiple UAV passes over the same areas are also capable of offering time-series images to the system so that it can differentiate the stress patterns as stable, expanding, declining or newly emergent (Anam et al., 2024; Lottes et al., 2017).

Multimodal AI introduces the idea that different data streams can be combined if they are not very independent of each other. For instance, if you see a canopy turning yellow in the RGB image, a number of different factors may be causing it; a combination of the thermal sensor showing the canopy temperature being raised with weather and soil observations hinting at a drought condition are some examples. On the other hand, visual symptoms and environmental conditions that are very supportive of pathogen development would serve to increase suspicion of a case of disease. Besides predictive success, multimodal systems should also be assessed as to whether they reduce biologically meaningful confusion caused by different data sources (Fahlgren et al., 2015; Mishra et al., 2020).

When decisions have to be very quick or when connectivity is an issue, edge AI could be the right choice. Local processing of the images at/near the sensor can also help reduce the amount of data transfer and the time lag. It is especially useful for autonomous tractors, robotic weeders, real-time sprayers, UAVs and mobile apps. The optimization problem that results from this situation is to get the best result without compromising on power usage, CPU time, etc. The use of model compression, quantization and/or other optimization methods would be necessary if these models are to operate in the field situation (Hu et al., 2024).

9. EXPLAINABILITY, GENERALIZATION, AND REAL-WORLD DEPLOYMENT

The major hurdle for agricultural AI is the discrepancy between benchmark accuracy and field reliability. For instance, models trained on clean lab images may produce very high accuracy, but become highly unpredictable once they are presented with actual field data. The work by Mohanty et al. (2016) shows this phenomenon. On the other hand, Ferentina (2018)'s study revealed great prospects with deep learning based on a very big dataset, but also pointed out that the use of image databases alone does not eliminate the demand for field testing (Ferentinos, 2018; Hu et al., 2024; Mohanty et al., 2016).

The changes in the domain may happen because of a different location, variety, soil, illumination, camera, viewing angle, disease phase, crop structure, or the background. A model could accidentally pick up certain image features that are only due to the settings of the image-capturing equipment but not the underlying biological aspects of crops. Hence, data sets must be robust so that they include different locations, seasons, growth stages, cultivation conditions, camera settings, weather patterns, and disease or weed severity levels. Furthermore, test sets ought to contain genuinely new and varied field data so as not to mimic the training images (Hu et al., 2024; Mohanty et al., 2016).

It is also the case of imbalance in the classification of the items. For example, images of healthy plants and common weeds may account for most of the dataset but the less common plant disease signs or rare weed plantings may not receive a similar amount of data. The overall correctness can hence hide the weakness in the classes that are most relevant for the protection of the crops. Evaluation should include measures specifically for each category and, where necessary, spatial and temporal performances instead of only one aggregated metric.

Having a transparent decision-making system can lead to increased model validation and enhanced human supervision. The utilization of methodologies such as saliency detection, Grad-CAM, SHAP, etc., reveals what parts of an image or what input variables contributed to the decision. The ideal agricultural system should not only give the predicted result but also indicate the level of certainty, the size of the affected region, spatial coordinates, temporal trends and possibly the need for expert verification (Lundberg & Lee, 2017; Selvaraju et al., 2017).

Other limitations brought by flying drone-based image acquisition are battery life, wind, rain, payload, sensor cost, regulations, data storage, and processing speed. Consequently, the most technically advanced sensor may not turn out to be the most realistic. The process of deploying new technology must be thoughtfully designed to cover everything from data collection to sensor calibration to the stage of decision-making, the physical action of executing the decision, and the post-implementation review of the effects (Anam et al., 2024; Lottes et al., 2017).

10. ROBOTICS, SUSTAINABILITY, AND ECONOMIC FEASIBILITY

At the heart of a smart crop protection system is the ability of the system to make decisions based on geographic data. It is conceivable that robotic systems in agriculture will carry out mechanical removal of weeds, targeted spraying, precision digging, plant-specific treatment, scouting, and autonomous navigating, to list a few. The entire set of actions could be a result of a closed-loop system where the input is aerial imagery. After the hotspots are identified and the areas of a map that require the greatest attention are defined, the plan of action is handed over to the ground robot and through a camera the targets are re-verified before either a mechanical or chemical action is carried out. Then the outcome is assessed by another sensor (Hu et al., 2024; Lottes et al., 2017).

The environmental value of precision AI is contingent on the actual result of managing resources through such a platform. It is worth pointing out that, for example, there are some possible advantages: less pesticides and herbicides sprayed, fewer tracings on the ground, reduced pesticide spread over the air through drift, better utilization of resources or inputs, and less intensive intervention. However, those benefits are not to be assumed automatically because AI applications in themselves are also made of things: they depend on sensors, batteries, computer facilities, data storage, system maintenance, and electronic components. Therefore, if one is to do full sustainability evaluation, the environmental impact of the whole technology system has to be taken into account (Anam et al., 2024; Lottes et al., 2017; Peña et al., 2013).

Cost-effectiveness is another major factor as well. Components like hardware, software, labor, maintenance, data processing, chemical cost savings, yield protection, etc., and long-term benefits of such a system altogether should be taken into account. A vast field farm probably will be able to distribute the cost of the UAV and precision-application devices over a considerable area, while smaller ones, for whom such expenses will amount to a bigger burden, can opt

for a more affordable system on a smartphone, shared or cooperative UAV-based farming solutions, or services hosted in the cloud. On top of that, the level of trust in such automated systems and perceived ease of use, as well as reliability, will also affect a technology change or the process of adoption of technology (Lottes et al., 2017; Peña et al., 2013).

11. FUTURE PERSPECTIVES AND HUMAN–AI COLLABORATION

Rapidly developing agricultural AI will possibly soon become more multimodal (e.g., combining image and environmental data) and more adaptive. Big pretrained or foundation models could give visual representations that can be adapted to different crops and plant diseases. Thus, a farm owner will not need to create an artificial intelligence (AI) model from scratch to, say, identify wheat leaf rust; instead, he or she could just use a model based on such a representation, which had been already adapted, perhaps, from the detection of grapevine powdery mildew. This could dramatically reduce the development cost of such models and their usage in different agricultural situations. However, agricultural foundation models will need various, well-annotated field datasets and must undergo strict testing in terms of spatial variability, temporal variation (like changing season), and farming practices (Hu et al., 2024; Too et al., 2019).

New technologies such as Vision Transformers and hybrid CNN–Transformer models provide different options for how machines understand visual data. Being able to grasp relationships not just between pixels in immediate proximity (a characteristic of conventional computer vision techniques) but also further apart might give them edge in handling scenes like a dense canopy and similar complex tasks. However, it must be remembered that these methods come with significant demand for computing power, processing, and amount of data required. Eventually, Digital Twin solutions might combine soil and weather data, historical yield and disease observations, drone imagery, weed maps, crop and management information to simulate interventions. It will also be necessary to train continual AI to adapt itself gradually over time to changes in cultivars or environments or disease forms as well as farming methods (Dosovitskiy et al., 2021; Liu et al., 2021).

Actually the realistic way forward of the development is likely to be a hybrid model of a human-AI collaboration rather than one in which the human is completely replaced and a machine makes all the decisions. AI will do all the background stuff-monitoring, detecting, quantifying, creating maps, forecasting, and giving a priority list, while human farmers and agronomists will still have the final say on the matter (interpreting the AI's decisions, validating them, deciding whether to act on that advice, intervening, changing the system or

overriding the recommendation when necessary). This sharing of responsibility between the robot/programmer and the human brings together the advantage of big machines that can process and store very large amounts of data with the biological, practical, and technical know-how, resulting in a feasible and safe way forward of automated production in agriculture fields of farming (Hu et al., 2024).

12. INTEGRATED FRAMEWORK FOR SUSTAINABLE AI-BASED CROP PROTECTION

A holistic approach is the proposed crop monitoring framework, which involves a cycle of gathering, analyzing, interpreting, and applying knowledge with respect to a field. The first part focuses on monitoring via cameras, drones, satellites, sensors plus in field surveys. Second part comprises processing data via image techniques and computer learning to recognize pests and calculate the disease extent or weed quantity in addition to mapping these to a place/feature. Following which, other data about conditions from surrounding environments are used, besides information from the past, for assessing a particular risk level. The justification or not of an intervention depends on certain benchmarks and costs. After deciding the suitable intervention, it is performed through a range of methods like chemical, physical, natural as well as robotically done procedures and it is further assessed. The fresh observations are used for enhancement of the next decisions as well as the predictive ability of the system (Hu et al., 2024; Lottes et al., 2017; Peña et al., 2013).

The whole design can be put in simple terms like Sense-Detection-Symptoms identification-Extent evaluation-Visualization-Forecasting-Decision making-Prevention-Checking-Adapting. The elements of measurement, localization, forecasting, learning, and providing feedback make this operational system quite different from an image classifier system that works in isolation. Besides that, it opens up possibilities to judge AI tools not only from recognition scores on benchmarked datasets, but also their impact on real agricultural practices.

13. CONCLUSIONS

Merging AI with robotics, computer vision, UAV-based technology (Drones), Deep learning, and Precision Ag are now offering an environment where spatially precise crop protection is a reality. The fact that deep artificial neural networks can outperform humans at recognizing images of plants infected by diseases was clearly demonstrated by studies carried out on the recognition of plant disease. Other work, including studies on weed mapping from crop fields and UAV-based classification of crops and weeds has shown quite clearly the

usefulness of spatially explicit sensing. Recent work shows the growing application of Deep-learning and AI in Weed recognition and Crop-protection from the sky. In fact, UAVs have become the most commonly used platform of AI (Anam et al., 2024; Hu et al., 2024).

However, the biggest challenge now is not just an AI system's ability to recognize a disease or weed. The far most important question is does the system be able to convey reliably, quickly, and with the proper localization, the needed information at an interpretable level to support the best economically and environmentally sound decision on intervention.

Agricultural situations are in constant change, so the performance can vary from place to place, season by season, cultivar, weather, device types, crop stage, and different management conditions. Hence, robust field datasets, multimodal sensing methods, uncertainty estimation, model explainability, and model adaptation, computing at the "Edge" to allow fast decisions or processing data locally on the device, validation across years and locations are critical (Hu et al., 2024; Mohanty et al., 2016).

An AI-enabled crop protection system should combine sensor technology with diagnosing, quantifying, geospatial analysis, prediction, decision making, prevention, and getting feedback together. When the components of a system get tested through an integrated workflow, AI could help cut down on inputs that are superfluous, targeting that is more precise, and timely decisions leading to more sustainable farming. It's therefore the future agricultural AI not just about standalone recognition, AI-driven cropping practices that are intelligent, adaptive in real-time, spatially aware, explainable & actionable, and in collaboration with agricultural expertise shall bring change (Hu et al., 2024; Lottes et al., 2017; Peña et al., 2013).

DECLARATION OF AI USE

AI-based tools were used to assist in the creation and design of some figures presented in this chapter. The authors take full responsibility for the scientific content, accuracy, and interpretation of the presented materials.

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Chapter 3

From Aquatic Biomass to Carbon Quantum Dots: Sustainable Conversion and Valorization

Sevim HAMZAÇEBİ¹

1.Introduction

Over the past decades, advances in scientific research have contributed substantially to technological innovation across numerous disciplines. These advances have supported progress in diverse areas, including medicine, environmental science, energy, and food technology. Within this broader technological landscape, nanotechnology has gained prominence as an interdisciplinary field with the potential to address emerging global challenges through nanoscale approaches. Nanotechnology focuses on materials at the nanoscale, generally within the 1–100 nm range, where size-dependent effects can substantially alter their physical, chemical, and optical properties compared with those of conventional bulk materials.

The integration of nanotechnology into aquaculture has attracted increasing research interest as a means of enhancing production efficiency while providing innovative strategies to address a range of biological and environmental constraints. Nanoparticle-based applications in the aquaculture and food sectors encompass a broad range of uses. In aquaculture, nanoparticle-based approaches can be employed for various purposes, including improving fish growth performance, enhancing water filtration and quality, removing pollutants such as heavy metals from aquatic environments, and increasing the efficiency of harvesting processes (Ahmed et al., 2024; Khalil et al., 2023; Mitra et al., 2023). In addition, increasing the bioavailability of functional compounds, delivering nutrients, nutraceuticals, enzymes, and various food additives, and enabling the controlled release of active ingredients are among the important applications of nanotechnology (George et al., 2023; Srinivasan et al., 2016).

Nanotechnological approaches can also contribute to preserving the quality characteristics of food products by enhancing the stability and shelf life of sensitive components. In addition, nanomaterials with antimicrobial properties are being investigated for preventing microbial contamination in seafood

¹ Izmir Katip Çelebi University, Faculty of Fisheries, Department of Aquaculture, 35620 Izmir, Türkiye
(ORCID: <http://orcid.org/0000-0002-2179-1900>)
Corresponding author: sevim.hamzacebi@ikcu.edu.tr

products and improving product safety (Torres-Giner et al., 2022). In feed technology, nanoscale applications can enhance the physical, chemical, and nutritional properties of feed ingredients, improve nutrient utilization efficiency, and facilitate the development of more functional feed formulations tailored to specific species (Fajardo et al., 2022; Lall & Dumas, 2022).

The applications of nanotechnology are not limited to aquaculture and feed-related practices; they also encompass the development of antifouling surfaces to reduce biofouling on fishing equipment and aquaculture nets, the use of antibacterial materials in aquaculture tanks, and the development of next-generation packaging materials designed to preserve the quality and safety of seafood products (Ogunkalu, 2019; Singh et al., 2023; Basha et al., 2026). Collectively, these developments highlight the versatility of nanotechnology within aquaculture and the broader seafood industry, with potential applications extending throughout the production chain, including processing, storage, and the maintenance of product quality and safety.

2. Structural and Functional Attributes of Carbon Quantum Dots

The rapid evolution of nanotechnology has opened up new opportunities for nanoscale materials in various sectors, as their properties can be tailored to achieve specific physical, chemical, and biological functions. Among these characteristics, the optical properties of nanomaterials have attracted considerable scientific interest, particularly because their behavior can vary depending on particle size, morphology, and surface characteristics. Various classes of nanomaterials, including carbon nanotubes, graphene oxide, metal oxide nanoparticles, and quantum dots, offer broad potential for applications in biomedical, environmental, and technological fields owing to their distinctive properties (Bayda et al., 2020). Unlike their bulk counterparts, nanoscale materials can exhibit unique characteristics in optical processes such as light absorption, scattering, and emission. Understanding and controlling these properties have contributed significantly to the development of next-generation technologies in a variety of fields, including bioimaging, sensor technologies, photocatalysis, optoelectronics, and energy applications (Dar et al., 2021). Carbon quantum dots (CQDs) have emerged as promising nanoscale materials, particularly for applications where biocompatibility, aqueous stability, low toxicity, and efficient light emission are important. These properties make CQDs promising candidates for a range of advanced technological applications, particularly in biosensor development, controlled drug delivery, and photocatalytic processes (Abu et al., 2023; Guan et al., 2023). Furthermore, the availability of numerous physical and chemical synthesis approaches enables the

properties of these nanomaterials to be tailored to specific application requirements. The combination of these features positions CQDs as a safer and more environmentally friendly alternative to conventional semiconductor quantum dots (Si et al., 2020; Zhao et al., 2018).

Carbon quantum dots (CQDs) are nanosized carbon-rich nanomaterials with semiconductor-like characteristics that exhibit distinct photoluminescence behavior upon excitation with light. These nanostructures can absorb light at specific wavelengths and subsequently emit light at longer wavelengths, a property that has been associated with quantum confinement effects arising at the nanoscale (Dong et al., 2024; Jan et al., 2024). A broad range of carbon-containing materials and organic precursors can serve as starting materials for CQD production, with the synthesis parameters being adjusted to obtain specific characteristics for particular applications. The favorable characteristics of CQDs, including excellent aqueous solubility or dispersibility, low toxicity, antibacterial effects, selective interactions, and high sensitivity, make these nanomaterials promising candidates for diverse applications, particularly in biological and environmental fields. Owing to these distinctive properties, CQDs have been investigated in a wide range of applications, including bioimaging, biosensor development, cancer diagnosis and therapy, targeted delivery of genetic materials and drugs, metal ion detection, and the removal of pollutants from water (Shandilya et al., 2020; Yadav et al., 2024).

At the nanoscale, CQDs possess a carbon-based framework whose surface chemistry is shaped by a range of attached functional groups. The elemental profile of CQDs is influenced by both the nature of the precursor and the synthesis conditions, with the resulting structures potentially containing, besides carbon, elements such as H, O, N, S, and P. CQDs generally have sizes on the order of a few nanometers, with core diameters frequently reported to be approximately 2–3 nm in the literature (Xu et al., 2004; Subramania et al., 2023). In some studies, the terms carbon dots (CDs) and carbon quantum dots (CQDs) are used interchangeably. Thus, similar carbon-based nanomaterials may be referred to as CDs or CQDs depending on the classification and nomenclature approach adopted by the authors (Zhang et al., 2019; Dhandapani et al., 2020). CQD characteristics can vary considerably depending on the precursor composition and synthesis conditions, including temperature, reaction time, pH, pressure, and the selected production route. Therefore, the morphological, structural, and surface properties of CQDs are closely related to the synthesis conditions, and controlled modification of the synthesis parameters enables the properties of the resulting nanomaterials to be tailored to specific applications (Chauhan et al., 2022; Guan et al., 2023).

3. Synthesis Approaches for Carbon Quantum Dots

The preparation of CQDs involves a range of synthesis routes, and the choice of processing conditions is generally tailored to the characteristics required for their specific applications. In addition to hydrothermal and solvothermal processes, commonly employed techniques include microwave-assisted synthesis, laser ablation, pyrolysis, arc discharge, ultrasonic methods, and chemical and electrochemical oxidation (Rasal et al., 2021; Sohal et al., 2023). Variations in the synthesis method and processing conditions may directly affect the particle size, surface functional groups, structural characteristics, photoluminescence behavior, and other physicochemical properties of the resulting quantum dots. Therefore, selecting an appropriate synthesis method is critical for obtaining CQDs with the desired properties. Owing to their tunable characteristics, these nanomaterials can be utilized in a wide range of fields, including bioimaging, water and wastewater treatment, photovoltaic systems, hydrogen production, energy storage, and optoelectronics (Bartkowski et al., 2024; Kwon et al., 2016).

Among the available routes for producing carbon quantum dots (CQDs), hydrothermal treatment and microwave-assisted carbonization are particularly attractive because they offer relatively simple processing and economical operation. Careful control of the synthesis conditions allows these approaches to generate CQDs with controlled size characteristics and greater structural consistency. Depending on the chemical composition of the starting material, carbonization may inherently introduce nitrogen and/or sulfur into the resulting CQDs, eliminating the need for an additional heteroatom-doping process. Such heteroatom incorporation can modify the electronic configuration and surface chemistry of CQDs, which may consequently influence their optical characteristics, surface reactivity, and overall functional performance in different applications.

4. Utilization of Biomass and Waste as Carbon Precursors for CQD Production

Early studies on CQD synthesis largely relied on purified, commercially sourced organic molecules and polymers as carbon precursors. Commonly employed materials included citric acid, glucose, urea, chitosan, ethylenediamine, and polyethylene glycol. These synthetic precursors have been widely employed for CQD production because of their well-defined compositions and the relative ease with which the synthesis conditions can be controlled. However, the cost of obtaining and consistently using these precursors at high purity can constitute a significant limitation, particularly for large-scale

production. Furthermore, increasing emphasis on sustainable and environmentally conscious nanomaterial production has encouraged researchers to explore renewable, readily available, and economically viable carbon feedstocks.

In this context, biomass, agricultural residues, and particularly marine-derived waste materials have attracted considerable interest as potential precursors due to their carbon-rich composition, renewable nature, wide availability, and low cost. The carbon-rich organic components present in these materials can provide the carbon source required for CQD formation under appropriate carbonization conditions. Furthermore, the diverse chemical constituents naturally present in biomass can influence the development of surface properties and functional moieties during CQD formation. Thus, the use of biomass resources not only provides an alternative carbon source but also enables the development of CQDs with diverse properties. In particular, converting waste materials into CQDs provides an effective valorization pathway by recovering resources that have limited economic value or would otherwise be discarded. Such a strategy enables the transformation of waste streams into high-value nanomaterials, improves resource efficiency, and contributes to the economic viability and environmental sustainability of CQD production. Therefore, the use of biomass and waste materials for CQD synthesis represents an important alternative to reliance on synthetic carbon precursors and promotes circular and environmentally friendly production approaches (Xu et al., 2020).

The production of CQDs is not limited to conventional chemical precursors; low-value biological resources and discarded biomass can also serve as suitable carbon sources. The use of waste biomass for this purpose contributes to reducing the environmental burden of waste while simultaneously enabling the conversion of low-value biological materials into value-added nanomaterials (Kang et al., 2020). Biomass is a renewable biological resource obtained from a wide range of sources, including agricultural activities, livestock production, fisheries, poultry farming, forestry, related industrial processes, and urban organic waste (Queiroz et al., 2020; Tripathi et al., 2016; Ghimire et al., 2015). This broad diversity of sources enables the utilization of numerous readily available biomass types with different characteristics for CQD production.

Structurally heterogeneous, biomass wastes are naturally occurring carbonaceous materials rich in cellulose, hemicellulose, and lignin, as well as proteins, lipids, ash, and various organic components. During carbonization and thermal treatment, the constituent compounds undergo degradation and structural transformations, ultimately giving rise to carbon-rich nanostructures. The carbohydrates, proteins, and other organic constituents present in biomass

contribute to the formation of the carbon core of CQDs, while biomass sources with high protein and amino acid contents can also promote the formation of nitrogen-containing functional structures. This suggests that the surface chemistry of the resulting CQDs, as well as their associated optical and physicochemical properties, may be related to the initial composition of the biomass (Koupaie et al., 2019; Babbar et al., 2016).

These attributes make biomass-derived wastes attractive feedstocks for carbon-dot production, as they offer renewable, economical, and readily accessible sources of carbon. In addition, the availability of biomass from diverse sources increases feedstock diversity for CQD production and reduces dependence on a specific raw material source. Using waste biomass directly as a carbon source promotes more efficient resource utilization by converting materials destined for disposal into useful inputs for nanomaterial production. Therefore, biomass-derived CQD production is considered an important approach that combines waste valorization with the sustainable production of functional nanomaterials (Queiroz et al., 2020).

Table 1. The properties and applications of C-dots produced from different biomass waste (Xu et al., 2004; Kang et al., 2020).

Biomass Waste	Application	References
Wheat bran	drug delivery	John et al., 2020
Sugarcane Bagasse char	drug delivery	Chung et al., 2020
Waste food	light emitting diodes	Sarswat et al. 2015
Orange peels	photocatalysis	Prasannan et al., 2013
Onion waste	Fe ³⁺ detection and multicolor imaging	Bandi et al., 2016
Waste food	Fe ³⁺ detection	Ying et al., 2018
Tobacco leaves	three kinds of tetracyclines detection	Miao et al., 2018
Coffee grounds	Fe ³⁺ , Cu ²⁺ detection	Liang et al., 2016
Rice residue	Fe ³⁺ ions and tetracyclines detection	Qi et al., 2019
Bael leaves	Fe ³⁺ detection	Pramanik et al., 2017
Wheat straw	labeling, imaging and sensing	Ming et al., 2015
Lemon peels	sensing and photocatalysis	Tyagi et al., 2016
Wheat straw and bamboo residues	cell imaging and in vivo bioimaging	Xing et al., 2019
Dried lemon peels	carmine detection	Su et al., 2018

Tulsi leaves	Pb ²⁺ detection	Kumar et al., 2017
Magnolia flower	Fe ³⁺ detection	Wang et al., 2020
Bamboo leaves	Cu ²⁺ detection	Liu et al., 2007
Pomelo peels	Hg ²⁺ detection	Lu et al., 2012
Coconut husks	pH sensor	Chunduri et al., 2016
Prawn shells	nitrite detection	Zhang et al., 2016
Shark cartilage	Bioimaging and biomedical applications	Kim et al., 2020
Crustacean waste	Antioxidant and antimicrobial applications	Salmas et al., 2024
Euclidean denticulatum (marine macroalgae)	Tetracycline detection and environmental monitoring	Li et al., 2024

5. Carbon Quantum Dots from Marine-Derived Polysaccharides

Marine organisms and microorganisms provide abundant sources of structurally diverse polysaccharides and other biopolymers, including alginate, carrageenan, chitin, chitosan, agarose and ulvan. Their renewable origin and naturally occurring carbon, oxygen, nitrogen, and sulfur make these biomolecules suitable starting materials for the sustainable production of carbon quantum dots. In particular, the carbon-rich backbones of polysaccharides can provide the carbon source required for the formation of the CQD core during hydrothermal, microwave-assisted, or other carbonization-based synthesis processes (Hammi et al., 2020; Helbert, 2017).

A key feature of marine-derived polysaccharides in CQD synthesis is their dual role as carbon precursors and contributors to the surface chemistry of the resulting nanomaterials. Their inherent structural functionalities, including hydroxyl, carboxyl, amino, and sulfate groups, can participate in chemical transformations during the synthesis process and subsequently influence the functional groups formed on the CQD surface. Moreover, polysaccharides containing nitrogen (N) or sulfur (S) may act as intrinsic sources of these heteroatoms, promoting their incorporation into the carbon framework and consequently altering the surface characteristics and electronic behavior of the resulting CQDs. The incorporation of heteroatoms into the CQD structure can play a role in enhancing their optical, electronic, and photoluminescence properties by altering surface-active sites and energy levels (Hammi et al., 2020; Helbert, 2017).

From this perspective, the natural presence of heteroatoms and functional groups in marine polysaccharides may reduce the need for additional nitrogen or sulfur sources during synthesis. Consequently, CQD production may be carried out using fewer chemical components, potentially simplifying the synthesis process. The structural diversity and varying functional-group composition of marine polysaccharides can influence the physicochemical characteristics of the resulting CQDs, including their particle size, surface chemistry, and optical response. Such variability offers opportunities to tailor CQD characteristics by selecting marine biopolymers according to the requirements of specific applications.

The availability of marine polysaccharides from renewable sources and, in some cases, their production from marine biomass wastes further enhances the importance of these materials for sustainable nanomaterial production. From this perspective, marine biopolymers represent valuable feedstocks for CQD production, offering a dual benefit by enabling the valorization of biological resources while reducing the need for expensive, synthetically derived carbon precursors. Therefore, the use of marine polysaccharides offers considerable potential for the development of more sustainable, low-chemical-consumption, and environmentally compatible synthesis approaches for CQD production (Torres et al., 2023).

Table 2. Marine polysaccharides classified by origin and main properties of CQDs synthesized using them as precursors (Torres et al., 2023).

Source	Marine Polysaccharide	Size (nm)	Color	Quantum Yield (%)	Heteroatoms Content (%)	References
Marine algae	Carrageenans	1.5–30 0.63–3.92 ^c	Yellow brown ^a , Yellow ^a , Green ^b	2–69.27	S%: 2.7–5.29% N%: 8.18%	Leuterio et al., 2022; Jorns et al., 2021; Das et al., 2019; Supchocksoonthorn et al., 2021; Suhail et al., 2022.
	Alginates	2–20 100	Yellow ^a , Brown ^a , Green ^b , Blue ^b	5.42–48.7	N%: 0.02%	Zhou et al., 2016; Sun et al., 2020; Atienza et al., 2013; Fong et al., 2015.
		4.14 ± 0.70	Yellow-green	-	N, S	Wang et al., 2026

	Others	1–40	Dark brown ^a Green ^b , Blue ^b	56–62	O%: 26.98– 32.34% S%: 3.15% N%: 2.41– 8.12%	Chauhan et al., 2021; Tang et al., 2022; Chen et al., 2016; Singh et al., 2020
Crustaceans	Chitins	5	Blue	-	-	Wang et al., 2025
		1–20.5	Yellow ^a , Brown ^a , Orange ^a , Blue ^b , Green ^b	5.1–35	O%: 4.67– 28.54% S%: 0.38– 1.54% N%: 2.63– 18.25%	Gedda et al., 2016; Naghdi et al., 2017; Sun et al., 2021; Su et al., 2019; Shchipunov et al., 2015; Gonçalves et al., 2020; Feng et al., 2022.
	Chitosans	0.6–11 2.7–3 c	Transpare nt ^a , Brown ^a , Yellow ^a , Blue ^b , Green ^b	3.3–40	O%: 1.47– 51.67% N%: 0.02– 9.63%	Mu et al., 2021; Feng et al., 2022; Pawar et al., 2019; Fu et al., 2022; Wang et al., 2016; Mathew et al., 2020; Xu et al., 2022; Zattar et al., 2022; Ni et al. 2022; Bera et al., 2020.
Fish	Chondroitin sulfate (shark cartilage)	19.6–60	Yellow-brown ^a Yellowish-green ^b Blue ^b	20.46	C% and O% > 90% S% < 10%	Kim et al., 2020
	Collagen and chitin (Fish scales)	13	-	17.3	O%: 26% N%: 8%	Campalani et al., 2021

(a) Appearance under daylight; (b) fluorescence under UV illumination; (c) hydrodynamic diameter determined by dynamic light scattering (DLS).

6. Properties and Applications of Aquatic Biomass-Derived CQDs

Aquatic organisms represent valuable sources for CQD synthesis, as their biomass contains abundant carbonaceous compounds and a broad range of structurally diverse biomolecules. Algae, crustaceans, mollusks, jellyfish, and

other marine biomass sources can be utilized as precursor materials for CQD synthesis because they contain carbohydrates, proteins, pigments, and other carbon-rich components. The use of these resources not only enables the production of high-value-added nanomaterials but also contributes to the sustainable conversion of marine biomass into functional products.

6.1. Valorization of Fish Biomass for Carbon Quantum Dot Production

Aquatic organisms provide important natural resources for the production of environmentally friendly, biocompatible, and low-toxicity fluorescent carbon-based nanomaterials. Chondroitin sulfate derived from shark cartilage has also been investigated as a marine polysaccharide precursor for carbon dot (CD) synthesis. Using an aqueous solution of chondroitin sulfate, CDs were produced through a single-step hydrothermal process and exhibited multicolor photoluminescence, with a quantum yield of 20.46%. Biological evaluation in zebrafish indicated that the resulting CDs exhibited low cytotoxicity and favorable biocompatibility. Moreover, their preferential localization within the intestinal region of zebrafish larvae highlights their potential for *in vivo* fluorescence imaging applications. These findings indicate that marine-derived polysaccharides can serve as sustainable precursors for the production of functional fluorescent nanomaterials and that the resulting CDs have considerable potential for bioimaging and biomedical applications (Kim et al., 2020).

Carp fish scales have been utilized as an inexpensive and readily available precursor for the hydrothermal synthesis of fluorescent carbon dots (CDs). The obtained CDs contained nitrogen- and oxygen-bearing surface functionalities and showed a strong emission peak at 430 nm, with a relative photoluminescence quantum yield of 6.9%. They also exhibited low cytotoxicity and good resistance to photobleaching, while their fluorescence remained stable under varying ionic strengths. Notably, the presence of Fe^{3+} ions caused a selective reduction in CD fluorescence, providing the basis for their use as fluorescent probes for Fe^{3+} detection. The proposed sensing system showed a linear response over 1–78 $\mu\text{mol L}^{-1}$, with a detection limit of 0.54 $\mu\text{mol L}^{-1}$. Its applicability was further supported by satisfactory recoveries and low standard deviations obtained from Fe^{3+} measurements in water and human serum samples. Overall, the results indicate that carp-scale-derived CDs can provide a stable and biocompatible platform for Fe^{3+} sensing in environmental and biological matrices (Zhang et al., 2019).

Fish-scale waste has been employed as an inexpensive biomass precursor for the hydrothermal preparation of water-dispersible carbon quantum dots (CQDs) exhibiting intense green fluorescence. The synthesized CQDs were characterized by UV–Vis absorption and photoluminescence (PL) spectroscopy to determine

their optical and physicochemical characteristics. Under excitation at 330 nm, the CQDs produced a pronounced green emission centered at 406 nm. Their UV–Vis spectrum also contained absorption features at 266 and 371 nm, which were assigned to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transitions, respectively. According to morphological characterization, the particles displayed a spherical structure with an average diameter of approximately 6 ± 1 nm. The combination of their optical response and physicochemical characteristics suggests that fish-scale-derived CQDs may be suitable for applications in areas such as energy technologies and water purification (Dhandapani et al., 2020).

6.2. Carbon Quantum Dots from Crustacean-Derived Biomass

Crayfish processing residues offer a valuable biomass feedstock for the recovery of multiple high-value products. In one integrated valorization strategy, these residues, which may account for as much as 80% of the original seafood biomass, were processed through a four-stage procedure to obtain chitosan, calcium hydroxide, calcium carbonate, and nitrogen/sulfur co-doped carbon quantum dots (N/S-CQDs). The resulting N/S-CQDs had an average particle size of 5.01 nm, as determined by atomic force microscopy (AFM). The incorporation of nitrogen and sulfur into the carbon framework can influence the surface composition, electronic characteristics, and optical response of CQDs, potentially enhancing their functional performance. In this study, the N/S-CQDs exhibited notable antioxidant and antimicrobial properties. DPPH assays confirmed their free-radical-scavenging activity, whereas minimum inhibitory concentration (MIC) assays demonstrated inhibitory effects against *Escherichia coli* and *Listeria monocytogenes*. These findings highlight the potential of heteroatom-doped CQDs obtained from aquatic processing residues as multifunctional nanomaterials. Their antioxidant and antimicrobial activities may support applications in areas such as active food packaging, food preservation, antimicrobial surface coatings, biosensing, and other biotechnology-related applications (Salmas et al., 2024).

Shrimp shell waste has been successfully valorized as a carbon-rich feedstock for the hydrothermal synthesis of carbon quantum dots (CQDs). Treatment at 180 °C for 12 h produced CQDs with strong photoluminescence characteristics and a quantum yield of 27.14%. Their bioimaging capability was demonstrated in nematode models, where the particles localized within the cells and generated readily detectable fluorescence, allowing different structures of the organisms to be visualized. In addition, the CQDs exhibited effective antibacterial activity against both Gram-positive and Gram-negative bacterial strains (Elango et al., 2022).

Shrimp powder represents an inexpensive and easily accessible biomass source for the preparation of nitrogen-doped carbon quantum dots (N-CQDs). Hydrothermal treatment of this precursor resulted in nanosized N-CQDs with an average particle diameter of approximately 5 nm. The obtained nanomaterials displayed an emission maximum near 420 nm upon excitation at 350 nm and produced intense blue fluorescence under 365 nm ultraviolet irradiation. Their sensing performance was subsequently investigated for the detection of metal ions, with particular emphasis on Fe^{3+} . Among the metal ions examined, Fe^{3+} produced the most pronounced and selective fluorescence response, indicating the high sensitivity of the N-CQDs toward this ion. Moreover, the fluorescence response exhibited a consistent concentration-dependent variation within the 10–140 μM Fe^{3+} range. Based on these characteristics, the N-CQD-based sensing platform achieved a detection limit of 1.42 μM for Fe^{3+} . These findings demonstrate that shrimp-derived biomass can be valorized for the production of functional CQDs, enabling the development of fluorescent sensors for environmental and analytical applications (Wang et al., 2025).

Crab shell residues served as a biomass-derived precursor for the synthesis of carbon quantum dots (CQDs), which displayed a predominantly spherical particle morphology and emitted distinct green fluorescence under ultraviolet irradiation. In addition, the high sensitivity of these CQDs toward Cd^{2+} detection demonstrates that carbon quantum dots derived from shellfish waste can be utilized not only in bioimaging and antimicrobial applications but also in sensor systems for the detection of heavy metal ions (Elango et al., 2022).

6.3. Bivalve-Derived Biomass for Carbon Quantum Dot Synthesis

Marine shell waste has been shown to be an effective acid-neutralizing agent during lactic acid fermentation, suggesting that this approach can contribute to the sustainability of the fermentation process. Lactic acid production through the co-utilization of food and marine shell waste has been demonstrated to have a lower environmental impact than conventional approaches. Following fermentation, the resulting cellular biomass was recovered and used as a precursor for the synthesis of biomass-derived carbon quantum dots (Bio-CQDs). The obtained Bio-CQDs demonstrated excellent dispersibility in water, together with favorable photophysical characteristics, supporting their potential application in antibiotic sensing. This approach highlights the potential of marine shell-derived waste as a versatile feedstock that can contribute both to fermentation-based processes and to the sustainable generation of value-added nanomaterials. Thus, the conversion of waste biomass into functional products

represents a promising approach for reducing environmental burdens and developing more sustainable production processes (Mou et al., 2024).

Mussels have been explored as a marine biomass precursor for the environmentally sustainable preparation of carbon dots (CDs). CDs produced at reaction temperatures of 140, 160, and 180 °C showed temperature-dependent variations in their structural and optical characteristics. Notably, increasing the synthesis temperature from 140 to 180 °C reduced the mean particle size from 2.06 to 1.30 nm. The resulting CDs exhibited a maximum fluorescence quantum yield of 15.20%, while surface functionalities, including –OH, –NH₂, and –COOH, enhanced their aqueous solubility and biocompatibility. Their fluorescent properties enabled successful imaging of onion epidermal cells, HepG2 cells, and zebrafish, and the synthesized CDs also showed promise as biosensing materials for riboflavin determination. These findings indicate that mussels represent a suitable carbon source for the production of N-doped carbon dots with potential applications in bioimaging and riboflavin detection (Zhao et al., 2022).

Oyster shells were converted into carbon quantum dots (CQDs) through a single-step hydrothermal process following the drying and grinding of the shell material. During synthesis, ethylenediamine (EDA) served simultaneously as a nitrogen precursor and surface-passivating reagent, with the reaction conducted at 160 °C. The obtained oyster shell-derived CQDs exhibited a mean particle diameter of approximately 6.0 nm and a photoluminescence quantum yield of 1.4%. The CQDs exhibited distinct fluorescence emission at around 380 nm and high photostability under 365 nm UV irradiation. The biocompatibility of the oyster shell-derived CQDs was further investigated in HeLa cells, cardiomyocytes, and induced pluripotent stem cells (iPSCs) for potential cellular imaging applications. During the initial 24 h exposure period, the CQDs did not produce significant cytotoxic effects in the tested cell types. These findings demonstrate that oyster shell waste can be converted into fluorescent and biocompatible CQDs through an environmentally friendly approach and that the resulting nanomaterials have considerable potential, particularly for bioimaging applications (Abu et al., 2023).

6.4. Other Invertebrate Biomass for Carbon Quantum Dot Synthesis

Jellyfish biomass was used as a marine-derived precursor for the hydrothermal carbonization of fluorescent carbon nanodots (CNDs). The synthesized nanodots were comprehensively characterized by UV–Vis and fluorescence spectroscopy, Fourier-transform infrared spectroscopy (FTIR), differential scanning calorimetry (DSC), atomic force microscopy (AFM), transmission electron microscopy (TEM), and zeta potential measurements. FTIR spectra indicated the

presence of abundant C=O and C–O functionalities, whereas microscopic observations revealed predominantly spherical particles with diameters of approximately 50 nm. Furthermore, coating a nylon mesh with 1% CNDs resulted in a clearly visible fluorescent response under ultraviolet illumination, demonstrating the suitability of the synthesized material for fluorescence-based applications. Owing to these properties, jellyfish-derived CNDs have been suggested as potential materials for monitoring abandoned, lost, or otherwise discarded fishing gear (ALDFG) in oceans, as well as for investigating fish behavior through attractive or deterrent stimuli (Chinnadurai et al., 2022).

Sea cucumbers, as echinoderm-derived marine biomass, have attracted attention as alternative carbon-rich feedstocks for the preparation of carbon dots. Xu et al. (2026) synthesized marine-derived carbon dots (CDs) using a one-step hydrothermal method, employing six macroalgal species as well as two sea cucumber species, *Stichopus japonicus* and *Holothuria fuscogilva*, as carbon precursors. The products derived from *S. japonicus* and *H. fuscogilva* were designated as SJCDs and HFCDs, respectively. Carbon dots synthesized from the eight marine-derived sources were predominantly characterized by a spherical morphology, with particle dimensions of approximately 2 nm. In addition, the resulting nanomaterials maintained good colloidal stability when dispersed in aqueous media. This study is particularly significant in demonstrating that sea cucumber biomass can be directly utilized as a carbon precursor for the production of carbon dots (Xu et al., 2026).

6.5. Algal Biomass-Derived Carbon Quantum Dots

The marine macroalga *Eucheuma denticulatum*, which is widely distributed in marine environments, has been explored as a renewable biomass precursor for the synthesis of carbon quantum dots (E-CQDs). An environmentally benign chemical oxidation approach was employed to produce E-CQDs with particle dimensions ranging from 2.45 to 9.08 nm. The resulting nanomaterials showed considerable potential as fluorescent sensing platforms for the selective and sensitive determination of tetracycline (TC) in aqueous systems. The E-CQD-based sensor achieved a limit of detection (LOD) of 0.47 μM and a limit of quantification (LOQ) of 1.57 μM for TC, while its applicability was further confirmed through analyses of real samples. Fluorescence quenching studies indicated that the response toward tetracycline was mainly governed by dynamic quenching associated with the inner filter effect. The experimental findings and subsequent analyses supported this proposed mechanism. Overall, the results demonstrated that E-CQDs produced from *E. denticulatum* biomass through an environmentally benign approach can be effectively used for the rapid and

sensitive detection of tetracycline in aqueous systems and may have potential for environmental monitoring applications (Li et al., 2024).

Pelagic Sargassum biomass has recently attracted considerable interest as a promising feedstock for the sustainable production of value-added materials. Its suitability for carbon quantum dot (CQD) synthesis is largely associated with its abundant protein and carbohydrate constituents, including mono- and polysaccharides, as well as its widespread availability and low economic cost. Moreover, the relatively low environmental burden associated with processing this biomass further supports its potential as a sustainable precursor for CQD production. This approach also enables invasive algal biomass to be transformed from an environmental problem into high-value-added nanomaterials. The production of CQDs from sustainable biomass resources such as Sargassum represents an important research area, both for the valorization of environmental waste and for the development of greener approaches to the production of functional nanomaterials (Castañeda-Serna et al., 2022).

Green macroalgae offer a renewable biomass platform for the preparation of carbon quantum dots (CQDs) with potential applications in energy-related technologies. In one such study, *Halimeda opuntia* biomass was employed as the precursor for the biological synthesis of CQDs, followed by characterization of their structural, optical, and electrochemical characteristics. The synthesized nanomaterials exhibited notable fluorescence behavior together with favorable electrochemical properties. These findings suggest that *H. opuntia*-derived CQDs may serve as functional materials for energy storage and conversion technologies, particularly as electrode components in supercapacitor systems. This study demonstrates that marine green algal biomass can be utilized not only for environmental and biotechnological applications but also for the production of functional carbon nanomaterials with potential applications in sustainable energy technologies (Al-Ghamdi et al., 2023).

Nitrogen- and sulfur-enriched carbon quantum dots (CQDs) were prepared from polysaccharides isolated from the coralline alga *Corallina* sp., with *o*-phenylenediamine serving as an additional precursor. The resulting CQDs had an average particle diameter of 4.14 ± 0.70 nm and produced yellow-green fluorescence centered at 530 nm under excitation at 427 nm. Notably, their photoluminescence remained highly stable across a broad pH range, under elevated salt concentrations, and during prolonged exposure to ultraviolet radiation. When incorporated into gelatin to form composite films, the CQDs improved the mechanical strength, barrier performance, and antibacterial activity of the films, with the extent of improvement depending on the CQD concentration. Hemolysis and overall migration analyses further indicated

satisfactory biocompatibility of the developed films. Among the formulations tested, the film containing 3% CQDs provided the greatest protective effect for strawberries, reducing the spoilage rate to $20.32 \pm 1.62\%$ after 10 days of storage. These findings demonstrate that CQDs derived from marine algal polysaccharides can be utilized for the development of functional materials with biocompatible and antibacterial properties and may have considerable potential, particularly for active food packaging and extending the shelf life of food products (Wang et al., 2026).

Noise-induced hearing loss (NIHL) is a major health concern, especially among individuals chronically exposed to excessive occupational noise, yet effective pharmacological interventions for preventing or reversing noise-related auditory injury remain unavailable. Against this background, carbon dots (CDs) were prepared from eight marine-derived animal and plant sources using a single-step hydrothermal synthesis route. The obtained CDs generally displayed good physicochemical stability and notable free-radical-scavenging activity; among the materials investigated, CDs derived from *Undaria pinnatifida* Suringar (USCDs) provided the most pronounced protection against noise-induced auditory impairment. Structure–activity relationship analyses indicated that reducing functionalities and heteroatom-containing groups contributed substantially to the antioxidant and anti-NIHL effects of the CDs. Mechanistic investigations further suggested that USCDs attenuated both apoptosis and ferroptosis by promoting ROS elimination and Fe^{2+} chelation. In addition, these CDs showed favorable biosafety characteristics and were capable of crossing the blood–labyrinth barrier in mice, supporting their potential as marine biomass-derived candidates for the prevention of NIHL. These findings demonstrate that marine-derived CDs may have considerable potential for the treatment of inner ear disorders and for various biomedical applications (Xu et al., 2026).

7. Conclusion

The abundant organic residues generated by agricultural, food-processing, and aquatic activities represent promising carbon sources for CQD production, particularly because of their carbon-rich composition, widespread availability, and economic advantages. In particular, biomass sources such as macroalgae and microalgae, shellfish, and fish-processing residues represent suitable carbon feedstocks for CQD production because of their high contents of proteins, carbohydrates, and lipids. Beyond reducing the environmental burden associated with waste disposal, this approach provides an effective means of converting low-value residues into value-added nanomaterials while supporting sustainable production practices consistent with the principles of green chemistry.

Biomass-derived CQDs can be prepared through several carbonization-based routes, including hydrothermal and solvothermal treatments, microwave-assisted synthesis, and pyrolytic processes. The selection and adjustment of synthesis conditions allow the structural dimensions, surface functionality, optical response, and photoluminescence behavior of the resulting CQDs to be tailored according to the desired characteristics. However, the compositional characteristics of the starting material and the reaction parameters directly influence the physicochemical properties of the final product. Therefore, a comprehensive understanding of the interactions between feedstock characteristics and process conditions is critical for the standardization and commercialization of CQD production.

CQDs produced from waste-based precursors have attracted increasing interest due to their favorable biocompatibility, stable dispersion in aqueous environments, tunable luminescence characteristics, and diverse possibilities for surface functionalization. These advantages provide broad potential for applications ranging from biomedical diagnostic systems and environmental pollutant detection to photocatalytic remediation, energy storage, and imaging technologies. From a circular economy perspective, transforming discarded or underutilized materials into functional nanomaterials offers a valuable pathway for improving resource efficiency and promoting more sustainable technological development.

To translate laboratory-scale achievements into industrial-scale production, several challenges must be addressed, including improving reaction yields, maintaining consistent product quality across production batches, optimizing purification procedures, and reducing energy and chemical consumption. Further research is also needed to establish the long-term stability of these nanomaterials and to comprehensively assess their ecotoxicological characteristics and potential biological risks.

The utilization of organic waste from aquatic sources for CQD production represents an innovative approach that integrates waste management, environmental sustainability, and nanoscale materials engineering. Future studies focusing on the development of environmentally friendly synthesis routes, scale-up optimization, and application-specific integration strategies are expected to further clarify the scientific and industrial potential of biomass-derived CQDs.

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Transformation of Phosphorus Fractions in Waterlogged Calcareous Soils

Mehmet KARAGOKTAS¹, Veli UYGUR^{2*}

Abstract

Changing redox conditions strongly influence the solubility and stability of phosphorus minerals. In this study, the inter-fraction mobility of mineral phosphorus was investigated under reducing conditions in the presence of cotton plants. A pot experiment was conducted with three calcareous soils and five waterlogging durations (0, 7, 14, 21, and 28 days) applied 55 days after sowing, using a completely randomized design with five replicates. Excess water was drained to approximately field capacity, after which pH, electrical conductivity (EC), cations, anions, and soluble reactive phosphate (SRP) concentrations were determined in the drainage water. Following cotton harvest, sequential fractionation of soil phosphorus was performed to quantify NaOH-extractable P (NaOH-P), citrate–dithionite–bicarbonate extractable P (CDB-P), and HCl-extractable P (HCl-P). Results indicated that drainage water pH decreased while SRP concentrations increased markedly with waterlogging duration. The Ca:P molar ratio declined with increasing waterlogging time, whereas Fe:P, Mg:P, and Mn:P molar ratios increased up to the 21st day and subsequently decreased by the 28th day. NaOH-P consistently increased with waterlogging duration, while HCl-P increased in one soil but decreased in the other two, resulting in no significant overall correlation with waterlogging time. The CDB-P fraction exhibited soil-specific responses. Overall, the findings demonstrate that waterlogging or temporarily elevated water table levels induce reducing soil environments that stimulate the inter-fraction mobility of phosphorus. This process enhances P availability, which may have detrimental consequences for surface water quality through increased risk of eutrophication.

Keywords: Waterlogging, redox, phosphorus fractions, phosphorus release, calcareous soils

¹ PhD, mkaragoktas@gmail.com, orcid: 0000-0002-7823-6852, Süleyman Demirel University, Institute of Natural Sciences, 32260, Çünür, Isparta, Turkey

² Prof. Dr, veliuygur@isparta.edu.tr, orcid:0000-0003-3971-7714, Applied Sciences University of Isparta, Agricultural Faculty, Soil Science Department, 32260 Çünür, Isparta, Turkey

* Corresponding author

Introduction

Phosphorus (P) is an essential nutrient for plant growth, but its availability in soils can be limited, particularly in highly acidic or calcareous environments. In such soils, the distribution and mobility of P are influenced by a complex interplay of factors including soil pH, redox conditions, soil mineralogy, organic matter treatments, fertilizer management strategies, etc. (Yin et al., 2018; Fahmi et al., 2023; Palihakkara et al., 2024). Waterlogged calcareous soils, characterized by high calcium carbonate (CaCO_3) content, are prone to periodic saturation with water under improper irrigation practices and/or topography-driven flood events, as occurred in the study area. Seasonal water-table fluctuations are critical soil conditions influencing phosphate solubility due to changing soil properties. For example, waterlogged or flooded calcareous soils behave differently than acid/paddy/wetland soils under submergence at different times (Ponnamperuma, 1972; Ma et al., 2010; Amarawansha et al., 2015; Jayarathne et al., 2016).

In semi-arid regions, even a high water table can cause a reduction of deeper soil layers and increase or decrease the availability of some plant nutrients (Ponnamperuma, 1984; Sposito, 2016; Fageria et al., 2011). Although P is not a redox element, its availability and solubility are influenced by changing soil properties under a reduced environment (Scalenghe et al., 2012; Amarawansha et al., 2015; Ylivainio et al., 2017). The direction of this response, however, is not uniform: reductive dissolution of Fe and Mn oxides generally enhances P solubility in flooded soils (Khalid et al., 1977; Shenker et al., 2005; Amarawansha et al., 2015), whereas in other soils the precipitation of Fe compounds and the formation of new sorption surfaces can lower P solubility. While increasing phosphate concentration of soil solution causes eutrophication in surface waters, decreasing concentrations lead to lesser plant uptake and yield losses.

Solubility of phosphorus compounds increases with decreasing soil pH and redox potential as a function of waterlogging time in calcareous soils; subsequently, highly reactive phosphates were re-sorbed by newly formed Fe^{2+} compounds such as vivianite or amorphous Fe compounds (Amer et al., 1991; Sanyal and De Datta, 1991; Ma et al., 2010; Amery and Smolders, 2012). Nutrient availability in waterlogged or flooded natural ponds and paddy soils has been investigated by many researchers (Ma et al., 2010; Amarawansha et al., 2015; Jayarathne et al., 2016). More recent work has shown that redox-sensitive inorganic and organic P forms dominate the P budget of flooded soils (~50% and ~18% of total P, respectively), with organic P dynamics playing a considerable role in P availability under fluctuating redox conditions (Menezes-Blackburn et al., 2017; Martinengo et al., 2025). Anoxic conditions can also mobilise colloidal P at pore-water concentrations exceeding soluble P by more than 2.7-fold,

extending P transport beyond the dissolved phase (Eltohamy et al., 2024). In parallel, climate-change projections of more frequent and intense wet events point to an increasing risk of P losses from agricultural land through waterlogging and flooding, with predicted increases in P loads of up to 30% under future climate scenarios (Ockenden et al., 2017; Tong et al., 2021; Djodjic et al., 2025) and landscape-scale water storage has been proposed as a measure to mitigate such flood-related nutrient losses. However, studies on P behaviour in waterlogged calcareous soils of semi-arid regions during active crop cultivation, typically occurring in the sampled sites due to excessive irrigation, are scarce. In this research, we conducted a greenhouse pot experiment with the cotton plant to investigate the behaviour of phosphorus in short to medium-duration waterlogged calcareous soils.

Materials and Methods

Site description and soil sampling

Soil samples were taken from different soil series of Harran Plain, Şanlıurfa, Turkey, where the water table is high or near the soil surface due to excessive irrigation in the region. Begdes (Entisol, Lithic Torriorthent), Akcakale (Vertisol, Typic Torrert), and Harran (Aridisol, Vertic Calciorthid) soil series have formed on alluvial parent materials (Dinç et al., 1991). The experimental soils have deep soil profiles with high calcium carbonate equivalent and clay content.

Clay mineralogy of the soils was reported as follows: Beğdes — smectite, chlorite, palygorskite, illite, kaolinite, and quartz; Akçakale — smectite, chlorite, palygorskite, illite, quartz, and feldspar; Akçakale — smectite, chlorite, palygorskite, illite, kaolinite, quartz, and calcite (Çakmaklı, 2008).

Composite surface soil samples were taken from farmer fields and passed through 8 mm and 2 mm sieves for pot trial and descriptive soil analysis, respectively.

Description of soil analyses

Soil pH and EC were determined in the saturation paste (Thomas, 1996; Rhoades, 1996). Organic matter content, according to the modified Walkley-Black method stated by Nelson and Sommers (1996), carbonate content with the Scheibler calcimeter reported by Loeppert and Suarez (1996), and soil texture with a hydrometer method described by Bouyoucos (1962) were determined. The sum of exchangeable and soluble cations was determined in 1 M NH₄OAc extracts with ICP-OES (Helmke and Sparks, 1996). The total element concentrations of soils were measured in aqua-regia digest (3:1 HNO₃ and HCl, V/V) with ICP-OES (Loeppert and Inskeep, 1996). Amorphous iron oxide

contents of soils were determined in two steps: i) carbonates were removed by employing 1 M sodium acetate at pH 5, ii) the residues were then shaken with 0.175 M ammonium oxalate and 0.1 M oxalic acid mixture for 4 h in darkness. Subsequently, the crystalline iron oxides were solubilized in a water bath at 80°C utilizing a 0.175 M ammonium oxalate + 0.1 M oxalic acid + 0.1 M ascorbic acid mixture whose pH was adjusted to 2.25 in centrifuge tubes which were used for the previous extraction (Shuman, 1988). The results were presented in Table 1.

Table 1. Descriptive physicochemical properties of the experimental soils

Series	pH	EC	OM	ΣN	CCE	ACC	Sand	Clay	FC	WP	BD	AFe	CFe
		μS cm ⁻¹					g kg ⁻¹				g cm ⁻³	g kg ⁻¹	
Harran	7.63	807	9.60	0.51	234	193	140	590	291	214	1.23	1.02	15.9
Akçakale	7.78	955	16.2	0.85	193	181	163	529	332	238	1.24	1.03	15.3
Beğdeş	7.74	982	9.40	0.49	183	174	158	545	291	204	1.35	1.25	20.9

Series	Ca	Mg	Na	K	CEC	P	Cu	Fe	Mn	Zn	ΣFe	ΣMn	ΣAl	ΣP
	cmol kg ⁻¹							mg kg ⁻¹			g kg ⁻¹			
Harran	33.5	6.82	0.39	1.98	46.6	17.6	1.76	4.97	15.3	2.96	31.3	1.08	23.3	0.09
Akçakale	28.2	9.87	2.20	1.86	47.2	7.30	1.79	4.36	4.97	1.25	32.5	0.99	24.0	0.12
Beğdeş	40.4	7.95	0.52	1.36	49.3	8.95	1.56	5.66	17.3	0.97	38.4	1.13	26.1	0.10

OM organic matter, CCE calcium carbonate equivalent, ACC active calcium carbonate, FC field capacity, WP wilting point, BD bulk density, AFe amorphous Fe, CFe cristalline Fe, CEC cation Exchange capacity

Experimental set-up

A pot experiment was established using three soils and five waterlogging durations in a completely randomized design with five replicates. Pots were filled with 20 kg of air-dried soil samples, and cotton (*Gossypium hirsutum* L.) seeds were sown in May, coinciding with the local sowing period in the Harran Plain. Basal phosphorus and nitrogen fertilizers were applied at sowing; ammonium sulfate and diammonium phosphate were used to supply 100 mg N kg⁻¹ and an availability of 25 mg P kg⁻¹, respectively. Irrigation was provided as required using a drip system. The chemical properties of the irrigation water are presented in Table 2.

Table 2. Descriptive properties of irrigation water

pH	Ca	Mg	K	Na	EC	P	B	S	Cl	NO ₃	HCO ₃
	mg L ⁻¹				μS cm ⁻¹	mg L ⁻¹					
8.00	35.2	14.7	1.97	16.2	403	0.17	0.096	11.2	1.68	2.27	2.95

Waterlogging of pots

Cotton plants were subjected to waterlogging for 7, 14, 21, and 28 days, with a 5 cm water layer maintained at the soil surface beginning at the onset of the blooming stage (55 days after sowing). This treatment simulated the common practice of farmers in the Harran Plain, with evaporative losses replenished daily

(Figure 1B). A non-waterlogged control was also included. At the end of each waterlogging period, pots were drained through a large funnel (Figure 1C), and the drainage water was collected for laboratory analysis of pH, electrical conductivity (EC), and concentrations of Ca, Mg, Fe, Mn, P, SO_4^{2-} , HCO_3^- , and CO_3^{2-} . Drained pots were subsequently maintained for cotton growth until full maturation (Figure 1D). After harvest, soil samples were collected from each pot to determine changes in geochemical phosphorus fractions. Prior to analysis, samples were air-dried.

Phosphate fractionation scheme

According to the sequential fractionation scheme of Kuo (1996), the three P fractions were determined on the same air-dried sample: (i) sodium hydroxide extractable phosphorus was determined in the supernatant obtained by 17 h shaking of 1 g soil with 50 mL of 0.1 M NaOH + 1 M NaCl mixture; (ii) citrate-bicarbonate-dithionite extractable or reducible P was solubilized from the residue with 0.3 M citric acid + 1 M NaHCO_3 and 1 g of $\text{Na}_2\text{S}_2\text{O}_4$ in a water bath at 80°C; (iii) acid-extractable phosphorus was determined in the filtered supernatant of the remaining residue shaken with 0.5 M HCl for 1 h. Sequential chemical fractionation is an operationally defined approach; the assignment of extracted P to specific mineral phases (e.g., Fe/Al-associated P, reducible P, Ca-P) is therefore an approximation of the underlying geochemical pools. Spectroscopic confirmation of mineral phases (e.g., ^{31}P NMR or P K-edge XANES) was beyond the scope of this study, and the phase assignments discussed below should be interpreted with this limitation in mind.

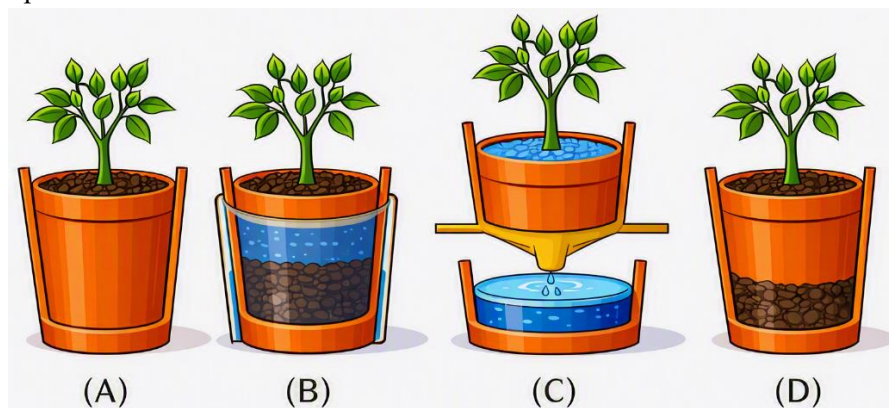


Figure 1. Waterlogging and drainage system of pots. A) control or before waterlogging, B) double-potted waterlogging, C) drain system, and D) post-drained pot

Statistical analyses

The effects of waterlogging time on the soils' phosphorus fractions and phosphate release were determined with ANOVA, and Duncan's multiple range test was used to compare the treatment means at $p < 0.05$ using the SPSS 17 statistics program. In addition, Pearson correlation was used to determine relationships among the analysed parameters. Correlation coefficients were calculated using the 60 samples from the five waterlogged treatments including the controls (4 durations $\times$ 3 soils $\times$ 5 replicates).

Results and Discussions

Initial soil properties

The studied soils in pot experiments may be classified as calcareous and clay-textured. Very high amounts of active lime ($174 \text{ g kg}^{-1} <$) and clay contents ($529 \text{ g kg}^{-1} <$) make difficult to manage P in these soils. The problem further complicated by low organic matter contents which are very similar for Harran and Begdes soils. Ammonium acetate extractable Ca is the highest in Begdes soil and the lowest in Akcakale soil (Table 1). Ammonium acetate extractable Mg and Na are the highest in Akcakale soil and the lowest in Harran soil, and K amounts are similar for the three soils. The extractable sodium and sulfate contents of Akcakale soil behaved differently than other soil series during all treatments. Some researchers stated that high sulfate contents in soils could delay reaching a negative Eh value and the reduction of Fe compounds slows down as a function of the amounts of sulfate (Ponnamperuma, 1972; Kozłowski and Pallardy, 1984; Sposito, 2016). Therefore, those soils' reduction and mineral dissolution differ from their experimental counterparts. In addition, Begdes soil, whose amorphous and crystalline iron contents are higher than those of the other soils, contains smectite-type clay minerals. Amarawansha et al. (2015) stated that the reduction of calcareous soils containing smectite clay was delayed depending on the clay type and content.

Drainage water's pH

Drainage water's pH decreased due to increased waterlogging time for the experimental soils (Figure 2a). pH values shifted towards neutrality after 14 days for Begdes and Harran soils, and the lowest pH decrease occurred in the Akcakale soil. Waterlogging time and pH of drainage waters have significant negative correlations ($p < 0.01$) for the soils (Table 3). The researchers explained the pH decrease under anoxic conditions in drainage waters or soil solutions differently. Some researchers stated that the decomposition of organic matter by anaerobic

bacteria produces CO₂, which decreases soil pH by forming carbonic acid (Ponnamperuma, 1972; Shenker et al., 2005; Amarawansha et al., 2015; Jayarathne et al., 2016), the others stated that decrease of soil pH was taken place according to organic acid producing by anaerobic bacteria during the anaerobic decomposition of organic matter (Craft, 2001; Oberson and Joner, 2001; Stevens and Tullos, 2011). Regardless of the exact mechanism, the released H⁺ ions resulted in the dissolution of carbonate minerals or reacted with evolved CO₂ and water, both producing HCO₃⁻ near a neutral pH in calcareous soils (Lindsay, 2001). Therefore a positive correlation between the pH and HCO₃⁻ in drainage water was evident (Table 3).

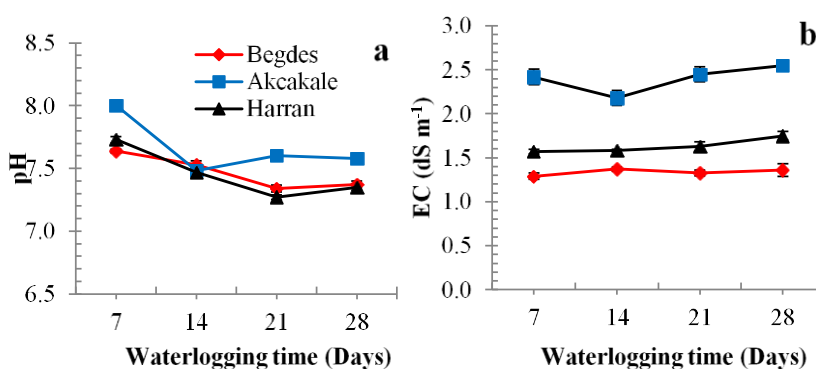


Figure 2. The effects of waterlogging time on the changes in drainage waters' pH (a) and EC (b)

Drainage waters' EC

Drainage waters' ECs were found statistically insignificant according to increasing waterlogging time for the soils (Figure 2b). Soil solution or drainage water ECs depicted the same trend for Begdes and Harran soils and a different trend for Akcakale soil, whose EC was approximately twofold higher than that of the other soils. The EC and waterlogging time did not have any significant correlations ($p < 0.05$) for the experimental soils (Table 3). Ponnamperuma (1972) stated that EC values of soil solutions of flooded soils increase in the first couple of weeks. The increase in EC of flooded soils during flooding as explained by Ponnamperuma (1972, 1984) with the ascended concentration of NH₄ and HCO₃ due to anaerobic decomposition of organic matter, Mn²⁺ and Fe²⁺ ions due to reduction of sesquioxides. In contrast, Amarawansha et al. (2015) and Jayarathne et al. (2016) stated that the rise of EC value in flooded calcareous soils resulting from increased solubility of Ca and Mg compounds with decreased soil pH and reduction of Fe and Mn compounds at anaerobic conditions. Ponnamperuma

(1972 and 1977) stated that ECs of flooded soils increase sharply within four days of submergence and decrease rapidly until eight days, and after that, a decrease occurs slowly. Therefore, the effective change in EC values occurs within eight days of flooding, which corresponds to our first drainage water sample on the 7th day.

Table 3. Bi-variate Pearson correlation coefficients among the ionic composition of drainage water and the phosphorus fractions (N=60)

	pH	EC	SRP	Ca	Mg	Mn	Fe	CO ₃	HCO ₃	SO ₄	NaOH-P	CDB-P	HCl-P
Time	-0.645**	0.089	0.225	-0.473**	0.058	0.408**	0.737**	0.467**	0.301*	-0.816**	0.748**	-0.110	-0.178
pH		0.450**	0.386**	0.204	0.445**	-0.731**	-0.550**	-0.074	0.265*	0.757**	-0.762**	0.096	0.535**
EC			0.941**	-0.081	0.976**	-0.464**	0.096	0.545**	0.798**	0.347**	-0.289*	0.457**	0.865**
SRP				-0.087	0.955**	-0.411**	0.144	0.499**	0.891**	0.223	-0.152	0.418**	0.796**
Ca					-0.048	0.090	-0.520**	-0.529**	-0.234	0.478**	0.029	0.309*	0.143
Mg						-0.470**	0.053	0.521**	0.827**	0.361*	-0.295*	0.511**	0.891**
Mn							0.411**	-0.131	-0.336**	-0.532**	0.691**	-0.077	-0.509**
Fe								0.595**	0.216	-0.682**	0.467**	-0.264*	-0.214
CO ₃									0.412**	-0.295*	0.046	0.003	0.345**
HCO ₃										0.045	-0.162	0.353**	0.621**
SO ₄											-0.723**	0.231	0.593**
NaOH-P												0.022	-0.402**
CDB-P													0.570**

EC electrical conductivity, SRP standard reactive phosphorus, NaOH-P moderately plant-available P, CDB-P reducible P or Fe-P; HCl-P sparingly soluble P or Ca-P

Reactive cation (Ca, Mg, Fe, and Mn) concentrations of drainage waters

Drainage water Ca concentrations decreased as a function of waterlogging time (Figure 3a) and have significant negative correlations ($p < 0.01$) with submergence duration (Table 3). Considering the decreasing soil pH, an increase in the Ca concentration in the equilibrium solution would be expected. However, the reducing condition enriched the concentration of Fe, Mn, and other metallic microelements (Ponnamperuma, 1972; Amarawansha et al., 2015; Jayarathne et al., 2016; Ören et al., 2018) that undergo acid-base reaction with carbonate surfaces (Uygur, 1998), especially those in the active lime fractions. These reactions cover the surface of carbonate minerals so that they no longer control the solution Ca (Uygur and Rimmer, 2000). Furthermore, montmorillonite-type clay minerals prefer Ca over monovalent cations. In conjunction with surface precipitation, these processes can reduce the calcium concentrations of the drainage water. The third mechanism could be the precipitation of the excess Ca with CO_3 produced from the anaerobic mineralization of organic matter (Ponnamperuma, 1984). The presence of phosphate increases the solubility of calcium carbonate, and that of calcium phosphate is increased by bicarbonate (Greenwald, 1945). Our results revealed that Ca concentrations of drainage waters have significant negative correlations ($p < 0.01$) with HCO_3^- and CO_3^{2-} anions (Table 3), indicating initial calcium carbonates maintained carbonate equilibria and subsequent adsorption of Ca by clay minerals. The aforementioned mechanisms explain why a decrease occurred in Ca concentrations of drainage waters.

Magnesium concentration didn't change with waterlogging time (Figure 3b). Magnesium concentrations in waterlogged acid soils increase according to waterlogging time because of the dissolution of Mg compounds or exchange of Mg from the surface of soil colloids by ions which are produced under reducing conditions such as Fe, Mn, and NH_4^+ (Ponnamperuma, 1972; Ann et al., 2000; Amarawansha et al., 2015; Jayarathne et al., 2016). Interestingly, Mg concentrations and ECs have a similar trend for the three soils and Mg concentration and EC have positive ($p < 0.01$) significant correlations (Table 3). This behavior suggested that i) a higher Mg buffer capacity of the experimental soils, ii) excess Ca displaced the Mg from the exchange sites, iii) richness of the soils with Mg carbonates, and Ca-carbonates were the primary components buffering to H ions generated, and iv) surface precipitation reactions mentioned above (see Ca results) also operated.

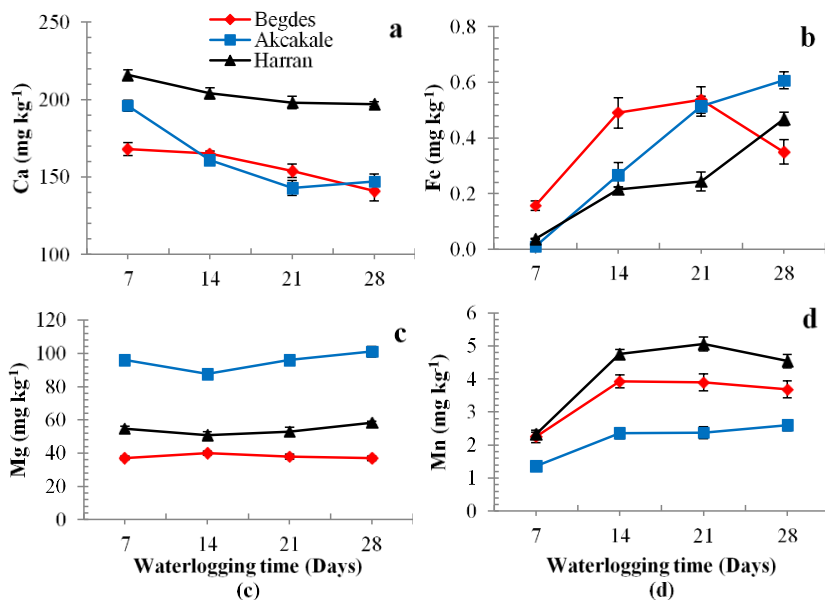


Figure 3. The reactive concentration of the cations in drainage water of the experimental soils

Iron and Mn concentrations of drainage waters, rose with increasing waterlogging time (Figure 3c,d) and have significant positive correlations ($p < 0.01$) with waterlogging time (Table 3). Iron and Mn, both of which were redox-sensitive elements increase concentrations of reduced Fe^{2+} and Mn^{2+} ions in soil solutions (Shahandeh et al., 2003; Sposito, 2008). Manganese compounds are much more sensitive to reduced conditions and dissolve at a relatively higher redox potential than Fe compounds; the critical redox potential at which Mn^{2+} first appears in solution is approximately 200 mV, whereas Fe^{2+} appears at approximately 100 mV (Patrick and Jugsujinda, 1992). Thus, the Mn concentration of drainage water rose within 14 days of waterlogging and decreased between 14 and 28 days of the experiment. The reduction of the Fe compounds occurs after the exhaustion of redox-sensitive compounds, and Fe concentration is reported to peak between 14-60 days of waterlogging duration (Ponnamperuma, 1984; Ören et al., 2018). Manganese and SRP concentrations of drainage water have negative significant ($p < 0.01$) correlations (Table 3), and Mn concentrations of drainage waters decreased as phosphates dissolved. Amer et al. (1991) stated that phosphates precipitate as $\text{MnPO}_4 \cdot 1.5 \text{H}_2\text{O}$ at high redox potential ($p_e + \text{pH} > 11$) and as $\text{Fe}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ at low redox potential ($3.4 < p_e + \text{pH} < 6.0$). Shahandeh et al. (2003) point out that the dissolution of Mn

compounds releases phosphates that occur before Fe compounds, where oxide compounds are high in soils.

Iron concentrations of drainage waters are low in the early days of waterlogging and rise according to increasing waterlogging time. Gotoh and Patrick (1974) stated that the threshold of Fe reduction is 100 mV. Amarawansha et al. (2015) suggest that a 100 mV redox potential may be observed within 3 weeks of waterlogging for calcareous soils. Dissolved Fe^{2+} ions can precipitate as Fe phosphates such as vivianite (Heiberg et al., 2010). However, this situation were likely to occur only in Akcakale soil on the 28th day of the experiment (Figure 3d).

Reactive anion (SRP, HCO_3 , CO_3 and SO_4) concentrations of drainage waters

SRP concentrations of drainage waters responded to increased waterlogging time in all three soils; however, the increasing effect started on the 28th day of waterlogging (Figure 4a). The release of phosphate from the soil matrix differs between soils according to total P and other soil properties such as clay type and amorphous iron content. The highest P release occurred in Akcakale soil, whose total P content is higher than that of the other two soils. The lowest P release was determined from Begdes soil, which has more amorphous iron and smectite-type clay minerals; these properties may have delayed the onset of reductive dissolution.

SRP concentrations of soil solutions have significant negative correlations ($p < 0.01$) with solution Mn concentration and significant positive correlations ($p < 0.01$) with CO_3 and HCO_3 concentrations (Table 3). In addition, HCO_3 and CO_3 concentrations in drainage waters have a significant positive correlation ($p < 0.01$) with waterlogged time. This result indicates that an increase in HCO_3 and CO_3 concentrations of drainage waters took place due to the decomposition of organic matter under anaerobic conditions (Figure 4c,d), as reported by Shenker et al. (2005) and Amarawansha et al. (2015), or dissolution of carbonate minerals. The production of HCO_3 and CO_3 may result in the dissolution of easily soluble Ca-P compounds, such as the NaHCO_3 -extractable Olsen-P pool, and the accompanying decrease in soil solution pH increased the SRP concentration of drainage waters (Palihakkara et al., 2024). Ann et al. (2000) and Racz (2006) stated that pH in alkaline soils decreased depending on the waterlogging-duration-induced dissolution of phosphate compounds such as apatite, β -tricalcium phosphate, octacalcium phosphate, brucite, and monetite. These phosphate minerals can increase the soil solution's phosphate, calcium, and magnesium concentrations (Radmann et al., 2023). Similarly, Amarawansha et

al. (2015) and Jayarathne et al. (2016) pointed out that Ca, Mg, Fe, and Mn concentrations of soil solution rose with increased waterlogging time. The rise of these elements' concentrations results from the dissolution of Ca-P compounds due to decreasing soil pH and the dissolution of Fe-Mn compounds at decreased redox potential (Gotoh & Patrick, 1974).

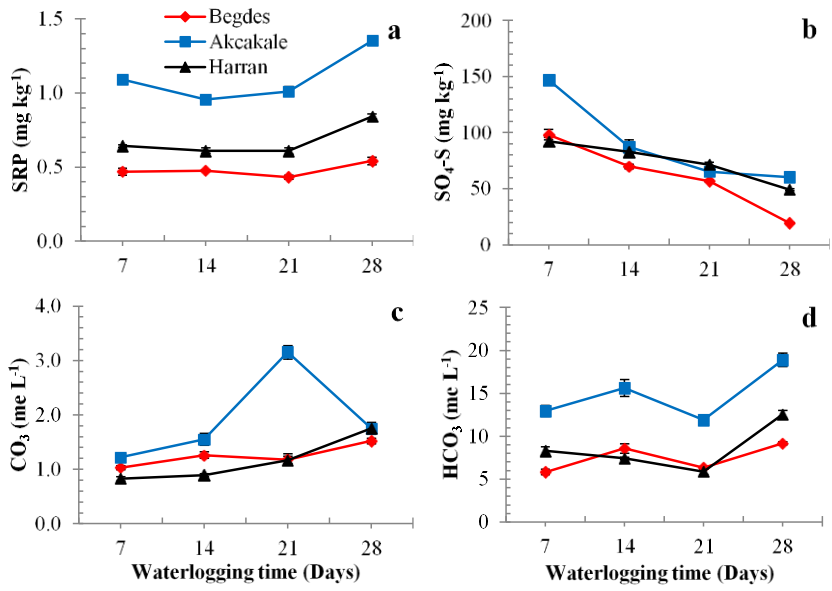


Figure 4. Reactive anion concentrations of drainage waters

The sulfate concentration of drainage waters decreased with increasing waterlogging time (Figure 4b) and had a significant negative correlation ($p < 0.01$) with waterlogging time (Table 3). Sulfate is an indicator of the reduction of soil under waterlogged conditions: SO_4^{2-} acts as an electron acceptor in the redox sequence after NO_3^- and is reduced at redox potentials near or below 0 mV (Ponnamperuma, 1972, 1984). Although Eh was not measured directly, the progressive decline in the SO_4 concentration of the drainage water indicates that the redox potential of the soils fell to values at which Fe compound dissolution occurs (critical Fe-reduction potentials range from +100 mV at pH 7 to -100 mV at pH 8) (Gotoh & Patrick, 1974). Iron phosphates exist in trivial amounts in alkaline soils because of the low ionic activity of Fe at high soil pH (Uygun and Karabatak, 2009; Bair, 2012); however, the adsorptive properties of Fe oxyhydroxides increase the amount of Fe-P in any soil (Bilgili et al., 2008; Mahdi et al., 2024). Therefore, Fe-P in waterlogged soils dissolved due to decreased redox potential, and this increased SRP concentrations of the soil solution (Ann et al., 2000; Scalenghe et al., 2012; Amarawansa et al., 2015). However, Fe-P

such as vivianite may occur under elevated Fe and PO_4 concentrations induced by the reduced conditions, and these compounds precipitate during the later stages of waterlogging.

Molar ratios of reactive cations and SRP

Molar phosphate ratios with Ca, Mg, Fe, and Mn, ions which are considerably high in drainage water, behaved differently. While the Ca:P molar ratio in drainage water decreased with waterlogging time (Figure 5a), the Fe:P, Mg:P, and Mn:P ratios increased on the 14th and 21st days of waterlogging; however, their ratios reduced on the 28th day of waterlogging. This situation revealed that during the first 21 days of waterlogging, phosphate compounds of Ca, Mg, Mn, and Fe released P, which then precipitated as Ca-P compounds where the Ca:P ratio graph consistently decreased (Figure 5a), the thermodynamically most stable mineral phase at neutral to alkaline pH values. The reducing conditions induced the dissolution of Mn and Fe compounds in flooded soils and released Fe, Mn, and PO_4 ions (Ajmone-Marsan et al., 2006; Scalenghe et al., 2012). The increased phosphate concentrations resulted in the precipitation of phosphate compounds such as Ca-P and Fe-P. Shenker et al. (2005) stated that Ca-P compounds control the dissolution and precipitation of phosphates in flooded calcareous soils. However, increasing waterlogging time altered the behaviour of the Fe/P relation, and phosphates may have been precipitated as Fe-P compounds towards the 28th day of waterlogging for Harran and Begdes soils (Figure 5b). This situation was previously stated by Khalid et al. (1977) and Amer et al. (1991). In addition, the formation of Mn-P compounds is negligible in the experimental soils, as pointed out by the negative correlation ($p < 0.01$) between Mn and SRP concentrations of drainage waters; the Mn compound is thermodynamically unstable under a reduced soil environment (Lindsay, 2001). Similarly, Amarawansa et al. (2015) stated that dissolved reactive phosphate and Mn ions of the pure water of waterlogged calcareous soils have a positive significant ($p < 0.01$) determination coefficient at linear regression analyses, despite the very high affinity of Mn oxides for P under oxic conditions. Consistent with this, recent incubation studies reported that waterlogging decreased soil Eh by 155 mV relative to field capacity and increased water-extractable P from 1.47 to 2.58 mg kg⁻¹, supporting the interpretation that reductive dissolution of Fe and Mn compounds was the driver of P release in the present experiment.

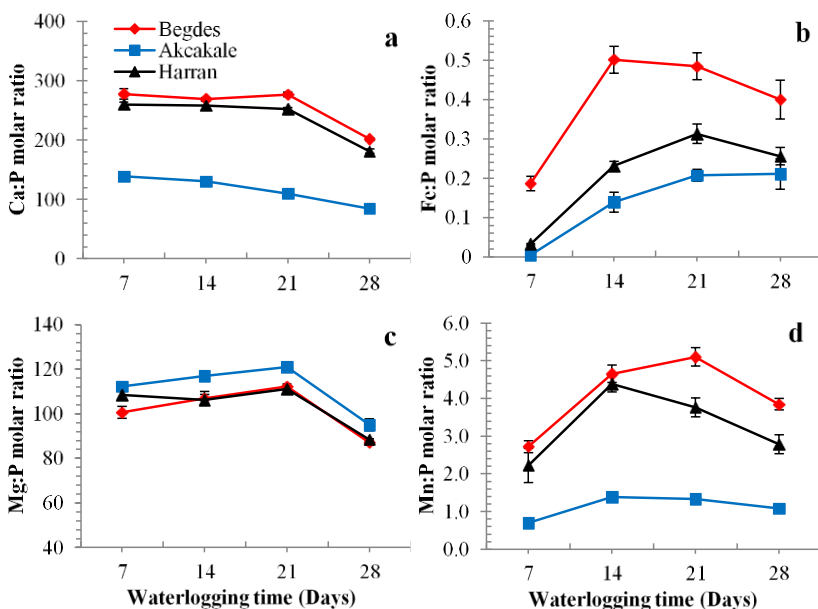


Figure 3. Molar ratios of dissolved cations and SRP in drainage waters

Phosphorus fractions

NaOH-soluble P fraction: NaOH-soluble P is a moderately plant-available P fraction associated with Fe/Al oxide hydroxyl surfaces. This potentially plant-available phosphorus fraction of the soils rose with increasing waterlogging time, and this change was statistically significant (Figure 6a, Table 3). An increase in the magnitude of NaOH-P indicated the dissolution of reducible Fe-P compounds and of Ca-P compounds, followed by adsorption on the Fe/Al oxides or reprecipitation of released P as dicalcium phosphate or dicalcium phosphate dihydrate. These forms are easily soluble Ca-P compounds (Lindsay, 2001). Shenker et al. (2005) stated that Ca-P compounds control phosphates released under reduced conditions. It was pointed out that the DCP content of sequentially reduced and oxidized rice soils increases under waterlogging conditions, resulting from the reducible Fe-P compounds (Fahmi et al., 2023). Similarly, Shah et al. (2010) reported that added P fertilizer was adsorbed as Fe-P compounds in aerobic surface soils and converted to Ca and Mg-P compounds in anaerobic subsurface alkaline waterlogged soils. However, the vivianite content of P-fertilized waterlogged soils increased due to the dissolution of Fe-Ps and reprecipitation as vivianite mineral (Sanyal and De Datta, 1991; Eshun et al., 2024). In the present study, the formation of specific Fe phases could not be confirmed without spectroscopic analysis, but the molar Fe:P trends in the drainage water were consistent with this mechanism. Quintero et al. (2007) stated

that the NaHCO_3 - and NaOH -extractable phosphate content of soils waterlogged for 45 days increased compared to the control soil, with the NaOH -extractable P fraction being predominant over NaHCO_3 -extractable P. Several researchers reported that NaOH extracts not only Fe-P, Al-P, and organic P compounds but also readily soluble Ca-P such as DCP (Hedley et al., 1982; Kuo, 1996; Villapando & Graetz, 2001; Williams et al., 1971). Therefore, it can be concluded that upon P fertilization, Ca-P and Fe-P fractions of the waterlogged soils increase as a function of increasing waterlogging time.

CBD-extractable P fraction: The CBD-extractable P is regarded as a moderately soluble and reducible phosphorus fraction. The CBD-P of the soils rose for Begdes soil with increasing waterlogging time, whereas there was an increase for the first 14 days of waterlogging for Harran and Akcakale soils, followed by a decreasing trend until 28 days. These changes were statistically significant (Figure 6b). At the same time, the CBD-P content of soils had a significant positive ($p < 0.01$) correlation with the SRP concentration of drainage water and a significant negative correlation ($p < 0.05$) with the Fe concentration of drainage waters (Table 3). These results revealed that a certain amount of dissolved SRP and added P fertilizer reprecipitated as Fe-P compounds or were adsorbed by Fe oxyhydroxides, increasing the soil CBD-P fraction. The CBD-P content of waterlogged soils changed according to the reduction level of soils, and the evaluation of soils can be separated into two groups. The 14th day of waterlogging was a milestone for the dissolution and adsorption of P by Fe compounds. The first 14 days of waterlogging increased the CBD-P of the experimental soils; this situation resulted from the adsorption of fertilizer-P by Fe oxyhydroxides and the dissolution of readily soluble P compounds. This behaviour of Harran and Akcakale soils was much more apparent. Increasing reduction level increased the dissolution of Fe compounds, and the CBD-P content of the two experimental soils decreased.

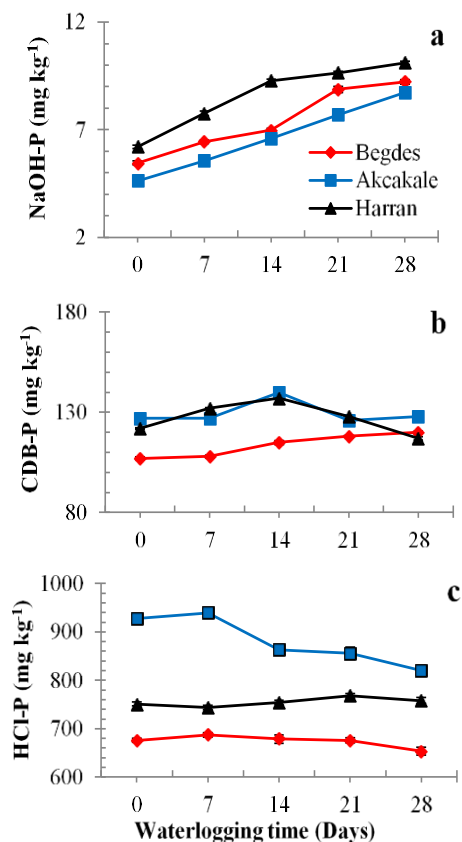


Figure 6. The effect of waterlogging time on the NaOH-P (a), CBD-P (b), and HCl-P (c) fractions.

However, the CBD-P content of Begdes soil, whose amorphous Fe content is higher than that of the other experimental soils, rose during the entire waterlogging period. If we co-consider the CBD-P content of soils and the SRP concentration of drainage water: (I) the CBD-P content of Begdes soil continuously increased up to the 28th day of waterlogging, revealing that dissolved P and added fertilizer P reacted with Fe oxyhydroxides; and the SRP content of Harran and Akcakale soils decreased until the 21st day of waterlogging and rose after that day, which implies the dissolution and re-adsorption of added P fertilizer and released P by Fe compounds. The reduction of Fe compounds takes place around 100 mV redox potential, which occurs after three weeks of flooding in calcareous soils (Gotoh and Patrick, 1974; Amarawansha et al., 2015). This result explains the increase of CBD-P content of Harran and Akcakale soils in the first 14 days of flooding. However, the high amorphous Fe content of Begdes soil increased buffer capacity, and a decrease in the CBD-P content was

not observed. Also, the SRP content of Begdes soil rose until the 28th day of waterlogging; until then, there was no statistically significant change in the SRP content of Begdes soil. As observed in this study, Willett (1986) related the P concentration of waterlogged soils to submergence duration and amorphous Fe oxides. Amer et al. (1991) determined that an increase in the amount of vivianite [$\text{Fe}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$] resulted from the reduced Fe oxides and fertilizer P. Villapando and Graetz (2001) stated that Fe/Al-P content of subsurface layers of heavily manured soils increased with flooding. Phosphorus fertilization for 14 years to paddy soils also led to phosphorus accumulation (Shen et al., 2004). All of the aforementioned literature is in line with the current findings.

HCl-P soluble fraction: Recalcitrant phosphorus fractions of the soils increased in Harran soil and decreased for Begdes and Akcakale soils due to waterlogging duration (Figure 6c). In addition, the HCl-P content of soils had a significant positive correlation ($p < 0.01$) with the SRP concentration of drainage water and a significant negative correlation ($p < 0.01$) with the NaOH-P content of soils.

The HCl-P fractions of the soils behaved differently as a function of soil properties and waterlogging time. Typically, the HCl-P content of calcareous soils increases after P fertilization (Halajnia et al., 2009; Jalali & Ranjbar, 2010; Wang et al., 2010). However, the HCl-P content of waterlogged calcareous soils increases or decreases depending on soil properties (Villapando & Graetz, 2001; Ma et al., 2010). The reduction in the HCl-P fraction of Akcakale and Begdes soils increased the NaOH-P and CBD-P fractions. The NaOH-P and HCl-P had significant negative ($p < 0.01$) correlations, which indicate inter-fraction mobility induced by excess water. Although HCl-P decreased in Begdes and Akcakale soils, it increased in Harran soil over the waterlogging period, and the overall correlation between HCl-P and waterlogging time was not significant ($r = -0.178$, $p > 0.05$; Table 3); the opposing soil-specific trends thus offset each other, and the fractional decrease observed in two of the three soils was accompanied by net accumulation of this fraction in Harran soil.

A decreasing pH-induced dissolution of octacalcium phosphate, β -tricalcium phosphate, hydroxyapatite, and fluorapatite was reported (Ponnamperuma, 1985). Similarly, the saturation index of fertilized and waterlogged calcareous soils decreased from 4.7 to 4.0 after 175 days of waterlogging due to the dissolution of the recalcitrant P fraction (Amer et al., 1991). In contrast, Villapando and Graetz (2001) and Ma et al. (2010) reported an increase in the HCl-P fraction of rice soils with increasing waterlogging time of up to 30 days.

Several researchers expressed that NaOH not only extracts Fe-P, Al-P, and organic P compounds but also readily soluble Ca-Ps such as DCP (Hedley et al.,

1982; Gu and Jiang, 1990; Kuo, 1996; Villapando and Graetz, 2001). Therefore, it can be concluded that Ca-Ps and Fe-Ps fractions of the waterlogged soils increase as a function of increasing waterlogging time.

Conclusions

This study underscores the significant impact of waterlogging on phosphorus mobility and availability in calcareous soils, particularly within the context of agricultural practices in semi-arid regions. Our findings indicate that prolonged waterlogging led to notable changes in soil pH, the composition of drainage water, and specific phosphorus fractions. As waterlogging time increased, a decrease in pH was observed along with an increase in soluble reactive phosphate concentrations, highlighting the potential for elevated phosphorus availability under reduced conditions. The observed shifts in the molar ratios of calcium, magnesium, iron, and manganese with respect to phosphorus further illustrate the intricate interactions within these soil systems.

The study reveals that while waterlogging initially promotes phosphorus release, the resulting changes in soil chemistry can have detrimental consequences for surface water quality due to potential eutrophication. The research also points to the varying responses of phosphorus fractions — with NaOH-extractable phosphorus increasing and HCl-extractable phosphorus responding in a soil-dependent manner (increasing in one soil, decreasing in the other two) — demonstrating the inter-fraction mobility of phosphorus under fluctuating redox conditions. Such insights are critical for developing best management practices in irrigated agriculture to mitigate the risks of nutrient runoff and maintain soil health.

These findings contribute significantly to our understanding of phosphorus dynamics in waterlogged calcareous soils and highlight the need for careful management of irrigation practices to optimize plant nutrient availability while safeguarding environmental quality. Future research should explore the long-term implications of these dynamics on both soil health and water ecosystems.

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