



Article Machine Learning Techniques for Improving Nanosensors in Agroenvironmental Applications

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Abstract: Nanotechnology, nanosensors in particular, has increasingly drawn researchers' attention in recent years since it has been shown to be a powerful tool for several fields like mining, robotics, medicine and agriculture amongst others. Challenges ahead, such as food availability, climate change and sustainability, have promoted such attention and pushed forward the use of nanosensors in agroindustry and environmental applications. However, issues with noise and confounding signals make the use of these tools a non-trivial technical challenge. Great advances in artificial intelligence, and more particularly machine learning, have provided new tools that have allowed researchers to improve the quality and functionality of nanosensor systems. This short review presents the latest work in the analysis of data from nanosensors using machine learning for agroenvironmental applications. It consists of an introduction to the topics of nanosensors and machine learning and the application of machine learning techniques to the utilisation of electrochemical, luminescent, SERS and colourimetric nanosensor classes. The final section consists of a short discussion and conclusion concerning the relevance of the material discussed in the review to the future of the agroenvironmental sector.

Keywords: machine learning; nanotechnology; agriculture

1. Introduction

There are a wide number of challenges in agriculture which have to be faced in order to achieve future goals for food availability and sustainability [1]. The demand for increasing production with a coincident need to preserve the environment and increasingly scarce resources means that many current agricultural methods may no longer be sustainable in the long run [2]. Many potential techniques have been proposed to improve the sustainability of agriculture, but one with a very high degree of interest is directed at the utilisation of computational techniques such as artificial intelligence in order to make intelligent agriculture, wherein agriculture is managed in such a way as to maximise the efficiency of production [3]. Of particular importance for the development of this type of agriculture would be a method to rapidly determine the conditions in the field through either more rapid laboratory analysis or sensors placed in the field [4].

A technology which would be very strongly linked to the concept of "intelligent agriculture" would be nanotechnology. Nanotechnology refers to the use of structures and devices with an average size between 1 and 100 nm [5,6]. This type of technology has been utilised for a wide range of applications, ranging from basic physics and materials science to medicine and information technology [7]. Nanotechnology has especially demonstrated a strong potential for making greener, more sustainable agriculture [8,9]. The earliest examples of the application of nanotechnology to agriculture were reported in 2004 and



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Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). have been linked with such areas as crop growth, animal breeding and aquaculture [10]. Reports have been published on a wide range of potential applications in the agroenvironmental sector, such as agrochemical delivery [11], nanofertilisers [12], improved stress tolerance [13], nanopesticides [14], soil protection [15], food packaging [16] and contaminant removal [17], amongst others. In general, the benefits of the use of nanotechnology would include a reduction in the use of scarce resources such as, for example, a 50% saving in the use of water for growing Egyptian clover (Trifolium alexandrinum) under desert conditions with the use of a nanoclay soil amendment [18], a reduction in environmental damage such as a study that demonstrated a reduction in nitrogen leaching of up to 55% using nanocomposite nanourea in the cultivation of mung beans (Vigna radiata L.) [19], and a reduction in the amount of agrochemical required for crop protection such as a study in which nanoemulsions containing the pesticide abamectin demonstrated a 50% lethal concentration (LC_50) nearly five times less than the emulsifiable concentrate of the same chemical against diamondback moth (Plutella xylostella) larvae [20]. Of particular interest for this review would be the utilisation of nanotechnology for the detection and analysis of a wide range of analytes. This technology, also known as nanosensing [21], is a means for the rapid, selective and sensitive detection of a wide range of chemical and biological species. This paper will concentrate on the application of nanosensors linked with other artificial intelligence methods. A further discussion of nanosensors may be found in Section 3. Another technology which would also be of interest in this area would be machine learning. This technology has shown great potential for developing applications in a wide range of fields such as medicine [22–26], business [27,28], marketing [29,30], finance [31-37], logistics [38,39], solar energy [40], biometry [41-45] and many others.

Since the mid-2010s there has been a substantial increase in the number of reports in the literature on the use of machine learning in the agricultural sector [46]. Since such recent beginnings, these techniques have been demonstrated to be a highly effective potential tool [47–55]. Some of the challenges addressed have been fruit classification and segmentation [56–59], crop [60] and biochar [61] yield estimation, timber defect detection [62], water stress measurement [63,64], weed detection [65,66], and pesticide detection [67] and design [68].

Machine learning has been demonstrated to be a tool for aiding in material generation [69–74] and nanoparticle synthesis [75–77]. These techniques have also been used to determine the biological impact of nanomaterials [78–80] which would be important for preventing environmental damage. In addition, these techniques have been turned towards the use of nanomaterials in agriculture, such as the design of a nanoagrochemical delivery system [11,81], predicting the potential impact of nanoagrochemicals on cultivar growth and yield [82] and the use of nanomaterials for seed priming [83].

The use of machine learning for the design and application of nanosensors has been regularly reported in the literature [84–94]. In short, machine learning techniques are utilised alongside large quantities of data and serve to aid in determining patterns that would not be readily apparent without such tools. Conventional nanosensor systems, due to their size, face issues with weak signals and high noise levels, making detection non-trivial. Machine learning techniques can serve to ameliorate these issues.

The application of machine learning with nanosensors has been explored specifically for medical applications [95,96] and food quality assurance [94,96–98].

The use of machine learning to improve the quality and functionalities of nanosensors has increasingly appeared in the literature in recent years. Figure 1 demonstrates the number of publications per year since 2004. This paper is organised as follows: Firstly, Section 2 briefly outlines the methodology used for this literature review. Sections 3 and 4 present a brief (and general) overview of nanosensors and the most used machine learning techniques, respectively. Section 5 is the main body of the review; it provides examples of nanosensors with machine learning, and it can be further divided into examples of different types of nanosensors, namely the Electrochemical Section 5.1, Luminescent Section 5.2, Surface-Enhanced Plasmon Resonance Section 5.3 and Colourimetric Section 5.4. Following

that is a short discussion in Section 6 and a conclusion in Section 7, wherein the importance of nanosensors and machine learning in the agroenvironmental sector is further elucidated.

Published papers per year

Google Scholar Search (terms: Nanosensors, Machine Learning and Agriculture)



Figure 1. This Figure shows the increasing use of machine learning in the development of nanosensors applied to agriculture. Data were obtained from Google Scholar using three keywords: "Nanosensors", "Machine Learning" and "Agriculture". The data were obtained in December 2023.

2. Methodology

This is a bibliographic review that aims to answer the following questions:

- 1. What are the main agroenvironmental applications addressed with nanosensors using machine learning?
- 2. What type of nanosensors are used in these applications?
- 3. How are machine learning techniques used with nanosensors?
- 4. What are the most-used machine learning algorithms and why?
- 5. What are the merits and demerits reported in the literature of using machine learning in nanosensors for agroenvironmental applications?

In order to address these questions, the following actions were performed:

- A search was conducted of the available literature based on the keywords "Nanosensors", "Machine Learning" and "Agriculture".
- The search results were examined carefully for their content and selected for inclusion in this review. In short, the papers were required to feature a (1) nanosensor whose output was processed using (2) machine learning for specific applications in the (3) agroenvironmental sector.
- The articles which passed this examination were then examined using the Connect Papers tool, which identified further possible linked articles to also be examined using the above criteria.
- In total, approximately 50 articles were finally selected for use in this review. They were examined again to gather specific information for use in this study. This information comprised (1) the class/classes of nanosensors used, (2) the type/types of machine learning tools used, (3) the agroenvironmental problem addressed using these techniques and (4) the merits and demerits (if reported) of each nanosensor/machine learning application.

3. Nanosensors

Nanosensors refers to the application of nanomaterials for the detection of analytes through the generation of signals that are then read using appropriate devices [99–101]. Nanomaterials are generally considered to be highly suitable for use as sensors due to the following general properties.

- High surface area: Due to their very small size, nanomaterials possess a very large surface area. This increases the level of interaction between the nanomaterial surface and the external environment.
- Ease of functionalisation: Nanomaterials have a high degree of surface chemistry variability, which permits the formation of receptor species designed for specific targets.
- Quantized properties: Nanomaterials exhibit particular behaviours derived from their size, which differs from that of the same material on the macroscale. Such properties include quantum confinement, surface plasmon resonance and superparamagnetism. These distinctive properties can be exploited for use in sensing applications.

In order to apply nanomaterials to sensing, they must be incorporated into a device from which a signal can be derived. These devices generally consist of the following components [102]:

- Receptor: The surface moieties which are involved in interactions with the target species. These are selected in order to bind specifically in a usually non-covalent and reversible manner. In some cases, more than one receptor may be used in order to generate specific interactions between different components.
- 2. Nanomaterial core: This is the active component of the sensor system. This would engage in the catalysis, luminescence, colour changes or whatever other property central to the activity of nanosensors. The core may consist of free nanoparticles, particles bound to a surface or some other material or nanostructures etched into larger structures.
- 3. Transducer: A system to convert the reception of the target into a signal that can be read. This can consist of the nanomaterial or composite which would generate the signal or also the surface in which the material would be bound and through which the signal would pass.
- 4. Receiver: The instrument or component which would take the signal passed through the transducer and convert it into information which can then be analysed to determine the nature of the signal. The receiver can be an external instrument, such as a spectrometer or human eyes, or additional equipment directly attached to the nanosensor, such as a potentiostat to measure electrical signals.

Several different types of nanosensors have been reported. Some of the more commonly seen types consist of the following:

- Colourimetric: Wherein colour changes due to alterations in the agglomeration or surface states of nanomaterials are demonstrated via alterations in their visible colour. This is most commonly reported for metallic nanoparticles, as the peak plasmon resonance energy or light wavelength of absorption is dependent on apparent particle size, which can be altered via agglomeration [103]. In addition, colour-changing organic dyes can be incorporated into nanocomposites optically responsive to chemical conditions such as pH [104]. The visible nature of the colour change can mean rapid analysis in the laboratory, but there has been much interest in their use as disposable kits for use in the field [105].
- Luminescent: The luminescence of nanomaterials and its quenching, de-quenching or shifts in energy are used to demonstrate interactions on their surfaces. This is most commonly reported for quantum dots [106] and similar materials [107]. The visible nature of luminescence has also resulted in an interest in their use as paper-based sensors for fieldwork [105]. Additionally, luminescent nanosensors can be used for the more efficient and rapid analysis of samples in the laboratory [108].
- Surface-enhanced Raman spectroscopy (SERS): SERS is a method for the enhancement
 of structurally rich but weak signals used in Raman spectroscopy in the presence of
 metal surfaces [109]. Anisotropic metallic nanoparticles were originally detected in

• Electrochemical: The redox reaction of materials on the surface of appropriate nanomaterials generates electrical signals that can be measured. This is regularly reported for graphene, carbon nanotubes, metal oxides and metallic nanomaterials [112]. These types of sensors may function in lab in chip devices for use in laboratory analysis or small portable devices for the creation of a linked network in the field [113].

Additional systems involving the applications of more than one system bound to another involving an inter-nanomaterial interaction which is then involved in the sensing application have been reported [114]. Nanosensors have been reported for a wide number of applications [100], especially in areas such as medicine [115], food safety [116], production [117] and logistics [118], and environmental applications [119]. These technologies have been reported as a potential "electronic nose" for the detection of gaseous chemicals [120]. Generally, nanosensors provide a potentially effective replacement for many forms of traditional analysis for a wide range of physical, chemical and biological targets, reducing costs and wait times and improving specificity [121]. Nanosensors have generally been considered to be an important potential agent for use in the real-time, low-cost determination of actual conditions as part of a Nano Internet of Things (NanoIoT), which involves the connectivity and networking of nanoscale devices into an extended system for the pervasive monitoring of the environment [122–127] and linking these devices to computational systems such as smartphones [128]. The application of nanosensors to agriculture and the related fields of food and environmental monitoring have been reported in a number of review articles. [13,129–133] Nanosensors have been proposed as a method for the rapid and effective analysis of a range of biological, chemical and physical factors in food [134] and agricultural [135] and environmental [136] samples, for the analysis of agrowastes [137] and for the control of irrigation systems [138].

4. Machine Learning

The use of machine learning techniques has considerably increased in recent years, mainly due to the availability of data, faster computer processors and a set of new techniques that allow their application in a wide range of fields. In this section, a brief review of the most used techniques is presented.

Machine learning algorithms are often categorised into several groups: supervised learning, unsupervised learning, reinforcement learning and bio-inspired learning. In this work, we focus mainly on supervised learning and non-supervised learning (see Figure 2). Supervised learning algorithms require labelled data to be trained, which means that, for a set of observed data X, we have their respective output vector y. With this data, a model is trained and then used to predict the output \bar{y} of a new observation x. Supervised learning usually solves two types of problems: classification and regression. Classification algorithms are performed when the problem requires a discrete value to be estimated, which can be a binary problem (two possible outputs) or a multi-class problem (multiple categories). Regression algorithms for classification and regression are presented as follows.

- Classification algorithms:
 - Support vector machine (SVM): This is a binary or multi-class classifier that separates data from different classes by finding the best hyperplane in the feature space that maximises the margin or distance between classes [139]. Figure 3 shows the result obtained when using this algorithm to classify a set of observed data in 2D into two classes. Since such a hyperplane is not always possible to find, there are several tricks that can help the classification, like the use of kernel functions that allow the algorithm to project data to higher-dimension space where the separation between classes could be easier to find [140].

- Random forest (RF): This is a decision-tree-based algorithm that can be used either for classification or regression. It is an ensemble method that uses a multitude of decision trees to estimate the best output [141]. In the case of classification, for instance, the output of the random forest is the class selected by most of the trees. Figure 4 shows an example of a single decision tree obtained when trying to classify the observation shown in Figure 3.
- K-nearest neighbour (K-NN): This algorithm uses proximity as a metric to achieve classification or to predict the grouping of an individual data point. Some of the most-used metrics for proximity (distance) are Euclidean distance, Manhattan distance and Minkowski distance. The k number of classes has to be defined by the user, and it has to be chosen wisely to prevent under-fitting or over-fitting. Fortunately, there are some techniques that help define the best k.
- Artificial neural networks (ANNs): This method was inspired by the way the human brain works; a set of interconnected nodes (neurons) are modelled in a layer structure in which each neuron is interconnected with each other [142–145]. Each node in the neural network has an activation function that defines the output of the node based on the input. The last activation function can be modified to be used for solving classification or regression tasks. There are several types of neural networks, such as the multilayer perceptron (MLP) [146,147], feed-forward neural network (FFNN) [148], recurrent neural network (RNN) [149] and convolutional neural network (CNN), amongst others [150]. Figure 5 shows an example of a multilayer perceptron with two hidden layers.
- Convolutional neural networks: These models are very powerful and have been widely used in image processing and computer vision. There are several convolutional neural network architectures that perform object detection, object segmentation and image classification [151], amongst others. Some examples of architectures for image classification are VGG16, Inception [152,153], Resnet [154] and MobiLenet [155]. Most of these architectures differ in complexity and the number of parameters used. VGG16, for instance, is a very well-known architecture introduced by Simonyan and Zisserman [156]. It is composed of 21 layers (13 convolutional layers, 5 max-pooling layers and 3 dense layers), but only 16 layers have weight (learnable parameter layers). This architecture has around 138 million parameters. There are other versions with similar architecture, such as VGG19 (19 layers instead of 16). An example of a CNN architecture is shown in Figure 6.
- Naive Bayes: This is a family of algorithms with the common principle that every pair of features being classified is independent of each other. This type of classifier assumes that the features used are conditionally independent, given the class label. It uses the Bayes theorem to compute the probability of an observation belonging to each class and pick up the output class with the maximum probability.
- Gradient-boosted regression trees (GBRTs): This is also a family of algorithms that can be used for both classification and regression. It is based on the combination of weak or shallow models (weak learners) like, for instance, shallow decision trees. A sequence of weak learners (i.e., decision trees) is implemented such that each additional tree improves the result of the previous one. There are several implementations of this algorithm. Some examples are LightBGM and XGBoost.
- Regression algorithms: All algorithms discussed above for classification can also be adapted to be used for regression. Other algorithms for regression are as follows:
 - Linear regression: A linear regression algorithm computes the linear relationship between a dependent variable (the output *y*) and one or more independent variables (*X*). The goal is to find the best fit line equation that can predict the values based on the independent variables. Linear regression can also be used for binary classification using a logistic curve to separate the two classes [157].
 - LASSO (least absolute shrinkage and selection operator) regression: This method adds a regularisation term to the linear regression equation. This term is the L_1 distance

and is computed as the sum of the absolute values of the coefficients multiplied by a parameter λ . The parameter λ helps to control the strength of the regularisation.

- Ridge regression: Similar to the previous method, ridge regression introduces a regularisation term to the linear regression based on the L_2 distance. This distance is computed as the sum of the square values of the coefficients multiplied for a parameter λ , where λ , as in LASSO, is the parameter that controls the strength of the regularisation. Adding regularisation terms to linear regression helps prevent overfitting. Ridge regularisation, in particular, also helps correct for multicollinearity in regression analysis (when independent variables are correlated).
- Kernel regression: This is a non-parametric statistical method that helps estimate a smooth curve that describes the relationship between the dependent variable and the independent variables. This method is particularly useful when such a relationship is complex or non-linear.
- Partial least square regression (PLSR): This method reduces the dimensions of the data to a small set of predictors and then performs the regression over them. Hence, it finds a linear regression model by projecting the variables into a new space.
- Polynomial regression: This regression model incorporates higher-order polynomial terms of the independent variables into the model. Such polynomial terms help the regression to capture non-linear relationships between variables.
- Decision tree: This applies a hierarchical structure like a tree, and it is composed of nodes (a root node and several internal nodes), branches and leaves. Each internal node represents a feature of a data set, branches represent the decision rules and each leaf node represents the outcome (see the example in Figure 3b, where a decision tree was implemented for classification). Some of the bestknown tree algorithms include ID3, CART and random forest. Most of them can be used for either regression or classification.
- Gaussian process: The Gaussian process is a non-parametric Bayesian technique that can be used for classification and regression. It is based on the assumption that observations were drawn from a Gaussian process. Instead of learning the values for every parameter in a model (like linear regression models), this approach infers a probability distribution over all possible values. It has the advantage of working well even when only a small data set of observations is available.
- Bayesian linear regression: The goal of Bayesian linear regression is to estimate the distribution over all the parameters and predictions instead of just the "best" value of the parameters like in standard linear regression. Some of the advantages are the chance to include prior information and the fact that the results provide a way to quantify uncertainty in the model parameters and predictions.



Figure 2. A scheme for the most-used algorithms in machine learning. They are classified into three categories: supervised, unsupervised and others. Most of the work done in the papers considered in this review corresponded to the supervised and unsupervised categories.



Figure 3. Support vector machine (SVM). An example of the best hyperplane that separates both classes. This process is not always straightforward, and there are usually observations that can not be well discriminated. The key is to find the best hyperplane that maximises the margin between observations in the boundary of each class.



Figure 4. Decision tree. A decision tree that best classifies the data in Figure 7a. Each node (box) represents a condition made over the feature data. In each branch, the final split represents the classification between the two data classes. The colour indicates which class the majority of the samples at each node belongs to (as darker the colour). The transparency is used to show the impurity of the nodes (in this case is used in the root node).



Input Layer $\in \mathbb{R}^2$ Hidden Layer $\in \mathbb{R}^5$ Hidden Layer $\in \mathbb{R}^5$ Hidden Layer $\in \mathbb{R}^2$ Output Layer $\in \mathbb{R}^1$ **Figure 5.** Multilayer perceptron. An example of a multilayer perceptron (MLP) with two hidden

layers of five neurons each. This model was drawn using the NN-SVG application published by [158].



Figure 6. Convolutional neural network. The main components of the CNN are the convolutional layers (and pooling), which usually perform the feature extraction step, and then the fully connected layer, which acts as classifiers. This image was retrieved from [159] and used under a Creative Commons Attribution (CC BY) license https://creativecommons.org/licenses/by/4.0/#ref-appropriate-credit, accessed on 17 December 2023.



Figure 7. Cont.



Figure 7. Examples of clustering algorithms computed using the set of observed data (a). Mean shift algorithm. (b) Hierarchical clustering. (c) K-mean algorithm computer for k = 2, (d) 3 and (e) 4. (f) Gaussian mixture using 2, (g) 3 (h) and 4 (i) models. (a) Observed data: example of 25 observations of two-dimensional data (two features). This set would be used to show the performance of several clustering algorithms (means shift, hierarchical clustering, K-means and GMM). (b) Mean shift algorithm computed using the observed data in (a). It gives as result k = 3 clusters (orange, green and blue). The mean of the observation belonging to each cluster is shown as a start. (c) Hierarchical clustering: the dendrogram shows the hierarchical relationship between clusters from the observed data. (d) K-mean algorithm computed for k = 2. The observations belonging to each cluster are shown in blue and orange, respectively. The mean of each cluster is shown with a star symbol. (e) K-mean algorithm computed for k = 3. The observations belonging to each cluster are shown in the colours blue, green and orange, respectively. The mean of each cluster is shown with a star symbol. (f) K-mean algorithm computed for k = 4. The observations belonging to each cluster are shown in the colours blue, green, red and orange, respectively. The mean of each cluster is shown with a star symbol. (g) Gaussian mixture: in this case, the data are modelled using two Gaussian functions. The mean and covariance are estimated for each Gaussian. The figure shows which observations belong to each Gaussian (cluster). (h) Gaussian mixture: here, the data are modelled under the assumption that there are three clusters (three Gaussian models). The mean and covariance are estimated for each Gaussian, which represents different clusters. (i) Gaussian mixture: this last example shows the clustering using four Gaussian distributions. As the figure shows, each observation belonging to a different Gaussian is displayed in a different colour.

4.1. Unsupervised Learning

Non-supervised algorithms are commonly used for analysis and cluster data that have not been labelled. Some of the most popular algorithms in this category are used for dimensionality reduction and clustering.

Dimensionality reduction: In most cases, more data improves accuracy results but can
also affect the performance of the machine learning algorithm, causing, for instance,
over-fitting. There are several methods to reduce the dimensions of data by reducing
redundancy. The best-known methods are principal component analysis, linear discriminant analysis (this can also be used as a regressor), singular value decomposition
(SVD) and auto encoders. Principal component analysis (PCA), for instance, provides
a new set of orthogonal vectors for which the principal component is the direction that

maximises the variance of the data set. The next principal component is the direction orthogonal to the prior components with the most variance. This process is repeated until a new feature space to represent the data set is reached. The original data can be computed as a linear combination of this new feature space.

- Clustering: This is the process for group observations that have similar patterns. Some of the most used methods are as follows:
 - K-means: As in K-NN, this algorithm also assumes a k number of clusters and defines the centre of each cluster. Each observation is associated to the closest cluster. The centroid of each cluster is updated by computing the barycenter of all observations belonging to the cluster. This process is repeated iteratively until the centroids of the cluster converge and its position does not change anymore.
 - Hierarchical clustering: This iterative method clusters data using the distance matrix between each observation. At the start, it assumes that each observation is a cluster. Each cluster is then merged with the closest neighbouring cluster, and the process is repeated until all clusters are merged together. As a result, a dendrogram is achieved which shows the hierarchical relationship between the clusters (Figure 7c shows the hierarchical relationship between clusters computed from the data in Figure 7a).
 - Mean shift: Mean shift does not require the user to specify the number of clusters k. It finds regions of high-density observations and iteratively shifts the data points towards such regions until convergence. In order to do so, it estimates the underlying probability density function of the observed data using kernel density estimation and shifts the data by computing the mean shift vector for each data point.
 - Gaussian mixture models (GMMs): In contrast to all the above clustering methods, for which there is no assumption about the distribution of the observation, this method assumes that all observations are generated from a mixture of a finite number of Gaussian distributions with unknown parameters. The goal of this algorithm is to estimate the parameters of each Gaussian distribution. This method can be seen as a generalisation of the K-means method in which not only the centroids but also the covariance matrix are estimated and, therefore, the Gaussian mixture that represents the clustering. An example of this algorithm is presented in Figure 7g–i, where different numbers of Gaussians were used to cluster the observed data in Figure 7a.

4.2. Reinforcement Learning and Others

Reinforcement algorithms allow AI-driven systems (also called agents) to learn using a trial and error strategy. The agent is rewarded when its action is positive or punished otherwise. The goal is to find the model that maximises the cumulative reward for the agent. Several methods have been proposed for model reinforcement learning agents, such as Monte Carlo, the Markov decision process and dynamic programming.

There is also a set of algorithms inspired by the processes of natural selection and evolution [160,161]. Among them are genetic algorithms, artificial neural networks, swarm intelligence and evolutionary computation. These algorithms can be particularly useful in optimisation problems when dealing with large searching spaces or when there are complex interactions between variables.

5. Nanosensor/Machine Learning in Agroenvironmental Applications

As stated previously, nanosensors have a number of potential applications in the agricultural field. However in the case with many different samples and nanosensor systems, the resulting signals may be too weak or noisy for direct analysis. As was shown in the previous section, machine learning techniques allow patterns in data to be recognised for several tasks, such as data analysis, classification (i.e., classification and clustering) and the estimation of a dependent variable, to be performed, given a set of observations. Such tasks are very useful in nanosensor applications since one of the most critical challenges

of a nanosensor is sensitivity to signal readings. In real-world applications in particular, nanosensors are capable of generating signals not only from the analyte they were designed for but also from all the surrounding signals in the environment. The mix of signals generates a noisy output in which specific analyte signals are not easy to identify. The ability of machine learning to recognise patterns allows it to discriminate different analytes from the signal and classify or cluster them accordingly. This allows a more accurate reading for the final sensor outcome to be achieved. Machine learning can also help nanosensors detect analytes indirectly through the measuring of other signals. In this case, regression algorithms can be used to model the response of the analyte by training the algorithms using a set of observed, labelled data. In most cases, the use of machine learning occurs at the end of the sensing process as a method to improve the output given by nanosensors.

The general process is shown in Figure 8. The process begins with agroenvironmental samples which may contain a diversity of materials with a similar chemical structure. They then interact with a nanosensor device, generating a weak or noisy signal. These signals are then processed using machine learning techniques to produce an improved output that can be more readily read.



Figure 8. The general process for the analysis of signals from nanosensors using machine learning.

5.1. Electrochemical Sensors

The use of electrochemical nanosensors can generate signals with a high degree of noise and make it highly difficult to generate clear detection [112]. Machine learning techniques can improve detection by training models to perform tasks such as clustering, classification and regression. Sheng et al. [162], for instance, detected the pesticide Maleic Hydrazide (MA) through the use of an electrochemical sensor made with poly(3,4-ethylenedioxythiophene) (PEDOT) coated with copper nanoparticles, which generated a high degree of noise. A regression ANN was then used to estimate the output of the sensor. In short, an ANN model consisting of an input, an output and two hidden layers was prepared and then trained using signals generated from sixteen samples with a known analyte concentration. Four additional samples were then tested using the trained ANN. Additionally, the performance of the ANN-trained nanosensor was used to test foods spiked with known quantities of Maleic Anhydride. These models demonstrated greater accuracy than high-pressure liquid chromatography (HPLC) used on the same samples.

A gas sensor composed of SnO_2 nanowires was utilised by Tonezzer et al. [163] for the determination of the methanol contamination of alcoholic beverages. Data from responses

of the sensor at various temperatures (5 temperatures: 180, 210, 240, 270 and 300 °C) were used and reduced to two-dimensional data using PCA. Linear discriminant analysis (LDA) and Euclidean UPGMA clustering were used to quantitatively evaluate the sensor's ability to separate the two gases (two classes). Additionally, a support vector machine algorithm was trained to estimate the gas concentration. The resulting sensor was able to accurately distinguish methanol from ethanol and measure their concentration with an average error of 3 PPM in approximately 2–3 min.

An ANN and a multi-ion sensor were used by Chen et al. [164] to detect and quantify nitrate (NO_3^{-3}), phosphate (H_2PO_4) and potassium (K^+) ions in the environment. The sensor's output was used to train a neural network as a regressor to estimate ion concentrations.

Wang et al. [165] utilised a similar system to detect the fungicide carbendazim (CBZ) in rice and tea using carbon nanocomposite electrodes functionalized with ZnS nanoparticles. SVM, a least-square support vector machine (LS-SVM), neural networks and a linear regression model were trained to predict the CBZ residues. The best results used an LS-SVM model. This allows CBZ pesticide residues to be rapidly detected, thanks to the use of a portable electrochemical detection system, and such a system can be very useful for testing food or the environment.

The detection of the toxin mycophenolic acid (MPA) with a nanocomposite mixture of metal organic frameworks (MOFs), a graphene analogue (Ti_3C_2 MXene) and magnetic graphene oxide (Fe₃O₄-MGO) was reported by Ge et al. [166]. Firstly, the oxidation of MPA was modelled using density functional theory (DFT), and an ANN was used to model its electrochemical response at different pH levels. The ANN architecture consisted of an input layer, a hidden layer and an output layer to simulate biological neural operations. The authors reported over 100 per cent recovery from different agricultural samples. The same authors reported another electrochemical sensing system made using violet phosphorene decorated with phosphorus-doped hierarchically porous carbon microspheres with the aid of a random forest algorithm [167]. This was used for the detection of MPA in silage, and the resulting data were analysed using a support vector machine algorithm.

A self-assembled phosphorene/ Ti_3C_2 -MXene nanohybrid was also reported by Zhu et al. [168]. The nanocomposite was generated through the etching of Ti_3AlC_2 with HF and then the incorporation of black phosphorous, followed by deposition onto a laser-induced porous graphene substrate developed from a polyimide film. The resulting nanozyme composite was incorporated into a portable mini-workstation and used to test for the phytoregulator Naphthalene acetic acid (NAA). The electrochemical signals were transmitted through Wi-Fi for analysis. An ANN was trained to estimate the presence of NAA. The performance of the ANN was reported to be better than that of standard linear regression models used previously. The authors reported a limit to the detection of the nanomolar (1.6 nM) using these devices.

Machine forming was used by Li et al. [169] for the creation of laser-induced porous graphene electrodes which were then used for the detection of salicylic acid (SA) with the aid of machine learning for data analysis. An LS-SVM and neural network were used to detect SA using signals from the sensors. The device was linked to a computer controller via Bluetooth, enabling miniaturisation.

Pyrolyzed and carbonised metal organic frameworks (MOFs) were investigated by Lu et al. [170], who used an ANN to analyse data to detect the antihelminthic drug niclosamide. They reported a wide linear sensing range with a low LOD of 0.3 nM. The same material also demonstrated a potential application, as supercapacitors with an ANN were used to predict this property.

Artificial noses are a promising technology for the detection of gases [171]. The incorporation of nanosensors and machine learning seeks to greatly improve their effectiveness [172]. Schroeder et al. [94] proposed chemiresistors generated using carbon nanotubes incorporated into a polyimide layer. Twenty were generated with different selector molecules which functioned to bind different chemical species, and they were incorporated into an array which was tested for its resistivity response in the presence

of volatile organic compounds generated via various foodstuffs. Through the use of a k-nn model and a random forest, the odours generated by cheeses, liquors and edible oils were differentiated.

An artificial tongue made with gold functionalised multi-wall carbon nanotubes (AU-fMWCNTs) coated with a molecularly imprinted polymer was used by Wang et al. [173] for the detection of Fluoroquinolone antibiotics in clinical and biological samples. Ciprofloxacin, levofloxacin and moxifloxacin were used as target templates for the formation of the polymer. The similar structures of the three target molecules made selective analysis non-trivial, so, in order to improve results, the functionalised sensors were incorporated into arrays, and the generated data were analysed by employing discrete cosine transform (DCT), which reduced the dimensions of the input signal. The resulting coefficients were analysed using PCA and ANNs. Limits to the detection of ca. 1 μ M and a wide detection range of up to 300 μ M for the three targets were reported.

Similar arrays made using GaN nanowires functionalised with TiO₂ and ZnO with additional metal functionalisation were reported by Khan et al. [174]. They reported an electrical signal under UV irradiation in the presence of NO₂, ethanol, SO₂ or H₂ in the presence of H₂O and O₂ gases at room temperature or in a mixture. These data were analysed using PCA to reduce dimensions and cluster the data. Then, four other algorithms were used (decision tree, SVM, naive Bayes (NB) (kernel) and k-NN) for gas classification.

An alpha Iron Oxide-reduced graphene oxide (α -Fe₃O₄-rGO) inkjet-printer-compatible composite material synthesised using hydrothermal synthesis was reported by Wu et al. [175]. These materials were used as an electrochemical sensor for the detection of N₂O across a range of environmental conditions. Through the use of PCA and clustering (KNN), the confounding effects of different temperatures and humidity levels could be counteracted. This technique achieved an overall discrimination accuracy of 97.3%.

The information of two different analysis techniques was analysed by Zhu et al. [176] for the detection of carbendazim using the graphene analogue Ti₂C MXene with detection via electrochemical sensors and with SERS. Regression models (ANN, SVM and RSVM) were used to estimate the detected signals. All models were trained using data from both electrochemical and SERS sensors, and 30 samples (about 80% for training and 20% for testing) were used. As a result, RVM displayed superiority in the electrochemical analysis of CBZ in a wide linear range of 0.006–9.8 μ M with a low limit of detection (LOD) of 0.002 μ M and SERS detection of CBZ in the wide linear range of 0.033 $\times 10^{-10}$ μ M with a low LOD of 0.01 μ M.

The direct monitoring of the internal conditions of a plant using a wearable sensor was reported by Lee et al. [177], who investigated the presence of VOCs, temperature and humidity through the use of a wearable sensor attached to the underside of tomato plants containing Au/Ag nanowires, multiwalled carbon nanotubes (MWCNTS) and sol-gels used to generate electrochemical signals to be analysed via PCA and Euclidean distance to detect changes in plant physiology due to infection with the tomato spotted wilt virus.

5.2. Luminescent Sensors

Luminescent nanosensors are viewed as a material with a high degree of potential for use in the field, as they generate a light emission signal that can be detected with a range of devices, from spectrophotometers to smartphones.

Ultraportable smartphone platforms were utilised by Ge et al. [178] for the detection of MA. This work involved screen-printed electrodes with Halloysite and black phosphorous that were able to detect MA through the use of the BP-ANN-GA algorithm to model the optimal relationship between the composite ratio and the analyte detection current. LS-SVM and ANN were also used to model the linear relationship between analyte concentrations and the current value, facilitating detection. An LOD of 0.3 μ M was reported.

The use of an algorithmically guided optical nanosensor selector (AGONS) was reported by Smith et al. [179,180]; data from optical nanosensor arrays were analysed in order to improve data for biomarker detection [179] and the detection of citrus juices' adulteration [180].

The signals from multiple materials were also reported by Sarmanova et al. [88], who looked at fluorescence signals generated via carbon dots in the presence of Cu^{2+} , Ni^{2+} , Cr^{3+} and NO_3^- ions in an aqueous solution. The use of one and dimensional CNN machine learning tools enabled the simultaneous determination of the metal ions Cu^{2+} , Ni^{2+} and Cr^{3+} with a root mean squared error of 0.28 mM, 0.79 mM and 0.24 mM, respectively. The best model was the 2D CNN since it better captured the dependency of the data in two dimensions.

Carbon dots were also used to detect the mycotoxins citrinin, alfatoxin B1 and ochratoxin A by Aggarwal et al. [181]. In short, the generated dots were dispersed in different solvents or water at different pHs as a barcode. The photoluminescence spectra of these dots were fed into a number of machine learning tools and used to differentiate between the mycotoxins (K-NN, SVM, XGBoost, AdaBoost, Gaussian naive Bayes, Gaussian process classifier, CNN and ANN). They reported a limit of detection in the ten picomolar range and noted that, of all the machine learning models that were used, XGBoost was found to have delivered the highest specificity and selectivity in the presence of wheat, rice, coffee, maize, milk and gram extracts.

Similar work was reported by Xu et al. [182], who generated carbon dots and CdTe quantum dots for the detection of antibiotics. They built a unified SX model using a "stepwise prediction" strategy combined with classification algorithms to screen optimal methods. After integrating classification and concentration models under a tree-based pipeline optimisation technique framework, the extreme random forest was selected as the most accurate classification model (multiclass model). The sensor array detected nine (nine classes of)antibiotics at 0.5–50 μ M with 95% accuracy and a 4.93% average concentration error for unknown samples outside the datasets.

The same researchers also examined the use of machine learning for the analysis of quenching data generated from fluorescent ss-DNA bound to 2D nanoparticles such as graphene, MoS₂ and WS₂ to differentiate between the strains of lysed bacterial cells [183]. A dimensionality reduction technique was first used to cluster the data (PLSDA), achieving classification results of 88%. Later, other tools like LR, SVM, kNN, random forest (RF), decision tree, ridge, gradient boosting and MLP were used. The best classification result was obtained with MLP (90% accuracy).

Noreldeen et al. [184] reported the development of an interesting 3D fluorescence spectra method for the detection of bioflavonoids in a biological sample using gold nanoclusters. The data generated from eight similar molecules were classified using BLSTM, achieving 98% accuracy with an LOD in the nanomolar range. The BLSTM classifier was trained to quantify bioflavonoids; then, random forest was used to select the most important feature, achieving better results.

The same authors also reported a similar method for the detection of vitamin B6 using cysteamine/N-acetyl-l-cysteine/AuNCs and convolutional neural networks [185] and the detection of metal ions using random forest and SVM for classification and LDA and PCA to reduce dimensionality [186].

Flavanol detection was reported by Li et al. [187], who analysed the fluorescence spectra of copper, magnesium and aluminium cation-doped cyclodextrin particles. Flavanols extracted from *Kaempferia galangal* and honey binding onto these nanocomposites were investigated through simulation, and the resulting shifts in fluorescence were analysed. An SVM technique was used to classify the sensors' output, and it correctly predicted the proportion of flavonols in the *Kaempferia galangal* and honey samples. In order to quantify the flavonol concentration, several regression techniques were implemented, and a back-propagation neural network (BPNN) achieved the best prediction performance.

Plant disease detection was reported by Rhamani et al. [188], who used carbon nanotubes functionalised with DNA aptamers and hemin whose NIR light emission was quenched in the presence of hydrogen peroxide, indicating plant stress. Tea plant (*Camellia Sinensis*) leaves with and without hydrogen peroxide were imaged using NIR and RGB cameras and analysed using CNN-based feature extraction and feature classification models. A classification efficiency of 98.75% for the Wasserstein distance-to-convolutional neural network (WD2CNN), also the most efficient model, was reported.

Ratiometric analysis is a particular form of analysis in which two or more collected signals are collated in order to generate a reading. Ratiometric sensors systems demonstrate greater resilience with regard to variations in signal intensity and can improve the sensitivity of nanosensor analysis in both the laboratory and the field. Examples of luminescent nanosensors with ratiometric analysis have been reported [189].

Lu et al. [190] proposed a machine learning-assisted multivariate detection smartphoneintegrated optical platform to detect the signals generated via green fluorescent carbon dots and a blue-emitting Fe/Zn metal organic framework in the presence of biogenic amines (BAs), including spermine (SP) and histamine (HIS). The detection and classification of the images obtained was performed using a YOLO V3 architecture and image processing techniques.

Dual-emission molecularly imprinted polymers (dual-em-imp) were reported by Liu et al. [191]. In short, a polymer matrix containing both red-emitting SiO₂-coated CdS quantum dots (QDs) and blue-emitting graphene QDs was made in the presence of the herbicide pretilachlor. Due to the green- but not red-emitting QDs being quenched by the herbicide, changes in the red–green–blue (RGB) colour balance were detected and analysed using a random forest method trained to estimate each of the concentration parameters of pretilachlor and assess the importance of fluorescence colour parameters. Thirty per cent of the data was used for testing in which the negative mean square error was used as an error metric for the estimation. The developed sensor apparatus was tested on environmental water samples and fish.

A similar electrochemiluminescent sensor was developed for the detection of tyramine by Lu et al. [192]. They deposited cerium tin oxide (Ce₂Sn₂O₇) nanoparticles on a glassy carbon electrode and electropolymerised pyrolle onto this in the presence of tyramine as a template. The electroluminescence of this material in the presence of the target as a cathode and Ru(bpy)²⁺₃ as an anode was used in a dual-emission system. The authors presented many simulations to outline the interaction nature and identify those involved in signal generation. Luminescence images were taken using a smartphone and analysed with the ANN and YoloV5 deep learning techniques. A tyramine detection range of 0.01 μ M to 1000 μ M with a detection limit as low as 0.005 μ M was reported.

5.3. Surface-Enhanced Plasmon Resonance-Involved Sensing

Surface-enhanced Raman spectroscopy (SERS) is an important technique for enhancing the structurally useful but weak chemical characterisation technique. SERS was used on polystyrene spheres coated with silver nanoparticles by Lin et al. [193]. The SERS signal of the resulting assemblage was analysed using PCA and SVM in order to differentiate multiple model contaminants with a similar chemical geometry. PCA was first performed with the data, and then a support vector machine algorithm was trained to achieve the classification of the data. The data used consisted of 50 SERS spectra each of RB aqueous (10⁻¹⁰ M) and R6G aqueous (10⁻¹⁴ M) solutions, of which 30% was the testing set (and the other 70% was used for training). The results showed better performance when the data were first reduced in dimensionality using PCA than when using the raw data directly.

SERS detection of the mycotoxin zearalenone (ZEN) in corn oil was accomplished by Zhu et al. [194] through the use of gold nanorods with the data analysed through regression techniques, partial least squares regression (PLSR), random forest regression (RFR), Gaussian progress regression (GPR), 1D CNN and 2D CNN. It was determined that the latter two systems achieved the best prediction performance with limits of detection of 6.81×10^{-4} and 7.24×10^{-4} µg/mL, respectively.

A localised surface Plasmon resonance (LSPR)-enhanced sensor array for the detection of plant volatile organic compounds (VOCs) emitted via plants suffering from pests and diseases was also reported by Shang et al. [195]. In short, a mixture of gold nanoparticles was embedded in a sol-gel imprinted with various target molecules based on previous work [196]. The interaction of the target and related molecules was measured through changes in adsorption and the effect on nanoparticle size and concentration on signal intensity. The signals generated were classified using several non-supervised machine learning methods, such as linear discriminant analysis, k-NN and naive Bayes. The presence of each gas was determined with a high degree of accuracy, and the best results were achieved using the k-NN

method (between 89–96%). The same authors reported silver/gold nano-urchin nanoparticles deposited on indium tin oxide glass and then coated with a sol-gel molecularly imprinted with hexanoic acid, heptanoic acid and octanoic acid [197]. Variations in optical properties due to the presence of these acids were then analysed using the PCA, LDA, KNN and NB methods, demonstrating an effective measure for identifying the nature of the gas mix. The NB and LDA methods showed a satisfactory performance for vapour classification.

The detection of pathogenic bacteria using machine-learning enhanced Raman spectroscopy was reported by Alia Colniță and her coworkers [198]. They coated *Lactobacillus casei* and *Listeria monocytogenes* bacterial strains with silver nanoparticles, either synthesised before or grown on the bacteria surface. It was noted that similar Raman bands were observed for both types of silver nanoparticles. PCA was used to differentiate the two bacterial strains by explaining up to 30% of the variance observed.

5.4. Colourimetric Sensors

The union of colourimetric sensors with machine learning enables subtler colour changes than those traditionally determined by eye. The potential for this to improve food safety and quality was recently enumerated [199,200].

The colourimetric detection of antibiotics in raw milk was performed by Gutiérrez et al. [201], who utilised a gold nanoparticle/aptamer-based nanobiosensor incorporated into an optomechanical device to measure their aggregation automatically. Spectral data were acquired and then processed using a linear combination of known continuousspectrum functions (legendre functions) to determine the presence and concentration of a range of antibiotics (kanamycin, ampicillin, oxytetracycline and sulfadimethoxine). The coefficients of the legendre function projection were used as a feature vector to train an SVM classifier (with three classes: without antibiotic, with a concentration below MRL and with a concentration above MRL). As a result, detection of 0.25 times the MRL was reported for these drugs.

Another method for the detection of antibiotics in milk was reported by Zhou et al. [202], who developed an immuno-assay based on polystyrene nanospheres and magnetic nanoparticles for the detection of chloramphenicol, kanamycin and neomycin. Differences in particle size distribution were detected through UV-VIS spectroscopy using a PLSR model. The authors reported more than 99% accuracy down to the pg/mg range.

Gou et al. [203] generated a method for determining meat freshness consisting of halochromic dye-filled chitosan nanoparticles embedded in cellulose acetate. Twenty different dye-np concentrations were prepared to form a barcode whose colour changed in the presence of decomposition gases. CNN was then used to process barcode images to classify meat freshness. Three classes were used, fresh, less and spoiled, using chicken, fish and meat. A total of 3475 images were used to train a Resnet101 architecture to classify the three classes. The overall accuracy obtained using 686 testing images was 98.5%.

Zadorozhnaya et al. [204] developed a system to replace the microtox analyzer with multisensory systems. Three models were studied, PLSR, random K-NN and random forest, and used to estimate the sample's toxicity index. In the case of random forest and K-NN, the models were first used for data classification, and then a regression was performed with data from each class separately. While the best results obtained 20 and 25%, this method showed excellent potential for further research in this area.

Three representative amino acids, namely methionine (Met), tryptophan (Trp) and histidine (His), were utilised for the modification of gold nanotubes as reported by Jia et al. [205] for the detection of distinctive organic acids found in the aromatic liquor Chinese baijiu. After training with eleven different organic compounds determined to be important for liquor flavour, the nanoparticles were used in a colourimetric sensor at two different pH values; through the use of an LDA model, fourteen different brands of baijiu could be discriminated.

MOFs were also reported by Ma et al. [206] for the detection of the amine-related gases generated in meat decay. They developed a composite material of the MOF, UiO-66, loaded with colour-changing dyes and incorporated into a PVA resin. It demonstrated distinct colour changes depending on the presence of ammonia, methyl amine and trimethylamine. Inception V3, VGG-16, Resnet-152 and WISeR-50 models were trained to recognise the colour change for high-accuracy freshness estimation. All the models were trained and tested with images taken from the sensor. The best classification results (spoiled versus fresh) were obtained when using the WISeR-50 architecture (98.95% accuracy).

The same authors also reported similar colourimetric array systems for the detection of beef freshness using oxidised chitin nanocrystals. The resulting images were classified as fresh or spoiled using the same four models. The best results were obtained when using Resnet-50 (99.27% accuracy) [207]. They also explored the use of these models to classify freshness in shrimp using ice-templated dye@ MOF(UiO-66-Br)/Chitosan [208]. In this case, the best accuracy obtained was 99.94% using the WISeR-50 architecture.

Other biogenic amines derived from meat degradation were the target of a research report by Zhong et al. [209]. Gold nanoparticles and graphene oxide were loaded with PH-sensitive organic dyes and incorporated into a spot array. Distinctive colour changes in these arrays were noted in the presence of selected amines, and reading these was accomplished using a plate scanner and clustering algorithms such as HCA, PCA and linear discriminant. A very low LOD and over 90% differentiation accuracy between eight different biogenic amines were reported.

MOFs, silica nanospheres and organic dyes were combined into nano-chemicalresponsive materials (nano-CRMS) by Kang et al. [210] to better elucidate between volatile alcohols normally found in fermented kombucha tea. The nanosensor agents were incorporated into arrays, and colour changes were detected and analysed using PCA. They reported a 90% variance in determining the fermentation degree in kombucha.

Li et al. [211] reported the use of a fingerprinting system involving cysteine-functionalised gold nanospheres and nanorods deposited on nitrocellulose paper for use as a wearable colourimetric nanosensor for plants. The interactions of VOCs with nanoparticles of distinct sizes demonstrated different agglomeration levels, which generated distinct colours that could then be measured with a smartphone. This process was utilised in order to determine infection with *Phytophthora infestans* blight in selected leaves. The resulting signals were analysed using PCA, for which clusters of leaves with and without infection could be visualised.

Ratiometric analysis with machine learning has also been reported for use with colourimetric systems. For example, RGB smartphone sensors enabled the formation of time-temperature integrators (TTIs) using polymer colloid crystals which were synthesised by Schöttle et al. [212], who then utilised an ANN to predict not only the sensor temperature but also the duration at that temperature, useful information for storage safety.

A double sensor based on a microfluidic device containing both gold nanoparticles and dyes embedded in a porous hydrophobic membrane made of polyvinylidene diflouride (PVDF) was reported by Zhao et al. [213]. Through the use of feature extraction and clustering techniques, HCA and PCA, the different signals generated by both the colourimetric and fluorescent signals were analysed in order to detect and quantify various carbamate pesticides.

Finally, a multivariant sensing system generated using multiple nanomaterials was reported by Qileng et al. [214]. In short, an immunosensor was made through the addition of magnetic beads, mesoporous silica, and silver and gold bi-pyramids, as well as cadmium sulfide-coated graphene oxide electrodes. The interactions between these components can be mediated with antibodies, generating signals in photoelectrochemistry, fluorescence and

colourimetry. The concentration of mycotoxins and the presence of analogues were determined using a neural network of three layers (an input layer, a hidden layer and an output layer).

6. Discussion

Addressing the first question associated with this review, "What are the main agroenvironmental applications addressed with nanosensors using machine learning?", it was demonstrated that the main problems addressed via the application of nanosensors in conjunction with machine learning are food safety and food classification, the detection of agrochemicals, plant protection and environmental sensing. In general, these applications involve the detection of marker chemicals associated with food decay [190], food adulteration [180] and plant infection [211], as well as the presence of contaminants in food [178] and environmental samples [104]. Table 1 is a summary of the reviewed work classified according to the problem addressed and the type of nanosensor used. Most of the work done has been for use in food applications, such as the detection of gases associated with decay in which luminescent and colourimetric nanosensors were amongst the most used. Addressing the second question associated with this review, "What type of nanosensors are used in these applications?", the results tables report four classes of nanosensors used for these problems; electrochemical, luminescent, SERS and colourimetric. Examining the reported literature reveals that the most commonly used nanomaterials for each class are as follows:

- (a) Electrochemical nanosensors favoured the use of functionalized carbon-based nanocomposite materials.
- (b) Luminescent nanosensors favoured the use of luminescent carbon dots.
- (c) SERS nanosensors favoured the use of gold and silver nanoparticles and composites containing these nanomaterials.
- (d) Colourimetric nanosensors favoured the use of gold nanomaterials (nanoparticles, nanorods and nanopyramids).

Problem	Electrochemical	Luminescent	SERS	Colourimetric
Food safety	Tonezzer et al. [163] Wang et al. [165] Ge et al. [166] Zhu et al. [168] Lu et al. [170] Wang et al. [173]	Smith et al. [179] Smith et al. [180] Aggarwal et al. [181] Xu et al. [182] Nandu et al. [183] Lu et al. [190] Lu et al. [192]	Zhu et al. [194]	Gutiérrez et al. [201] Zhou et al. [202] Gou et al. [203] Zadorozhnaya et al. [204] Ma et al. [206] Ma et al. [208] Zhong et al. [209] Qileng et al. [214]
Agrochemicals	Sheng et al. [162] Li et al. [169]	Ge et al. [178] Liu et al. [191]		Zhao et al. [213]
Enviromental sensing	Chen et al. [164] Khan et al. [174] Wu et al. [175]	Sarmanova et al. [88] Noreldeen et al. [186]	Lin et al. [193] Chen et al. [197] Colniță et al. [198]	Schottle et al. [212] Qileng et al. [214]
Food classification	Schroeder et al. [94]	Noreldeen et al. [184] Noreldeen et al. [185] Li et al. [187] Lu et al. [190] Lu et al. [192]		Gou et al. [203] Jia et al. [205] Ma et al. [206] Jia et al. [207] Ma et al. [208] Kang et al. [210] Zhong et al. [209]
Plant protection	Lee et al. [177]	Rhamani et al. [188]	Shang et al. [195]	Wei et al. [211]

Table 1. Summary of the most important work on nanosensors using machine learning classified according to the type of sensor and the problem addressed.

Let us address the third question, "How are machine learning techniques used with nanosensors?"

We have seen that nanosensors have a high degree of potential for use in agriculture, but a low signal strength and a high degree of noise make their application using traditional forms of analysis very challenging. Machine learning, on the other hand, is a set of tools that allows the analysis of data and making sense of them. As was presented in Section 4, there are several tasks that can be performed using machine learning techniques, such as regression, classification, clustering and dimensionality reduction, amongst others. They help find patterns in data and make the analysis of very complex signal sets possible. There were two specific examples noted in this review:

- Selectively designed nanosensors analysing samples with analogous materials that may generate confounding signals [88]. This may make these more traditionally designed nanosensors function as originally envisaged.
- 2. The use of barcodes or multiple sensing systems selected to partially interact with more than one analyte, generating many detections [181,203]. The technology described involves the formation of an array of nanosensors containing receptors which only bind the target analytes partially. Each nanosensor in the array would only demonstrate what would be considered partial detection. However, as an array, each component may generate partial detection in a reproducible manner. Such signals would be necessarily complex and need special tools to detect a single analyte in a single sample. This form of analysis would enable the formation of effective nanosensors without the need for the development of highly specific binding agents for every potential analyte. This may aid in producing more efficient and generally applicable sensing systems.

To address such problems, machine learning has been proposed in the literature; the techniques used varied, depending on the type of nanosensor and the target problem. Electrochemical sensors, for instance, mostly utilised regression models. This can be linked to the fact that most electrochemical sensors aim at estimating continuous voltage and current values. As such, regression models are better suited for that task. In these cases, machine learning helps estimate the output signal even when data are weak and noisy. Classification techniques, on the other hand, are mostly used for luminescent and colourimetric nanosensors for which, in most cases, the output signal needs to be categorised in a particular class. For instance, colourimetric sensors are used to classify the freshness of food (or quality) by categorising the output colour of the nanosensor. In cases where labelled databases are not available and supervised techniques such as classification and regression cannot be used, other techniques such as clustering help classify the signals and allow nanosensors to identify or detect single analytes.

The use of machine learning techniques is not necessarily strict in the sense that only one method has to be used for a given application. In most cases, several methods were trained and tested to find the most suitable for the problem to be solved. Even different types of machine learning methods (supervised and unsupervised) can be used in conjunction to improve results or to address different stages of the problem. Table 2 shows that, in many cases, regression or classification methods were used together with dimensionality reduction methods. This is reasonable since dimensionality reduction techniques such as PCA, for instance, allow data to be transformed into orthogonal vectors in which the principal component is the direction that maximises the variance of the data set. Such transformations help select features with the greatest amount of information in the transformed space and are particularly helpful when the number of observations is reduced in comparison with the number of independent variables (features). The most used type of machine learning is classification (see Table 3).

Type of Sensor	Reference	Regression	Classification	Clustering	Dim. Reduc.
	Tonezzer et al. [163]	\checkmark	-	-	\checkmark
	Wang et al. [165]	\checkmark	-	-	-
	Ge et al. [166]	\checkmark	-	-	-
	Zhu et al. [168]	\checkmark	-	-	-
	Lu et al. [170]	\checkmark	-	-	-
	Wang et al. [173]	-	\checkmark	-	\checkmark
	Sheng et al. [162]	\checkmark	-	-	-
	Li et al. [169]	\checkmark	-	-	-
	Chen et al. [164]	\checkmark	-	-	-
	Khan et al. [174]	-	\checkmark	-	\checkmark
	Wu et al. [175]	-	-	\checkmark	\checkmark
	Schroeder et al. [94]	-	\checkmark	-	-
	Lee et al. [177]	-	-	-	\checkmark
Electro ab operical	Smith et al. [179]	-	\checkmark	-	\checkmark
Electrochemical	Smith et al. [180]	-	-	\checkmark	\checkmark
	Aggarwal et al. [181]	-	\checkmark	-	-
	Xu et al. [182]	-	\checkmark	-	-
	Nandu et al. [183]	-	\checkmark	-	\checkmark
	Lu et al. [190]	-	\checkmark	-	-
	Lu et al. [192]	-	\checkmark	-	-
	Ge et al. [178]	\checkmark	-	-	-
	Liu et al. [191]	\checkmark	-	-	-
	Sarmanova et al. [88]	-	\checkmark	-	-
	Noreldeen et al. [186]	-	\checkmark	-	\checkmark
	Noreldeen et al. [184]	-	\checkmark	-	-
	Noreldeen et al. [185]	-	\checkmark	-	-
	Li et al. [187]	\checkmark	\checkmark	-	-
	Rhamani et al. [188]	-	\checkmark	-	-
	Zhu et al. [194]	\checkmark	-	-	-
	Lin et al. [193]	-	\checkmark	-	\checkmark
SERS	Chen et al. [197]	-√	-	\checkmark	
	Colniță et al. [198]	-	-	-	\checkmark
	Shang et al. [195]	-	\checkmark	-	-
	Gutiérrez et al. [201]	-	\checkmark	-	-
	Zhou et al. [202]	\checkmark	-	-	-
	Gou et al. [203]	-	\checkmark	-	-
	Zadorozhnaya et al. [204]	-	\checkmark	-	-
	Ma et al. [206]	-	\checkmark	-	-
	Ma et al. [208]	-	\checkmark	-	-
Colourinostria	Zhong et al. [209]	-	-	\checkmark	\checkmark
Colourimetric	Qileng et al. [214]	\checkmark	-	-	-
	Zhao et al. [213]	-	-	\checkmark	\checkmark
	Schottle et al. [212]	\checkmark	-	-	-
	Jia et al. [205]	-	-	-	\checkmark
	Jia et al. [207]	-	\checkmark	-	-
	Kang et al. [210]				
	Wei et al. [211]	-	-	\checkmark	\checkmark

Table 2. Type of machine learning algorithms used in the reviewed work classified by type of sensor and machine learning task: supervised learning (regression and classification) and unsupervised learning (clustering and dimensionality reduction).

Table 3. The number of each algorithm class used to solve the problems in the agroenvironmental field. The reported number does not represent the number of papers since there were papers that could be categorised into more than one application, as can be seen in Table 1. As can be observed, classification and regression are the most-used techniques.

Problem	Regression	Classification	Clustering	Dim. Reduc.
Food safety	8	12	2	5
Agrochemicals	4	-	1	1
Environmental	3	5	1	4
Food classification	1	10	-	4
Plant protection	0	2	1	2

Let us address the fourth question, "What are the most used machine learning algorithms and why?" When the specific methods used are examined separately, the PCA appears to be the most-used method amongst the different types of sensors (see Table 4). This could be explained in that PCA is also an excellent tool to visualise the strongest trends in a dataset or between groups in a dataset. Such graphical displays offer a visual approximation of the information contained in the data. Therefore, they can directly give a preliminary view of the information of the observed data.

Another widely used method is artificial neural networks (ANNs). This technique can be used for both classification and regression. In this review, most examples of their use were estimating signals from electrochemical sensors in which signals, such as, for instance, the electrochemical response to PH [166], the concentration of the pesticide Maleic Hydrazide [162], ion concentrations [164] and Naphthalene acetic acid concentration [168] were estimated using ANNs as a regression method. Convolutional neural networks (CNNs) (a type of ANN applied to image processing) are also amongst the most-used methods). This is particularly important in the development of colourimetric sensors, for instance, when the output of nanosensors are colour changes that can be analysed as images. In such problems, CNNs can be used as classifiers and are able to detect colour changes more effectively than traditional image processing techniques. Applications to food safety and food classification have been observed for this type of machine learning technique.

Table 4. Ranking of the most-used machine learning algorithms from the top one (first row) to the top five (fifth row). The table shows which problems and sensors were used; for problems, we have FA—food safety, A—agrochemical, E—environmental, FC—food classification and PD—plant protection. For sensors, we have E—electrochemical, L—luminescent, S—SERS and C—colourimetric.

Deterri		Problems					Sensors			Mashina I associate (Tash)
Dataset	FS	Α	Ε	FC	PP	P E L S C Machine Learning (Task)				
PCA	\checkmark	\checkmark	\checkmark	\checkmark	\checkmark	\checkmark	\checkmark	\checkmark	\checkmark	Dimentionality reduction
ANN	\checkmark	\checkmark	\checkmark	\checkmark	-	\checkmark	\checkmark	-	\checkmark	Regression/classification
SVM	\checkmark	-	\checkmark	\checkmark	-	\checkmark	\checkmark	\checkmark	\checkmark	Regression/classification
CNN	\checkmark	-	\checkmark	\checkmark	\checkmark	-	\checkmark	\checkmark	\checkmark	Regression/classification
K-NN	\checkmark	-	\checkmark	\checkmark	\checkmark	\checkmark	\checkmark	\checkmark	\checkmark	classification

Let us address the fifth question, "What are the merits and demerits reported in the literature of using machine learning in nanosensors for agroenvironmental applications?" From the above sections, it is clear that the incorporation of machine learning into the application of nanosensors provides a noticeable increase in the effectiveness and selectivity of this highly relevant technology. The relevance for agriculture that the reports included in this review are the following general areas.

- 1. More effective and efficient sensors for the detection of selected analytes in the laboratory. These nanosensor/machine learning pairings may mean a more accessible form of analysis, enabling quicker and cheaper laboratory analysis of agricultural and food samples.
- 2. The possibility of the development of handheld or portable nanosensor systems for field use. This is a rapidly growing area of the agribusiness sector with considerable interest for use in the plant growth, livestock and fish farming sectors [215]. Some examples of this include the use of electrochemical sensor patches [177] and paper sensors which can be used in conjunction with a smartphone [206], meaning the possibility for automatic and continuous monitoring in the field. This provides a benefit above laboratory analysis in that the agronomist would be provided with up-to-date conditions on the ground without the need to remove samples to an exterior facility, also enabling a rapid response to potentially deleterious conditions [216,217]. In addition, the continuous placement of a portable nanosensor system makes possible a more complete understanding of changes in the physio-chemical properties of their location over time than random samples with possible unintended variance.

- 3. Improved analysis using nanosensors compared to nanosensors on their own. This is especially the case for the use of ML techniques to decollate the signals generated via chemically similar species.
- 4. Reducing the effect of cross-responses. This allows several analytes to be sensed simultaneously.
- 5. Nanosensor/machine learning techniques reported improvements compared to traditional forms of sample analysis such as improved accuracy, faster response, and reduced costs in many cases.

In both cases, these devices (nanosensors with machine learning) have the potential to greatly increase the quantity of information available to agriculturalists and enable the better monitoring of conditions and, with it, an important stage in the development of a technological "intelligent" agriculture capable of serving societies' needs in the future.

As has been reviewed, machine learning techniques have already helped in improving nanosensor utilisation in several applications that may change the condition of the whole food supply chain in the near future. However, there are still many opportunities to expand their use in other agroenviromental applications. Some of the demerits of using such technology are as follows:

- 1. The training of ML algorithms requires the availability of good-quality data to train the models (specially labelled data to train supervised models). Such data may be difficult or expensive to acquire using nanosensors.
- 2. Problems such as overfitting may appear due to noisy datasets, which are usually the case for signals captured via nanosensors. Expertise in data analysis, the selection of the techniques to be used and the pre-processing of the data may help reduce this problem. In general, any issues with the data can greatly reduce system performance.
- 3. The deployment of machine learning techniques in small electronic devices can also be a challenge. This is especially difficult when large models are used (a high number of parameters), like in the case of CNN models applied to colourimetric or luminescent nanosensors, for instance.
- 4. Nanosensor formation can be synthetically challenging and involve toxic materials such as cadmium-series quantum dots. In some cases, specific receptor moieties would need to be redesigned for each analyte.
- 5. The use of nanomaterials in the field can lead to environmental concerns due to their possible release.
- Regulatory and social considerations may prevent their adoption into the market.

7. Conclusions

This paper has shown the latest work on nanosensors applied to agroenviromental problems using machine learning. The literature shows that these technologies are increasingly being used in this field and have already contributed to improved nanosensors and the analysis of data from existing nanosensor systems. Nanotechnology, especially nanosensors, has immense potential to improve agroindustrial management and environmental monitoring. Some of the most pressing issues for the widespread application of this technology would be issues with noise and confounding signals. The application of machine learning techniques provides a highly effective tool for confronting these issues and improving the potential viability of this technology. There are, however, other issues in the widespread application of this technology. Some of these are technical in nature, such as assuring accurate and reproducible nanomaterials with the exact properties needed for use in nanosensors and the development of scalable assembly methods for nanosensor devices [117]. Interestingly, machine learning techniques can aid in the development of effective solutions for both of these problems [75,85]. With the use of these nanosensors in food production, another issue would be nanotoxicology, as the widespread use of persistent nanomaterials can lead to increased contamination. The selection and design of nanomaterials and their use can help reduce the risk in this respect [218]. Other challenges would be economic. Nanomaterials' synthesis and purification can be costly, as is the formation of arrays for use as nanosensors [100]. Economies of scale for nanosensor production, as well as improved efficiencies compared to more traditional forms of monitoring and downstream savings due to the information gained, may help reduce these costs for large commercial users. However, they may still be prohibitive for smallholders, preventing their universal application. Finally, regulatory and social issues with both strong regulations on the use of nanomaterials in consumer products and fears among potential users can both serve to prevent the adoption of this technology [117]. Complete transparency in the investigation of the safety of nanosensors, coupled with an education programme on the benefits and promise of nanosensors, can help confront these concerns. Possible developments in this technology would be linked to increased computational power for analysis and improved networking between nanosensor devices [219]. Another possibility involves the generation of multiple sensing systems into single devices aided by improved manufacturing techniques and computational tools that can aid the analysis of multiple data streams [95]. Regardless of the future direction, it is clear that the union of machine learning and nanosensor technology will be a keystone technology for the analysis of agroenvironmental conditions in the future.

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Abbreviations

The following abbreviations are used in this manuscript:

ANN	Artificial neural network
AGONS	Algorithmically guided optical nanosensor selector
AU-fMWCNTs	Gold functionalised multi-wall carbon nanotubes
AuNCs	Gold nanocrystals
BPNN	Back-propagation neural network
BLSTM	Bidirectional long short-term memory
CBZ	Carbendazim
CNNs	Convolutional neural networks
CNT	Carbon nanotube
CRM	Chemical-responsive material
DCT	Discrete cosine transform
DFT	Density functional theory
Dual-em-IMP	Dual-emission molecularly imprinted polymers
FFNN	Feed-forward neural network
GMMs	Gaussian mixture models
HCA	Hierarchical cluster analysis
HF	Hydrofluoric acid
His	Histidine
HPLC	High-pressure liquid chromatography
kNN	k-nearest neighbour
LDA	Linear discriminant analysis
LOD	Limit of detection

LR	Logistic regression
LSPR	Localized-surface Plasmon resonance
MGO	Magnetic graphene oxide
mM	Millimoles
MOF	Metal organic framework
MLP	Multilayer perceptron
MRL	Maximum residual limits
MWCNT	Multiwall carbon nanotube
NAA	Naphthalene acetic acid
NanoIOT	Nanotechnology Internet of Things
NB	Naive Bayes
NIR	Near-infrared
nm	Nanometers
OPPs	Organophosphate pesticides
PCA	Principal component analysis
PEDOT	Poly(3,4-ethylenedioxythiophene)
PLSR	Partial least square regressor
PVA	Poly-vinyl alcohol
PVDF	Polyvinylidene diflouride
QDs	Quantum dots
RBF	Radial basal function
RFR	Random forest regression
RGB	Red–green–blue
RNN	Recurrent neural network
RVM	Relevance vector machine
SA	Salicylic acid
SERS	Surface-enhanced Raman spectroscopy
SVM	Support vector machine
SVD	Singular value decomposition
SVR	Support vector regression
SP	Spermine
TTIs	Time-temperature integrators
μΜ	Micro-moles
VOCs	Volatile organic compounds
ZEN	Zearalenone
Lasso	Least absolute shrinkage and selection operator
PLSR	Partial least square regression

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