IMPEDANCE-BASED MOISTURE CONTENT SENSOR ASSESSMENT FOR GAS-PHASE BIOFILTERS

BY

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THESIS

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ABSTRACT

A woodchips-based gas-phase biofilter is capable of mitigating airborne ammonia efficiently. The moisture content (MC) of the biofilter media is important to determine ammonia mitigation and nitrous oxide generation. It is critical to monitor real-time moisture content of the biofilter media for maintaining biofilter performance. The objectives of this research are to obtain a deep insight into the impedance-based moisture content measurement and to improve methodologies to monitor the moisture content of gas-phase biofilters. A sensor consisting of a sensing unit (three parallel plates) and a circuit generating DC voltage outputs was used in this study to measure moisture content. The sensor readings changed with step-wise increase of moisture content as well as different particle size distribution and nitrogen (ammonia-nitrogen, nitrate-nitrogen) concentrations of biofilter media. The results show that both particle size distribution and nitrogen concentrations significantly affected impedance-based moisture sensing. A mathematical model was formulated, which was able to demonstrate the relationship between the sensor reading and moisture content of the biofilter media. A model was established to predict the moisture content of the biofilter media based on sensor reading, ammonia-nitrogen concentration and nitrate-nitrogen concentration.

Keywords: Gas-phase biofilter, Impedance, Moisture sensor, Nitrogen compounds, Particle size distribution.

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

Chapter 1 INTRODUCTION

1.1 Background

According to emission estimates, agricultural production is a major source of atmospheric ammonia, with an approximate average of 32 Tg (Tg = 10^6 tonne = 10^{12} g) each year, which accounts for 60% of global ammonia emissions. Livestock production accounts for 21 Tg per year (Beusen et al., 2008). In the United States, concentrated animal feeding operations are the largest contributor (55%) to the agricultural ammonia emissions (Balasubramanian et al., 2015). Ammonia emissions have posed serious problems to the environment, e.g., reactive nitrogen cascade, which describes a multiple sequence effects on ecosystems (atmosphere, terrestrial ecosystems, and freshwater and marine systems) caused by reactive nitrogen (Galloway, 1998, 2003, 2013). A typical example of the reactive nitrogen cascade is the overgrowth of plants (eutrophication), which leads to depletion of dissolved oxygen and detrimental impacts on aquatic life and vegetation (Erisman et al., 2013). Additionally, ammonia emissions negatively impact human health through deposition and formation of particulate matters (Galloway et al., 2003; Pope III & Dockery, 2006), since enhancement of particulate matter concentrations corresponded to increased pulmonary and cardiac diseases (Pope III, 2000). In 2011, the Environmental Integrity Project (EIP) petitioned the United States Environmental Protection Agency (U.S. EPA) to consider regulating ammonia as a criteria pollutant under the Clean Air Act (EIP, 2011). At the same time, the U.S. EPA (2011) Science Advisory Board (SAB) stated that “the EPA presumption that NH_3 is not a $PM_{2.5}$ precursor should be reversed and states should be encouraged to address NH_3 as a harmful $PM_{2.5}$ precursor”. Given the concerns about the adverse impacts of livestock ammonia emissions, research related to control of the livestock-derived airborne ammonia emissions is critically needed.

Biofiltration is considered as a relatively low-cost, but efficient technology for the treatment of livestock ammonia emissions with concentrations of 0.1 to 30 ppm (Yang et al., 2013). The packing materials of the biofilter can be organic and inorganic, but organic media possesses superior ammonia mitigation performance as compared to inorganic packing media at the ammonia range of 0-300 ppm (Kim et al., 2000). The performance of a gas-phase biofilter is affected by several variables, such as pressure drop, biofilter media, moisture content, pH value, and temperature (Pagans et al., 2005). Pressure drop, an important characteristic of the biofilter operation, is determined by the particle size distribution of the biofilter media, biomass accumulation, particular matter loading, and media degradation (Maia et al., 2012; Sales et al., 2008; Yang et al., 2011). Woodchips are widely used as the primary media for agricultural biofilters to minimize air resistance while keeping high ammonia mitigation efficiency (Chen et al., 2009). The most critical variable is moisture content of the biofilter media, which largely determines the success of the biofilter operation (Yang et al., 2014). It was observed that an increase in moisture content from 40% to 50% (wet basis) significantly improved ammonia mitigation efficiency from 40% to 70%. However, further increasing moisture content above 60% had no obvious effect on its mitigation efficiency (Nicolai et al., 2006). Moreover, nitrous oxide, a powerful greenhouse gas (IPCC, 2007) can be produced when media moisture content changed from 52% to 65% (Maia et al., 2012). There is no precise moisture content setting for a biofilter operation, but a certain recommendable moisture content range for woodchips (hereinafter referred to as biofilter media) is required (Chen & Hoff, 2009). Previous research has found that moisture content from 35% to 65% was an appropriate range to balance the mitigation efficiency and generation of nitrous oxide (Yang et al., 2014).

Monitoring moisture content of the biofilter media would be possible if a real-time measurement sensor existed; ideally, it would be able to detect the moisture content in a certain sample volume representing many particles. Few soil moisture sensors are available for biofilter media (e.g. woodchips) due to the much larger and varied particle size compared to soil (Yang et al., 2011). Several moisture sensing approaches have been tested. Dual energy x-ray (Hultnäs & Fernandez-Cano, 2012; Nyström & Dahlquist, 2004) and the time domain reflectometry method (Reyes et al., 2000; Okamura, 2000) have been proven to detect moisture content accurately, but the high-cost hampers their application in agriculture. An impedance-based moisture sensor was recently developed to determine moisture content with low cost and its fundamental function has been tested (Yang et al., 2013). Previous studies showed that the sensor can be employed as a predictor of moisture content (Yang et al., 2014).

Few mature methods have been applied to the practical operation, because most of the performance tests were conducted under ideal conditions, that is, only with respect to moisture content, without consideration of other potential interference variables. In a real world scenario, some ammonia remains in the biofilter when it passes through the biofilter. In fact, a woodchips-based biofilter is comprised of different components: main amongst them are woodchips, free water, trapped air molecules, and different forms of nitrogen. The dominant forms of nitrogen in the biofilter are total ammonia-nitrogen (TAN, hereinafter referred to as ammonia-nitrogen) and nitrate-nitrogen (Yasuda et al., 2009; Yang et al., 2012). Baquerizo et al. (2009) found that more than half of the ammonia fed to the biofilter was oxidized to nitrate, and the rest was recovered as ammonium. As a result, there is a large amount of nitrogen loading in the biofilter that cannot be ignored in a practical operation. Recent research has shown that the maximum accumulating concentrations of nitrate-nitrogen and ammonia-nitrogen were 0.25 mg g^{-1} and 0.079 mg g^{-1} dry basis, respectively (Hood et al., 2011; Hood et al., 2015). However, limited research was conducted to set-up a series of experimental conditions similar to actual operating conditions.

To date, the impedance-based moisture measurement is a direct and feasible method as the above discussion attested. However, it is still very experimental and has a long way to go to before this method is put into practice. There are still considerations regarding the possible interference variables. The role of temperature (from 22℃ to 32℃) in affecting impedance measurement has been examined in a well-controlled environment, and the results suggest the impact of temperature was minor (Yang et al., 2013). But the effects of nitrogen enriching in the biofilter have not been tested. It is known that the impedance-based moisture measurement method takes advantage of dielectric properties of the biofilter media. In a biofiltration system, the dielectric constant (an important index for the dielectric properties) of liquid water (80.1 at 20ºC) is much higher than the air (1 at 20^{o} C) and woodchips (1 to 5). But the dielectric constant of ammonia is 16.61 at 20^{o} C (Billaud & Demortier, 1975), which may influence this sensing result. Such influence deepens as moisture content increases, since more ammonia dissolves in water. Apart from this, particle size distribution is another key issue which influences the impedance of biofilter media by changing the contact area between the sensing components of the sensor and the biofilter media. The selection of particle size distribution of the biofilter media may vary from one location to another. Due to these variables, the accuracy of this method should be evaluated. As a practical matter, the particle size distribution of the biofilter media should be determined before construction. Since accumulated nitrogen compounds have a negative effect on ammonia mitigation efficiency (Yang et al., 2012), farmers need to check the nitrogen concentration and replace the media regularly. Plenty of commercial nitrogen sensors as well as regular extension services are available for nitrogen measurement in practice, which provides the precondition for this study. It remains a major issue to make full use of the data regarding nitrogen concentrations and to build a relationship with the moisture content measurement.

1.2 Objectives

The overall objectives of this research were to obtain a deep insight into the impedance-based moisture content measurement and improve methodologies to monitor the real-time moisture content of a gas-phase biofilter. The performance of the moisture sensor regarding different particle size distribution of the biofilter media, and nitrogen loading, were tested and analyzed. Nitrogen loading includes: (1) ammonium hydroxide enriching, and (2) ammonium nitrate enriching. This was the first time that nitrogen enriching was considered as a factor on the moisture measurement in biofilters. A novel impedance-based moisture sensor developed by our group was employed in this research. The specific tasks included the following:

1) Experimentally determine the effect of particle size distribution of biofilter media on the performance of impedance-based moisture content measurement.

2) Experimentally determine the influence of nitrogen concentration of biofilter media on the performance of impedance-based moisture content measurement.

3) Develop a model to predict the moisture content based on ammonia-nitrogen concentration, nitrate-nitrogen concentration and the sensor reading of the impedance-based moisture sensor.

Chapter 2 LITERATURE REVIEW

2.1 Biofilter and ammonia mitigation

Biofiltration has been recognized as an effective pollution controlling method (Devinny et al., 1998). The biofilter study can be traced back to 1923, when the basic conception of controlling odorous emission by using soil beds was discussed (Leson & Winer, 1991). Biofilters were designed to deal with the issue of odors in both the United States and West Germany (Pomeroy, 1957). German scholars first proposed the use of a biofilter for livestock odor reduction in the early 1980s (Zeisig et al., 1988). In the United States, research on biofilters for livestock facilities started in the 1990s (Nicolai & Janni, 1997). Since then, biofiltration has gradually gained recognition in the United States. Recently, it has been listed by the Illinois State Office of the Natural Resource Conservation Service as a promising technology for livestock air pollution control (Yang et al., 2013).

In general, biofiltration includes biotrickling, bioscrubber, and gas-phase biofiltration (Mudliar et al., 2010). This study focuses only on the gas-phase biofilter, which is specifically designed for gas-phase biofiltration and operates on an air stream. The process of ammonia mitigation is a combination of sorption, degradation and desorption of gas-phase contaminants. The air is forced to pass through the biofilter media continuously via mechanical ventilation (Nicolai et al., 2006). The byproducts of the above reactions consist of water (vapor), carbon dioxide, mineral salts, etc. (Nicolai & Janni, 2001). This basic procedure is shown in the following Figure 2-1. Bench-scale, pilot-scale and full-scale studies of biofilter operations at agricultural facilities illustrate a high removal efficiency (RE) of ammonia, hydrogen sulfide and odor/VOC, respectively. Compared to other absorption and catalytic oxidation technologies, the low investment costs and operating costs is another advantage of biofilters preferred by farmers (Chen & Hoff, 2009).

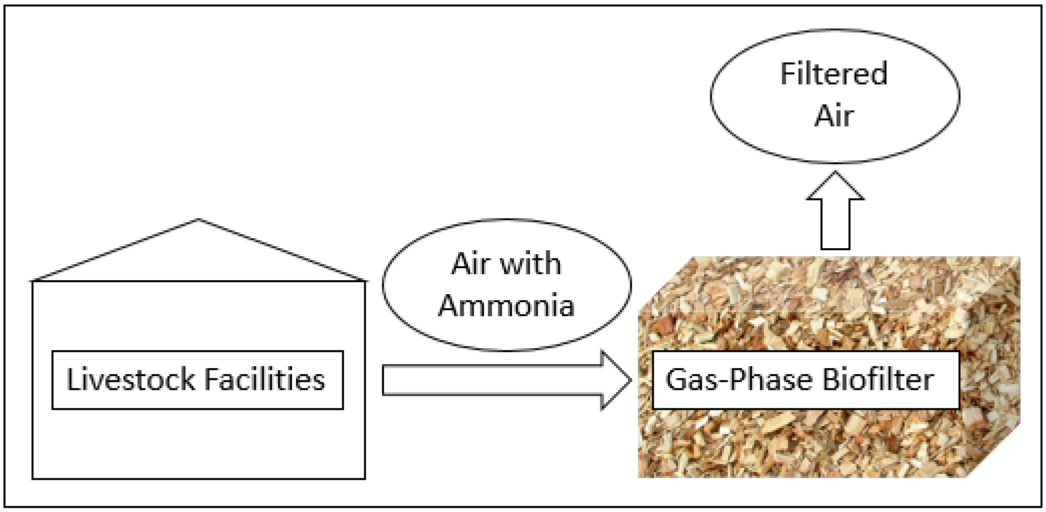


Figure 2-1. Schematic diagram of a biofilter.

In the past two decades, scientific researches show that gas-phase biofiltration is a useful method to mitigate ammonia from the composting process among numerous available technologies for gas treatment (Webster, 1996; Hong & Park, 2004; Pagans et al., 2005). Since it has the potential to deal with the high volumes of air pollutants at low concentrations (Maia et al., 2012), it is suitable for maintaining the air quality in Animal Feeding Operations (AFOs).

The primary mechanism of ammonia mitigation in a gas-phase biofilter is the absorption process. The contribution of the ammonia biodegradation process is significantly less than adsorption and the absorption processes (Pagans et al., 2007). The main byproducts of ammonia biofiltration are nitrate (NO_3^-) and nitrite (NO_2^-) ions resulting from microbial nitrification (Baquerizo et al., 2005; Maia et al., 2012). Of the two, nitrate is the dominant inorganic nitrogen form after a long-term (>8 month) operation (Liang et al., 2000). Yasuda et al. (2009) used a full-scale biofilter with rock wool packing materials to mitigate ammonia gas. This investigation suggested the concentration of NH_4^+ and NO_3^- were always higher (10 times) than NO_2^-.

Baquerizo et al. (2009) used a coconut fiber-based biofilter to conduct a comprehensive study to investigate the long-term stable and continuous ammonia removal under different ammonia loading conditions. The concentration ratio of 1:1 and 2:1 of ammonium and nitrate were found under low and high ammonia loading conditions in the leachate analysis, respectively. Yang et al. (2012) studied the transport and fate of N within a gas-phase biofilter made of woodchips. This study included two nitrogen enriching steps and one nitrogen depleting step as a swing test. This three-month experiment showed NH_4^+ and NO_3^- were the two major byproduct components in the biofilter media. Recent research conducted from a real swine barn in Raleigh, NC. demonstrated that the concentration of accumulated total ammoniac nitrogen (TAN) and nitrate-nitrogen could be up to 78.90 mg kg^{-1} and 250.66 mg kg^{-1} (dry basis), respectively (Hood et al., 2011; Hood et al., 2015).

2.2 Determinants of biofilter performance

The critical variables which influence the performance of biofilters have been investigated intensively in recent years. Biofilter media is one of the major factors. Kim et al. (2000) compared two organic (peat and rock wool) and two inorganic (fuyolite and ceramics) packing materials, and found that organic packing materials performed a higher ammonia mitigation efficiency than inorganic packing materials. One of the main reasons is the organic based materials are a better biological support system for microorganisms. A long-term operation of a biofilter regarding an ammonia removal test indicated compost and granulated sludge successfully served as a biofilter media with a high ammonia elimination capacity (Chen et al., 2005). Chen et al. (2009) developed a pilot-scale biofilter using woodchips as the carrier material for thirteen weeks, and they suggested a woodchip based biofilter was capable of achieving a high ammonia mitigation efficiency.

Another research issue that needs to be studied is the operation of the biofilter. The operation of biofilters need the support of agricultural ventilation fans. However, it is impractical to add booster fans to boost the pressure during operation. A suitable media selection, especially controlling the composition of the biofilter media is a practical way to control pressure drop across the biofilter. Nicolai and Janni (2001) found both pressure drop across the biofilter media and ammonia mitigation efficiency increased when more compost was included in the woodchips-compost media mixture. Yang et al. (2011) tested the airflow resistances of eleven biofilter media and their mixtures, and suggested particle size distribution and media compaction were significant in determining pressure drop during operation.

Maintaining the pH value of the biofilter media slightly higher than neutral improves the biofilter performance, but the exact impact of pH on the biofilter function and on microbial communities is far from certain, which involves complicated processes (Yang et al., 2014). The performance of the biofilter can also be influenced by the process temperature (Hong & Park, 2004; Pagans et al., 2006) and inlet air temperature (Nicolai et al., 2006). But in reality, biofilters are generally exposed to the atmosphere, without insulation. It is costly for farmers to control operation temperature of biofilters outdoors.

Among the variables, moisture content is regarded as the key determinant for biofilter performance (Wani et al., 1997). It affects the biofilter performance both chemically and biologically (Yang et al., 2013), since the biofilter can be considered as an aerobic reaction environment where air passes through continuously and microorganisms need water to maintain their metabolic reactions. If too much water fills the biofilter media pores, it will inhibit the transfer of reactants limiting the reaction rate; while too little moisture content deprives microorganisms of water, which can result in significant reduced, even completely impeded biological activities (Wani et al., 1997). Besides, nitrous oxide (N_2O), a greenhouse gas, can be produced during the nitrification and denitrification processes within a biofilter. The generation of nitrous oxide is closely related to media moisture content, especially at high moisture content conditions (Maia et al., 2012). There is no precise moisture content setting for a biofilter operation, but a recommendable moisture content range for certain biofilter media is required (Chen & Hoff, 2009). In general, optimal moisture content varies with different biofilter media (Table 2-1), depending on the characteristics of different medias (Hodge et al., 1991).

Table 2-1. Recommendable moisture content of different biofilter media.

Reference	Biofilter media	Recommendable moisture content
Prokop et al., 1985	Peat moss	40-60%
Bohn, 1992	Compost	40-50%
Chang et al., 2004	Chaff of pine & perlite	60-80%
Nicolai and Lefers, 2006	Mixture of compost & woodchips	35-65%
Yang et al., 2014	Woodchips	35-65%

2.3 Moisture sensor for biofilter

Maintaining the moisture content at a desired level requires a precise biofilter moisture content measurement. There has been many attempts to measure the moisture content of a biofilter media in real time. The traditional method is based on weight analytical techniques. In the traditional method, the biofilter media were dried in a 105^{o} C oven for 12 to 24 hours to determine weight loss. This method assumes all the water was removed after drying, and the final moisture content is zero, but it is tedious and time-consuming. Continuous weight-based sensing was introduced by using load cells to calculate the water loss during operation (Young et al., 1997; Classen et al., 2000). This method failed to consider the influence of dust loading and decomposition of media (Nicolai & Lefers, 2006). Because this method assumes all the losses in the biofilter weight should be ascribed to the losses of water.

Several soil moisture sensors have been developed (Wagner et al., 2007; Liu et al., 2008). However, it is inadequate to simply apply soil moisture sensors to biofilter media because of the much larger and varied particle size distribution of woodchips compared to soil (Yang et al., 2011). Even if the probe successfully contacted the biofilter media, local measurements are not suitable for global moisture content measurement. Great effort has been made to satisfy real-time measurement by previous researchers and several novel moisture sensors for biofilter media have been developed and tested.

Reyes et al. (2000) suggested using a time domain reflectometry (TDR) probe to monitor a mixed biofilter media consisting of 60% compost and 40% pearlite, which can reflect the real moisture content in real time. But such a biofilter media mixture is not ideal for practical application due to the excessive pressure drop. D'Amico et al. (2010) successfully stimulates a wire probe with pulse signals to measure round trip time to refer the moisture content. This approach has the advantages of short response time and low cost, but the measurement variation increased significantly when the moisture contents exceeded 40%.

Hultnäs and Fernandez-Cano (2012) tested and evaluated the Mantex Desktop Scanner based on dual energy X-ray with pine woodchips. They reported that no obvious difference was observed between this method and the gravimetric method. This method was also applicable for different temperatures (frozen and room temperature). However, the setup of this method requires hospital instruments, which are expensive.

Hartmann and Böhm (2000) verified and compared methods based on the thermo-gravimetric method (freeze drying, infrared drying and microwave drying), the electric method (capacitive methods, microwave method and TDR), and the optical method (infrared reflectometric method). They determined the results of the thermo-gravimetric are accurate since all of them are based on drying effect, and the electric methods need bulk density variations of the sample. The infrared reflectometric method was the most accurate method, but needed decisive technical design. Comparisons of different methods and their application are listed in Table 2-2.

Table 2-2. Comparisons of different methods for moisture content measurement.

Methods	Can be applied on-line	Applicable on bulk (B) or flow (F)
Dual energy x-ray	YES	B, about 2 kg
Gravimetric methods	NO	B, F
Indirect moisture measurement	YES	B
Microwaves	YES	F
Near Infrared Spectroscopy	YES	F
Nuclear Magnetic Resonance	YES	B, F
Radio Frequent Electromagnetic Waves	YES	B, F

Note: B represents method reported applicable on bulk measurement while F means the measurements were suited in a flowing material (D'Amico et al., 2010).

Robert et al. (2005) tested five different types of moisture meters (Lincoln Irrigation soil moisture meter, Farmex HMT-3 digital hay moisture meter, Campbell Scientific Hydrosense digital soil moisture meter, Vaisala Hummiter 50Y relative humidity probe, and a site-built radio-frequency large-area capacitive plate sensor) in a biofilter media (the byproduct of stripping wood of its bark for processing). It was determined that all the soil and hay moisture meters and relative humidity probes in their experiment were inaccurate. The large-area capacitive plate sensor showed clear results, and was promising to be applied over energized frequencies of 300 kHz to 15 MHz. Funk et al. (2007) developed and tested the Hummiter 50Y relative humidity probe and the site-built radio-frequency large-area capacitive plate sensor for several months to evaluate their stability. They suggested the output of the relative humidity probe fluctuated with the relative humidity of the incoming airstream while the capacitor sensor performed well to estimate the moisture of the biofilter media.

Yang et al. (2013) refined the sensing unit large-area capacitive plate sensor into three parallel plates and developed the sensor circuit by considering the influences of conductance of the media. An impedance-based sensor was proposed and tested under different conditions (temperature and compaction of media). The results suggested that the sensor is a reliable predictor to reflect the real-moisture content within a certain range (35-65%). However, all the tests did not take into account the role of other important variables in affecting impedance measurement, since large amounts of ammonium and nitrate ions accumulated in the biofilter media.

To date, none of these methods developed have been perfected enough to be put into practice. More research on moisture sensors is needed, especially on a low-cost real-time continuous measurement, and on an agricultural application with accurate measurement, e.g., livestock facilities.

2.4 Ionic strength and dielectric properties

The nitrogen in biofilter media exists in the form of ions. To quantify the ions, the concept of ionic strength is induced by electrolytic chemistry, which has been a foundation in this subject (Adams, 1971). The ionic strength is defined as μ, a function of the concentration of all presented ions in the solution.

$$\mu = \frac{1}{2}\sum_{i=1}^{n} c_i z_i^2 \qquad \textbf{(2.1)}$$

Where c_i is the molar concentration of ion i (M, mol/L), z_i is the charge number of that ion, and the sum is taken over all ions in the solution.

Ionic strength is one of the important characteristics of a solution with dissolved ions since it provides the approaches for calculating ionic activities. The ionic strength principle developed by Lewis and Randall (1961) suggests the activity coefficient of the mixed electrolyte solutions is the same as the activity coefficient of the pure electrolyte when they are at the same ionic strength. For some electrolytes, the ionic strength principle provides reasonable values at low ionic strength. Several studies related to ionic strength have shown ionic strength has impacts on adsorption of phosphate and sulphate by soils (Bolan et al., 1986), anions on zinc adsorption by soils (Shuman, 1986), boron adsorption by clay minerals and soils (Goldberg et al., 1993), and sorption of cadmium of soils (Naidu et al., 1994). There are limited studies in the literature that experimentally investigate the influence on nitrate and ammonium ions absorption or adsorption by woodchips.

Dielectric material is material that has the ability to store energy when exposed to an external electric field. The dielectric constant is an expression to describe electric flux density of materials. It is defined by the given equation:

$$\boldsymbol{\varepsilon_m^a = \sum \varepsilon_i^a v_i} \tag{2.2}$$

Where ε_m is the dielectric constant of the material, i means the each component of the material, v represents the volume fraction of each component, and constant a is close to 0.5.

Electrical impedance is defined as the complex ratio of voltage to the current in an alternative current, which is described by magnitude and phase (frequency). The electrical resistance is defined as the ratio of voltage to the current in a direct current circuit, which is the real part of impedance; while the reactance is the imaginary part of impedance. In summary, impedance is the combination of resistance and reactance. Typically, capacitance is a kind of reactance, which is determined by the dielectric constant of material.

The dielectric properties of woodchips have been studied in combination with moisture content and temperature (Anagnostopoulou-Konsta & Pissis, 1988; James, 1975; Tsutsumi & Watenabe, 1965). Literature suggests temperature influences the dielectric to a minor degree. However, moisture content affects the dielectric constant of the biofilter dramatically since the biofilter is a mixture of water and organic material. The dielectric properties of water is 80.1 at 20℃, while organic material is from 1 to 5. Ultimately, increasing moisture content can increase the dielectric constant.

Influence on the dielectric properties from the frequency of applied electric fields cannot be neglected. Torgovnikov (1993) stated that changes of dielectric properties of wood resulted from ionic conductivity, wood dipoles, interfacial and electrolytic polarization. Sacilik and Colak (2010) observed loss factor and tangent were greater at lower frequencies than at higher frequencies when they determined dielectric properties of corn seeds from 1 to 100 MHz. However, limited research investigated the effects of ions on dielectric properties of woodchips. Oh et al. (1993) found increasing ionic strength of bathing medium (0.1 M NaCl) resulted in decreased resistance and unchanged capacitance. A previous study found impedance frequency of 100 kHz is appropriate for biofilter media moisture sensing (Yang et al., 2013).

Limited research studies have been carried out to build relationships between ionic concentration and impedance. Pandey et al. (2013, May) proposed an approach to estimate the soil ionic concentration by using the quasi-static dielectric mixing model and the measured multi-frequency impedance to infer the different ionic concentrations. This method was good for estimation of individual components such as air, water and nitrates ions. And they developed this dielectric mixture model based approach to detect the soil moisture and nitrates in real time (Pandey et al., 2013, Oct). With less than 12% error, this method can determine the nitrate solution accurately.

2.5 Summary of literature review

Review of the topics provided the following conclusive background knowledge for the study of this research. Gas-phase biofiltration is an effective method for livestock ammonia mitigation from air sources. In biofilter systems, a majority of nitrogen exists in the forms of ammonia-nitrogen and nitrate-nitrogen; particle size distribution and moisture content of biofilter media are the most important factors for a biofilter operation. An impedance-based sensor (with

100 kHz voltage frequency) is a promising technology to reflect the real-moisture content within a certain range (35-65%). But previous studies failed to consider the influences of particle size distribution and nitrogen concentrations of biofilter media on sensor performance. Limited studies have been conducted on the relationship between nitrogen concentrations and impedance.

Chapter 3 MATERIALS AND METHODOLOGIES

3.1 Project overview

Figure 3-1 shows the schematic overview of this study. To begin with, three sets of impedance-based moisture sensors were built and calibrated before subsequent experiments. The influences of different particles size distribution and different nitrogen loading were investigated. In order to understand the impact of different particle size distribution on the impedance of biofilter media, two scenarios of particle size distribution were analyzed. To study the impact of different nitrogen loading on biofilter media impedance, the procedures were divided into four steps. The first step was aimed to compare the impedance of biofilter media with same ammonia-nitrogen but different nitrate-nitrogen. In order to gain a deeper insight into the influences of ammonia-nitrogen and nitrate-nitrogen individually, ammonium hydroxide enriching, and ammonium nitrate enriching were investigated, respectively. A mathematical model which describes the relationship between sensor reading and moisture content was developed, and it served as a precursor of the final model. Finally, to quantitatively correlate the ammonia-nitrogen concentration, nitrate-nitrogen concentration, and moisture content with the sensor reading, a statistical model was developed.

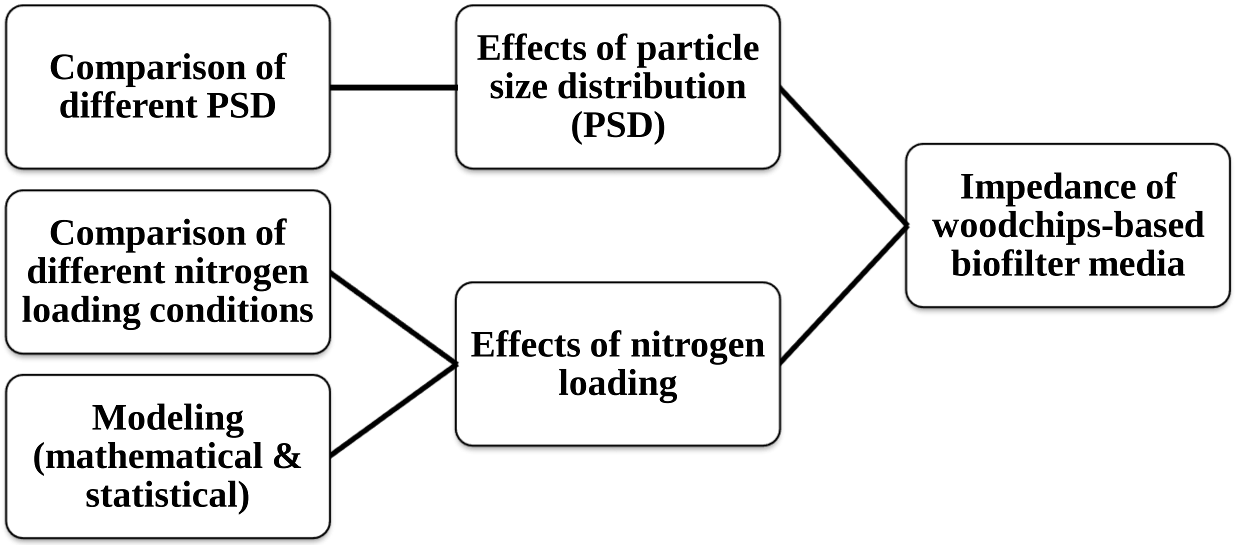


Figure 3-1. Project overview.

3.2 Media selection

A mixture consisting of shredded and chipped woodchips was selected as the biofilter media in this study. Woodchips were obtained from a landscape recycle center (Grounds Storage Barn) in Urbana, IL. They were dried naturally to 10-15% wet basis moisture content for 3-5 days (Figure 3-2). The media were sieved using a Penn State Forage Particle Separator (Product No. C24682N, Nasco, Fort Atkinson, WI.). The separator consisted of four trays which stack on top of each other as shown in Figure 3-3.

Figure 3-2. Woodchips were dried naturally to 10-15% wet basis moisture content.

Figure 3-3. Penn State Forage Particle Separator.

The top three trays have different size holes (1.9 cm, 0.8 cm and 0.2 cm, respectively) to separate media samples into four particle size ranges (Figure 3-4): (a) larger than 1.9 cm, (b) 0.8-1.9 cm, (c) 0.2-0.8 cm, and (d) smaller than 0.2 cm. Two media groups (particle size distribution: 0.2-0.8 cm and 0.8-1.9 cm) were selected for this study.

Figure 3-4. Media separated into four particle size ranges.

3.3 Impedance-based sensor construction

The impedance-based moisture sensor was employed as an indicator of impedance of the biofilter media. The sensor was designed based on the impedance of sensing materials, and it was composed of a circuit and a sensing unit (Yang et al., 2013). The circuit unit of this sensor consisted of a voltage-divider circuit and a peak-detector circuit. The principle of the circuit unit is to compare the impedance of biofilter media mounted between sensor plates $Z_{biofilter}$ to that of a reference capacitor Z_{ref}. The sine-wave AC voltages with ±2.0V P-P were used, and they were converted to two DC voltages V_{in} and V_{out} by voltage divider circuit. The voltages can be recorded through a data acquisition system (Personal Daq/56, Measurement Computing, Norton, MA.). The acquired signals then were applied in the following equation to calculate the ratio.

$$reading = \frac{|V_{in}|}{|V_{out}|} = \frac{|Z_{biofilter}| + |Z_{ref}|}{|Z_{biofilter}|} = 1 + \frac{|Z_{ref}|}{|Z_{biofilter}|} \quad \textbf{(3.1)}$$

Previous tests on the sensor showed that the impedance was very distinguishable at a high frequency of 100 kHz (Yang et al., 2013). The ratio of V_{in} / V_{out} was a function of media impedance, which was highly related to moisture content of biofilter media. The sensing unit consisted of three parallel steel plates (80% hollow with 2.54 mm holes), separated by sets of three plastic bars between each plate pair. The top and the bottom plates were grounded while the middle plate was connected with above circuits. The size of each plate was 30 cm × 30 cm, the distance between the center and side plates was 7.5 cm (Figure 3-5). The sensing plates were installed in the center of a sealed plastic testing chamber (62.5 cm × 47.6 cm × 35.2 cm, L × W ×H) filled with biofilter media (Figure 3-6). The height between the lowest plate and the bottom of chamber was 5 cm, while height between the highest plate and the top of the biofilter media was also 5 cm. A total of three sets of sensors were used for this study (Figure 3-7).

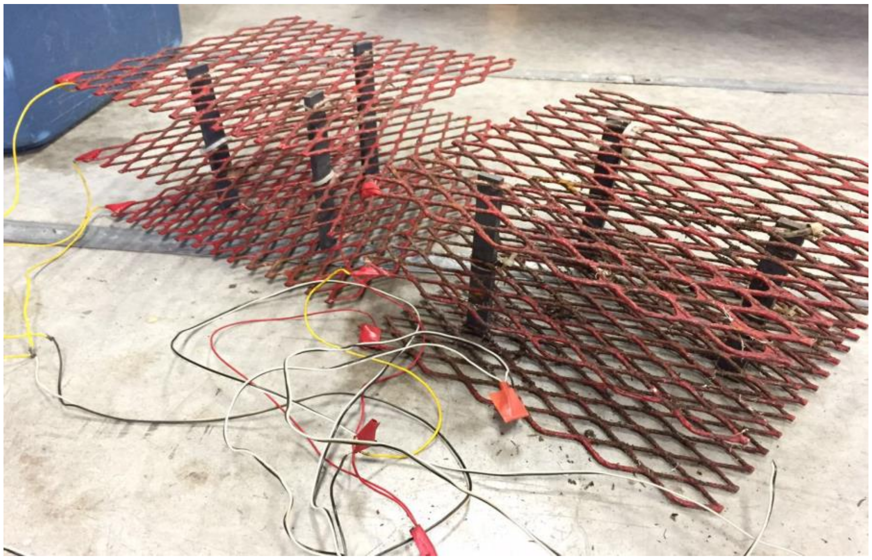


Figure 3-5. Sensing unit of the impedance-based moisture sensor.

Figure 3-6. Plastic testing chambers for biofilter media and sensing unit (5 cm from the lowest plate to the bottom of chamber, 5 cm from the highest plate to the top of the biofilter media).

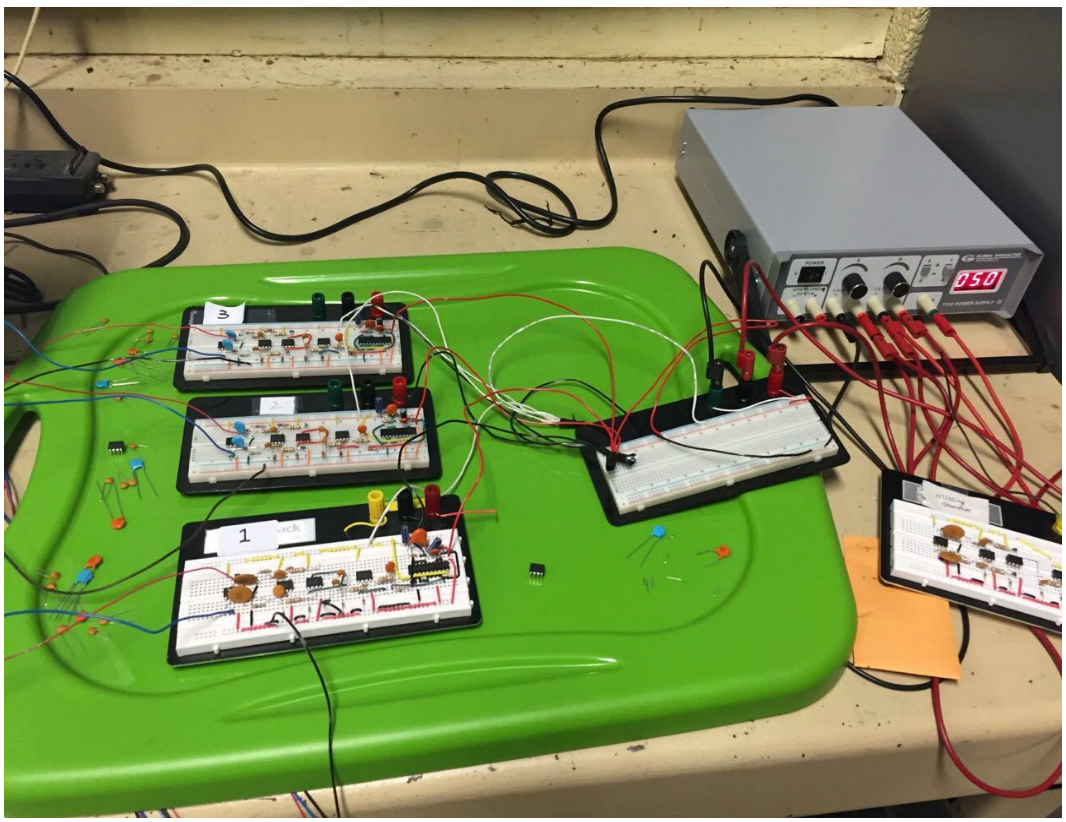


Figure 3-7. Circuit units of the impedance-based moisture sensor (energized by 5 Volt DC power supply).

3.4 Particle size and moisture content

To control the particle size distribution of the biofilter media, two groups (particle size: 0.2-0.8 cm and 0.8-1.9 cm) were re-mixed at a volume ratio of (1) 1:1 and (2) 1:4. The moisture content of the woodchips was increased gradually by adding DI water manually in a 5% increments. For each operation, the biofilter media were taken out of the testing chamber and mixed with water on a tray, and then placed back into the chamber as soon as possible. The sensor outputs V_{in} and V_{out} were continuously recorded every 10 min for 2-3 days. For each batch, the biofilter media was sealed by a plastic testing chamber, and located at normal room temperature (22ºC).

3.5 Nitrogen enriching

Biofilter media with a 1:4 mixture ratio was used for this test. Nitrogen enriching was carried out by adding: (1) ammonium hydroxide and (2) ammonium nitrate. The objective of adding ammonium hydroxide was to introduce only the ammonia-nitrogen into the biofilter media and determine the influence of ammonia-nitrogen on the impedance of the biofilter media. But the ammonium hydroxide would certainly increase the pH value of the biofilter media. The ammonium nitrate enriching was to introduce both the ammonia-nitrogen and nitrate-nitrogen at the ratio of 1:1 (stoichiometry ratio of ammonium and nitrate) into the biofilter and to study their influence. The biggest challenge of this series of experiments was that neither the added ammonia-nitrogen nor the nitrate can be completely absorbed/adsorbed by the biofilter media. Nitrogen might be lost in the forms of ammonia gas and leachate during the mixing and measurement steps. The measured nitrogen concentration may have deviated from the desired concentration based on calculation.

Moisture content of the biofilter media was increased with a 5% increment. To increase the moisture content, the biofilter media were taken out of the testing chamber and mixed with water as evenly as possible, and then placed back into the chamber. The ammonium hydroxide/ammonium nitrate was added at the moisture content of 30%. Sensor outputs V_{in} and V_{out} continuously recorded every 10 min for 2-3 days. Limited by the time and experimental conditions, at most three batches were available for each scenario. Each batch was a treatment, with certain nitrogen loading. There was no replication for each batch, because it is not attainable to control the emission of the ammonia during operation. For the purpose of covering wider ranges of nitrogen concentrations and some special scenarios, the parameters of each biofilter batch as well as the procedure of each experiment are shown in Table 3-1:

Table 3-1. Summary of different scenarios of nitrogen enriching treatments (nitrogen concentration: mg ammonia-N/nitrate-N per g dry media).

Testing batch	Scenario 1: Nitrogen loading	Scenario 2: Ammonium-hydroxide	Ammonium-nitrate	
			Scenario 3:	Scenario 4:
Batch A	Control group	0.25 ammonia-N	0.0625 nitrate-N + 0.0625 ammonia-N	1.25 nitrate-N + 1.25 ammonia-N
Batch B	0.75 ammonia-N	0.50 ammonia-N	0.50 nitrate-N + 0.50 ammonia-N	1.75 nitrate-N + 1.75 ammonia-N
Batch C	0.75 nitrate-N + 0.75 ammonia-N			

Scenario 1: To analyze the influence of nitrogen loading on the impedance of biofilter media, batch B was treated with 0.75 mg ammonia-N per gram dry media of ammonium hydroxide enriching, and batch C was treated with 0.75 mg nitrate-N per gram dry biofilter media of ammonium nitrate enriching. The moisture content of the biofilter media ranged from 35% to 65%.

Scenario 2: To analyze the change of the impedance of the biofilter media caused by ammonia nitrogen, two concentration levels of ammonia hydroxide enriching were used for the test: the biofilter batch A was treated with 0.25 mg ammonia-nitrogen per gram dry biofilter media, and the biofilter batch B was treated with 0.50 mg ammonia-nitrogen per gram dry biofilter media. The moisture content of the above batches ranged from 30% to 65%.

Scenario 3: To analyze the influence of the nitrate-nitrogen associated with ammonia-nitrogen on the impedance of the biofilter media, three concentration levels of ammonium nitrate enriching were used for the test: the biofilter batch A and B were treated with nitrate-nitrogen associated with the same ammonia-nitrogen at the concentration of 0.0625 and 0.5 mg per gram dry biofilter media, respectively. A slight adjustment regarding this series of experiments was that the increment for moisture content was uneven. The starting moisture content for measurement was 35%, and then all were increased to 50% immediately.

Scenario 4: To analyze the influence of a high concentration of nitrate-nitrogen associated with ammonia-nitrogen on the impedance of the biofilter media. Two concentration levels of ammonium nitrate enriching were tested: batch A and batch B were treated with nitrate-nitrogen associated with the same ammonia-nitrogen at the concentration of 1.25 and 1.75 mg per gram dry biofilter media. In this scenario, the concentrations were far above those in scenario 3, so this scenario cannot be treated as the same as scenario 3. The moisture content of the above batches was ranged from 35% to 65%.

3.6 Sampling and analysis

3.6.1 Sampling

Media were sampled from the upper (2 samples), middle (2 samples), and lower layers (2 samples) of the testing chamber (Figure 3-6). The weight of each sample was 30 gram. Then the samples were re-mixed as a whole. The moisture content was measured two hours after each operation, while the nitrogen concentrations and the pH of the biofilter media were measured 36 hours after each operation.

3.6.2 Moisture content measurement

Wet-basis moisture content is adopted in our study, which is described by the percentage equivalent of the ratio of water m_w to the total mass m_t. The formula for wet-basis moisture content is:

$$\boldsymbol{MC = \frac{m_w}{m_t} \times 100\% = \frac{m_w}{m_w + m_d} \times 100\%} \qquad \textbf{(3.2)}$$

Where, m_d is the final mass of dry woodchips that cannot lose weight anymore in the oven. A practical formula applied in this study is:

$$\boldsymbol{MC = \frac{m_{ini} - m_d}{m_{ini}} \times 100\%} \qquad \textbf{(3.3)}$$

Where,

m_{ini} represents mass of woodchips in the initial condition.

30 g sample was dried in a 105ºC oven for 24 hours (Figure 3-8) to determine the wet-basis moisture content of the media (Blake, 1965). Three samples were collected and measured in each test.

Figure 3-8. Oven for drying biofilter media.

3.6.3 Nitrogen concentrations and pH measurement

Nitrogen concentrations were measured based on modified TMECC 04.02 standards (Thompson et al., 2001). A 4 g sample of woodchips was collected and nitrogen was extracted using 40 ml DI water.

Each sample was mixed using a mixer for five minutes to dissolve the molecules, ions and gases in the DI water. The mixture was centrifuged (3000 rpm for 30 minutes), and the pH value of supernatant was measured using a pH meter (PH1100 Series, Oakton Instruments, Vernon Hills, IL.) according to the TMECC 04.11 method (Thompson et al., 2001). The nitrogen concentrations of filtrate were analyzed in a Hach DR/2010 spectrophotometer (Hach Co., Loveland, CO.) Ammonia-nitrogen was measured using method 8155 [0~0.50mg/L], and nitrate-nitrogen was measured using method 8171 [0~4.50mg/L]. Each compound concentration in the media was calculated as:

$$C_{actual\ nitrogen} = \frac{C_{measured\ nitrogen} \times 0.4}{4 \times (1 - MC(\%))} \quad \textbf{(3.4)}$$

Where,

$C_{actual\ nitrogen}$ is the actual nitrogen concentration (mg/g) of biofilter media, while $C_{measured\ nitrogen}$ is the measured nitrogen concentration (mg/L) of the solution. What should be noted is that the unit of nitrogen concentration is milligram (nitrogen) per gram (dry media).

3.7 Mathematical model

To our knowledge, a model coupling the sensor readings along with the moisture content of biofilter media, as well as, the different forms of nitrogen concentrations has not yet been developed. This is the first time a model has been developed to describe the relationships among these variables. The relationship between sensor reading and moisture content can be established by a mathematical model. Since the causation between nitrogen concentrations and sensor reading is far from certain, to make use of the data, statistical models were used to explore correlation patterns.

The impedance of the biofilter can be regarded as a simplified parallel connection of a resistor and capacitor with single time constant (Kandala et al., 1996; Yang et al., 2013).

Equations 3.1, 3.5 and 3.6 are the impedance sensing principle of the impedance-based moisture sensor.

$$Z_{ref} = \frac{1}{j\omega C_{ref}} = \frac{1}{j2\pi f C_{ref}} \tag{3.5}$$

Since the impedance of the biofilter media can be regarded as a parallel connection of resistor and capacitor, the impedance can be calculated as:

$$Z_{biofilter} = R_{biofilter} // \frac{1}{j\omega C_{biofilter}} = \frac{R_{biofilter} \times \frac{1}{j\omega C_{biofilter}}}{R_{biofilter} + \frac{1}{j\omega C_{biofilter}}} \tag{3.6}$$

Where,

C_{ref} : Capacitance of reference capacitor (constant)

ω : Angular frequency

$R_{biofilter}$: Resistance of biofilter media

f : Frequency of the imposed alternating field

j : Index of imaginary part, the square root of -1

This simplified parallel connection model is the prerequisite for subsequent mathematical model deduction.

3.8 Statistical model

A t-test was applied to determine the influence of particle size distribution of the biofilter media. Multiple linear regression (MLR) was used to model the relationship among sensor reading, moisture content and nitrogen concentrations. Linear regression was also used for evaluating the predictive model. Data analysis was carried out in R statistical environment (R Studio, Boston, MA) and Origin 2016 (OriginLab Corporation, Northampton, MA). Packages "forecast" (Hyndman et al., 2015) in R was applied for data treatment, which generated the predicted value of the sensor reading.

Chapter 4 RESULTS AND DISCUSSION

4.1 Sensor setting and testing

To optimize the sensor response regarding the biofilter media in this study, three levels of reference capacitance (0.47 nF, 1.0 nF, and 2.2 nF) were tested. Woodchips, 0.2-0.8 cm and 0.8-1.9 cm in diameter were mixed at a 1:1 volume ratio as the biofilter media. For each moisture content step, experiments were conducted for 3-5 days. Data were recorded every ten minutes when the sensor reading became stable. Figure 4-1 gives the response characteristics.

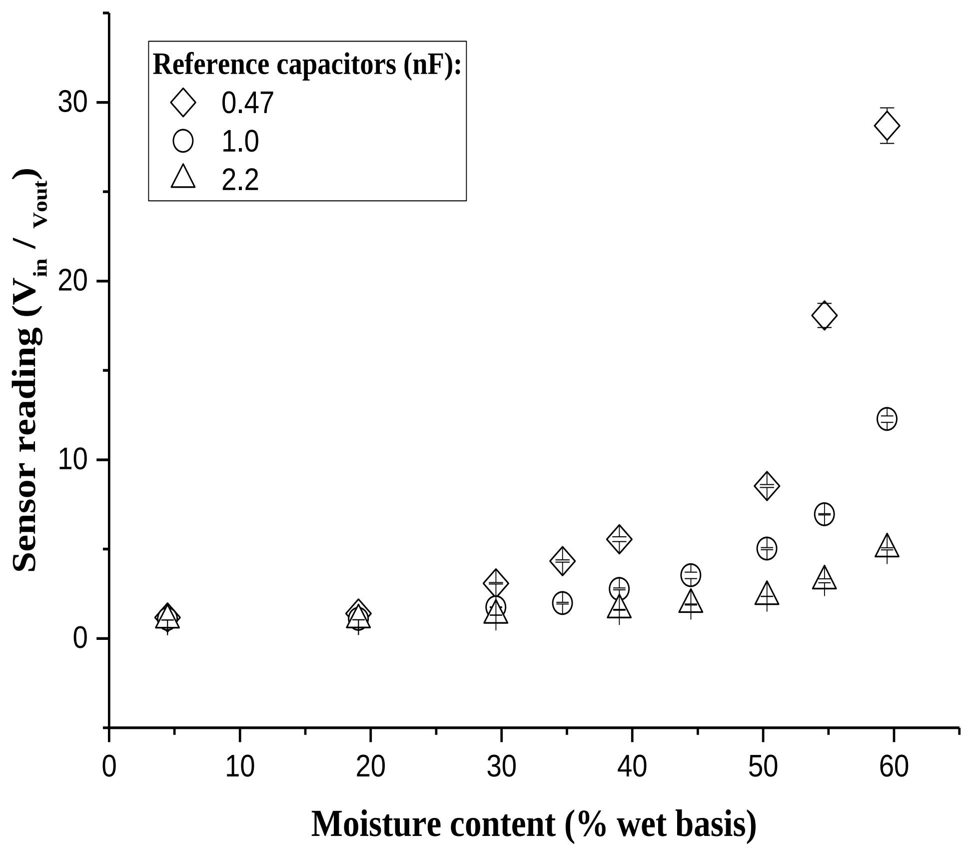


Figure 4-1. Sensor response at different moisture content conditions (mean and standard deviation).

All sensor outputs associated with three different capacitors produced a positive relationship between sensor reading and moisture content. Sensor response was flat for dry woodchips but increased rapidly with increased moisture content from 30% to 60%. This result agrees with the results in previous literature (Yang et al., 2013). Among the three reference capacitors, sensor responses with 0.47 nF and 1.0 nF showed relatively higher sensitivity than 2.2 nF at a moisture content ranging from 35% to 60%, which is the optimal moisture content range for the biofilter. But with the highest variation, the sensor responses corresponding to 0.47 nF might not be accurate.

The correlation between sensor reading and the moisture content of woodchips has been studied by former research fellows in our group by using an existing sensor. However, some parts of the existing sensor are no longer available for purchase (e.g., MAX 038 EPP High-Frequency Waveform Generator). As a result, the immediate task was to build two more sensors with similar levels of performance as the existing one. The newly-built sensors were used to increase the efficiency of subsequent experiments presented in this study (i.e., continuously and simultaneously monitoring more biofilters). The goal of performance testing was to validate the performance of the newly-build sensors was the same as the existing sensor.

Figure 4-2 gives the performance characteristics of these three sensors. “a”, “b”, and “c” represented the existing sensor and the two newly-built sensors, respectively. The value of reference capacitors are equivalent to reference impedance of biofilter media. All the capacitors were standard capacitors. The above three levels of reference capacitance (0.47 nF, 1.0 nF, and 2.2 nF) were selected for the experiments. Sensor readings were collected every two minutes. There was a total of 60 repeated samples (2 * 60 = 120 minutes) collected for each point. The average, as well as standard deviation, was calculated and are shown in Figure 4-2.

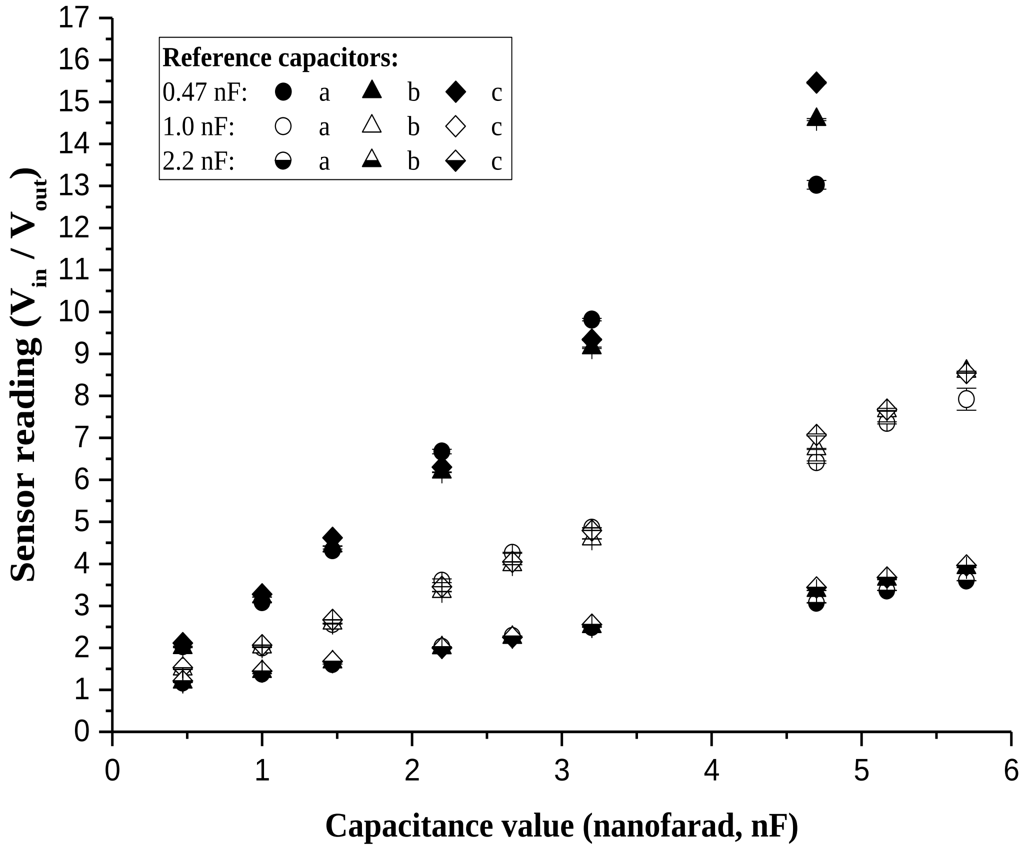


Figure 4-2. Performance characteristics of three sensors.

Note: “a”, “b”, and “c” represent the existing sensor, and two newly-built sensors, respectively

The variation (standard deviation) of the sensor readings were small in all test points. For sensors with 0.47 nF reference capacitors, they had the highest sensitivity among the three reference capacitance levels. However, it was found that the higher capacitance value, the greater the difference among the sensor readings. In particular, when the testing capacitance exceeded 3.2 nF, the difference was obvious. It suggested that 0.47 nF reference capacitance was inapplicable for higher capacitance detection, which corresponds to high moisture content of the biofilter media. These errors cannot be eliminated manually since the newly-built sensors were constructed with the same parts (e.g., same breadboards and same chips). To compare the performance characteristics of the sensor with 1.0 nF and 2.2 nF reference capacitors, a multiple linear regression (MLR) model was employed (Equation 4.1). Table 4-1 shows the differentiation of these two reference capacitance levels.

$$reading = \frac{|V_{in}|}{|V_{out}|} = \frac{|Z_{capacitor}| + |Z_{ref}|}{|Z_{capacitor}|} = 1 + \frac{|Z_{ref}|}{|Z_{capacitor}|} = 1 + \frac{|C_{capacitor}|}{|C_{ref}|} \quad \textbf{(4.1)}$$

Table 4-1. Differentiation of sensor performance characteristics.

Reference capacitances		Estimate	Std. Error	T value	Pr(>|t|)	
1.0 nF	(Intercept)	-0.45349	0.10382	-4.368	0.000178	***
	Sensor b	0.02902	0.10706	0.271	0.788	
	Sensor c	0.11885	0.10706	1.110	0.277	
	Capacitance	1.35612	0.02122	63.902	< 2e-16	***
2.2 nF	(Intercept)	-0.192750	0.037078	-5.199	1.99e-05	***
	Sensor b	0.099561	0.038235	2.604	0.0150	*
	Sensor c	0.127370	0.037078	3.331	0.0026	**
	Capacitance	0.521970	0.007579	68.872	< 2e-16	***

Note: “Sensor b” and “Sensor c” represent the two newly-built sensors.

The regression analyses of the data show that the 1.0 nF reference capacitor was the best choice for these three sensors. For the sensor with 2.2 nF reference capacitor, there were significant differences among these three sensors. With 1.0 nF reference capacitor, the sensor readings depended primarily on the capacitance of the measured object. An additional advantage of the 1.0 nF reference capacitors was the higher sensitivity of the sensor response. The results obtained from the experiments suggest that the three sensors with the 1.0 nF reference capacitors was appropriate for subsequent experiments.

4.2 Sensor response to particle size distributions with changing moisture

By using the Penn State Forage Particle Separator, woodchips can be separated into four ranges by diameter: larger than 1.9 cm, 0.8 - 1.9 cm, 0.2 - 0.8 cm, and smaller than 0.8 cm. A preliminary experiment was performed to determine the percentage ratio of each diameter range above is close to 1:4:1:0.5 (by volume). It can be assumed that similar particle size distribution of woodchips will be used to construct a biofilter facility if farmers want to save on the expense and pick up woodchips from the same landscape recycling center. It is presumed that the particle size distribution of woodchips would change the impedance of the moisture content at different degrees, but there is limited research concerning this important factor. Thus, studying the influence of particle size distribution of woodchips on sensor readings allows a more intuitive understanding of the impedance-based moisture measurement method.

Since the shape of woodchips whose particle size is larger than 1.9 cm are irregular, they were discarded in this study to reduce uncertainty. Woodchips whose particle size was smaller than 0.8 cm also needed to be discarded, as they would add additional difficulty to experimental manipulations. It is known that the smaller particle has a larger specific surface area, which means a larger water-holding capacitor. An uneven mix of different woodchip particles will result in an uneven distribution of moisture content, which introduces errors to the results. For convenience, woodchips in two size ranges of 0.8-1.9 cm and 0.2-0.8 cm were mixed with a 4:1 ratio in volume in order to be close to the natural particle size distribution of woodchips.

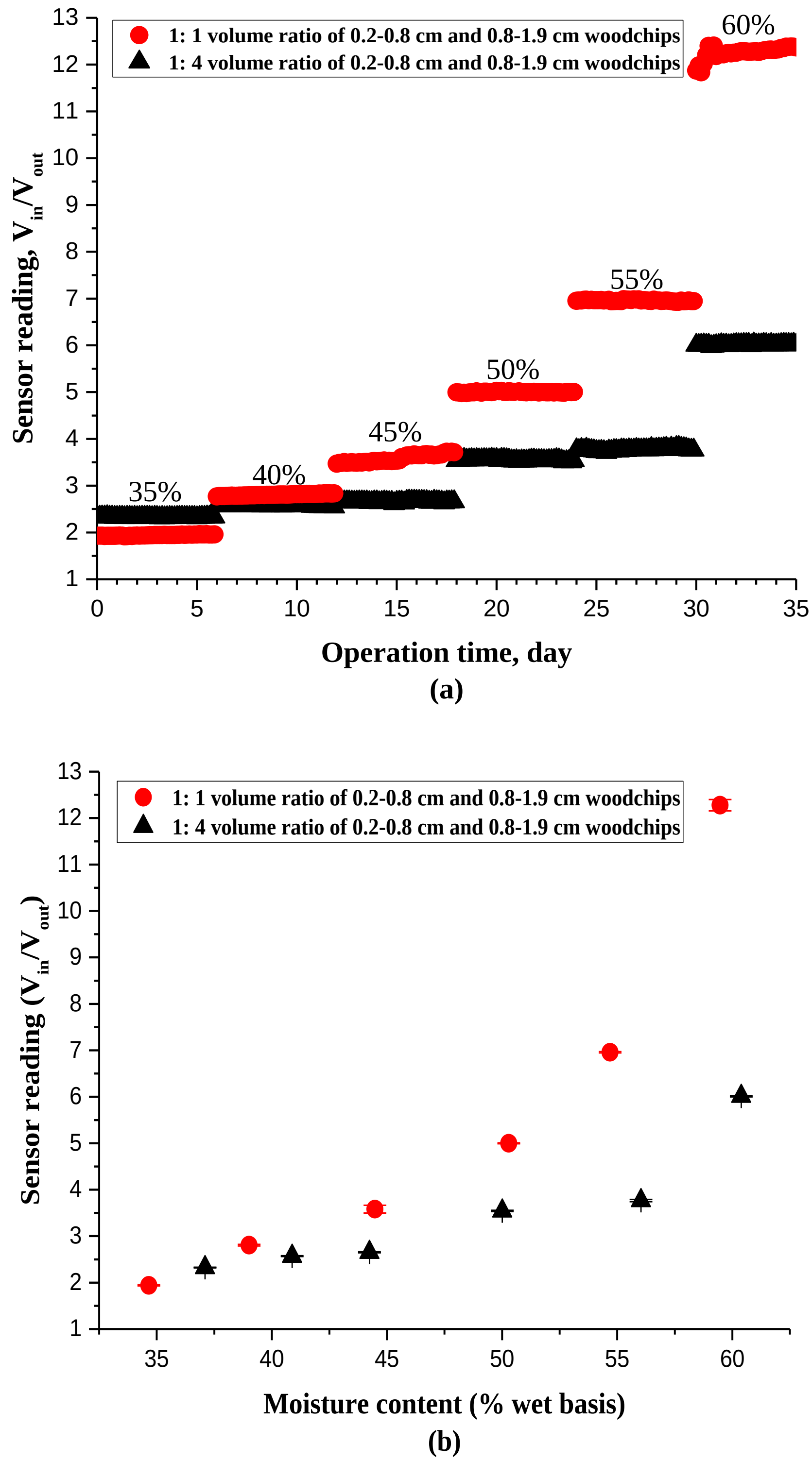


Figure 4-3. Sensor responses to two different particle size distributions: (a) real-time sensor reading at varied moisture content, and (b) mean and standard deviation of sensor readings at varied moisture contents.

Figure 4-3 shows that there was an obvious difference between the sensor readings (V_{in}/ V_{out}) of these two batches with different particle size distributions. The sensor reading remained steady during each moisture measurement with very small standard deviations, which indicated the stability of the sensor. For moisture content ranging from 40% to 60%, the sensor reading of the biofilter with a 1:1 volume ratio is higher than that with a 1:4 volume ratio. The disparity of the sensor readings increased with moisture content. A t-test was applied to determine if the particle size distribution caused a difference in moisture sensing. The sensor reading obtained from these two batches ranging from 40% to 60% were used as the input for the t-test. Table 4-2 presents the result of the paired t-test, which shows that there is significant difference caused by particle size distribution.

Table 4-2. Paired t-test of sensor reading regarding different particle size distribution.

Data: 1:1 and 4:1 volume ratio of 0.2 - 0.8 cm and 0.8 - 1.9 cm of woodchips
t = -17.292, df = 239, p-value < 2.2e-16
Alternative hypothesis: true difference in means is not equal to 0

One potential explanation is the contact area between woodchip particles and the sensing unit of the sensors. When the volume percentage of small particles decreased from 50% (1:1 volume ratio) to 20% (1:4 volume ratio), the small particles had fewer chances to contact with the sensor plates, which led to the decrease of total contact area, and resulted in higher impedance, thus lower sensor reading.

Another possible reason is the compaction effect of the media. Small particles have the ability to adsorb more water molecules than the larger particles on their surface, and they tend to settle due to gravitational force. The smaller particles the biofilter contained, the more compressed the biofilter media would be, which resulted in more water within the sensing unit of the sensor, and took over air space. Since the water has a lower impedance than air and woodchips, impedance of the biofilter media with many smaller particles would decrease, thus increasing sensor reading.

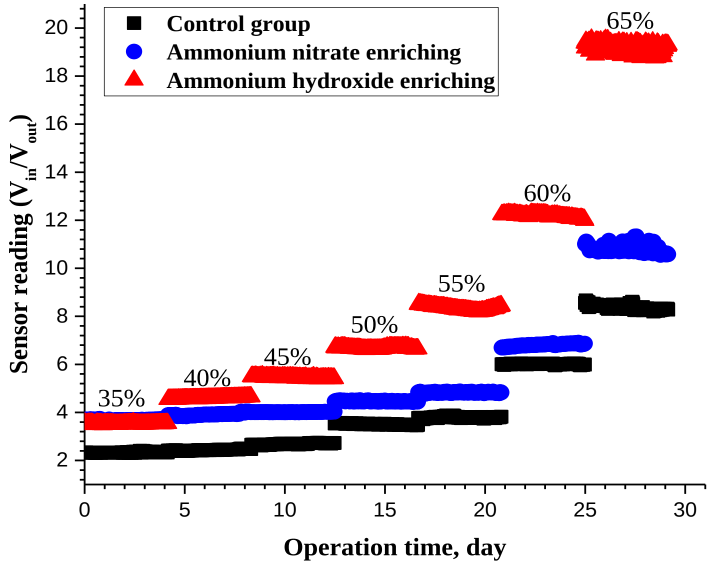
4.3 Sensor response to nitrogen concentration with changing moisture

4.3.1 Influence of nitrogen loading

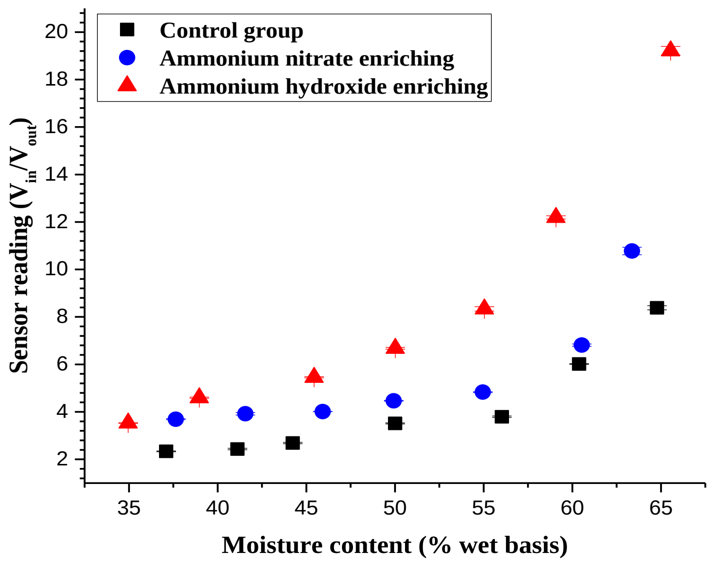
The sensor response regarding different ways of nitrogen enriching is shown in Figure 4-4. The sensor reading of the control group was in the range of 2.3~8.5 during the test, while the sensor readings of nitrogen loading batches were in the range of 3.6~10.8 for ammonia nitrate enriching and 3.5~19.3 for ammonia hydroxide enriching. The figure reflects the large differences that exist between the nitrogen loading batches and the control group. The most rapid increase of all three batches was the batch with ammonium hydroxide enriching. During the same period, there was a large increase of the sensor reading for this batch when the moisture content was higher than 50%. The disparities of sensor readings between the two groups increased along with the increase in moisture content. The contribution of moisture content to the increase of the sensor reading has been demonstrated by previous research (Yang et al., 2013). These results show that nitrogen loading can significantly affect the sensor reading.

To correlate the sensor reading with different forms of nitrogen, Figure 4-5 shows the concentration of N during the ammonium hydroxide enriching and ammonium nitrate enriching tests. With respect to the ammonium hydroxide enriching, the most impressive feature was that the ammonia-nitrogen concentration remained constant, which is also the dominant form of nitrogen within the biofilter media during the test. There was no significant difference in terms of the nitrate-nitrogen concentration between the control group and ammonium hydroxide enriching group. With respect to the ammonium nitrate enriching, both the concentrations of nitrate-nitrogen and ammonia-nitrogen are far higher than those in the control group. However, a very noticeable trend was that both forms of nitrogen decayed over time. Though similar concentrations were added at the beginning, the concentration of these two forms of nitrogen differed from each other during operation. This phenomenon can be explained by the nitrification process for nitrogen compounds (Yang et al., 2012).

Accordingly, the distinctions of sensor response may be explained by the introduction of the nitrogen, which changed the impedance of the biofilter media. The lower sensor reading of ammonium-nitrate enriching compared with ammonium-hydroxide enriching might be attributed to the introduction of nitrate-nitrogen, which increased the impedance of the biofilter media, resulting in the decrease of the sensor reading. The impedance of biofilter media was determined by the dielectric constant, the smaller the dielectric constant, the larger the impedance would be. In this study, the introduction of nitrate-nitrogen dissolved as nitrate ion, which decreased the dielectric constant of the media. This result is consistent with the study conducted by Lileev et al., (2003), since they found the values of dielectric constant of the solution containing nitrate ion decreased with the increase of salt concentration.



(a)



(b)

Figure 4-4. Sensor response to different ways of nitrogen enrichment: (a) real-time recording of sensor reading at each moisture step, and (b) mean and standard deviation of sensor readings at varied moisture contents.

Note: No nitrogen enriching for the control group, 0.75 mg nitrate-nitrogen per gram dry biofilter media of ammonium nitrate enriching, 0.75 mg ammonia-nitrogen associated 0.75 mg nitrate-nitrogen per gram dry biofilter media of ammonium hydroxide enriching, 0.2-0.8 cm: 0.8-1.9 cm = 1:4 of particle size distribution

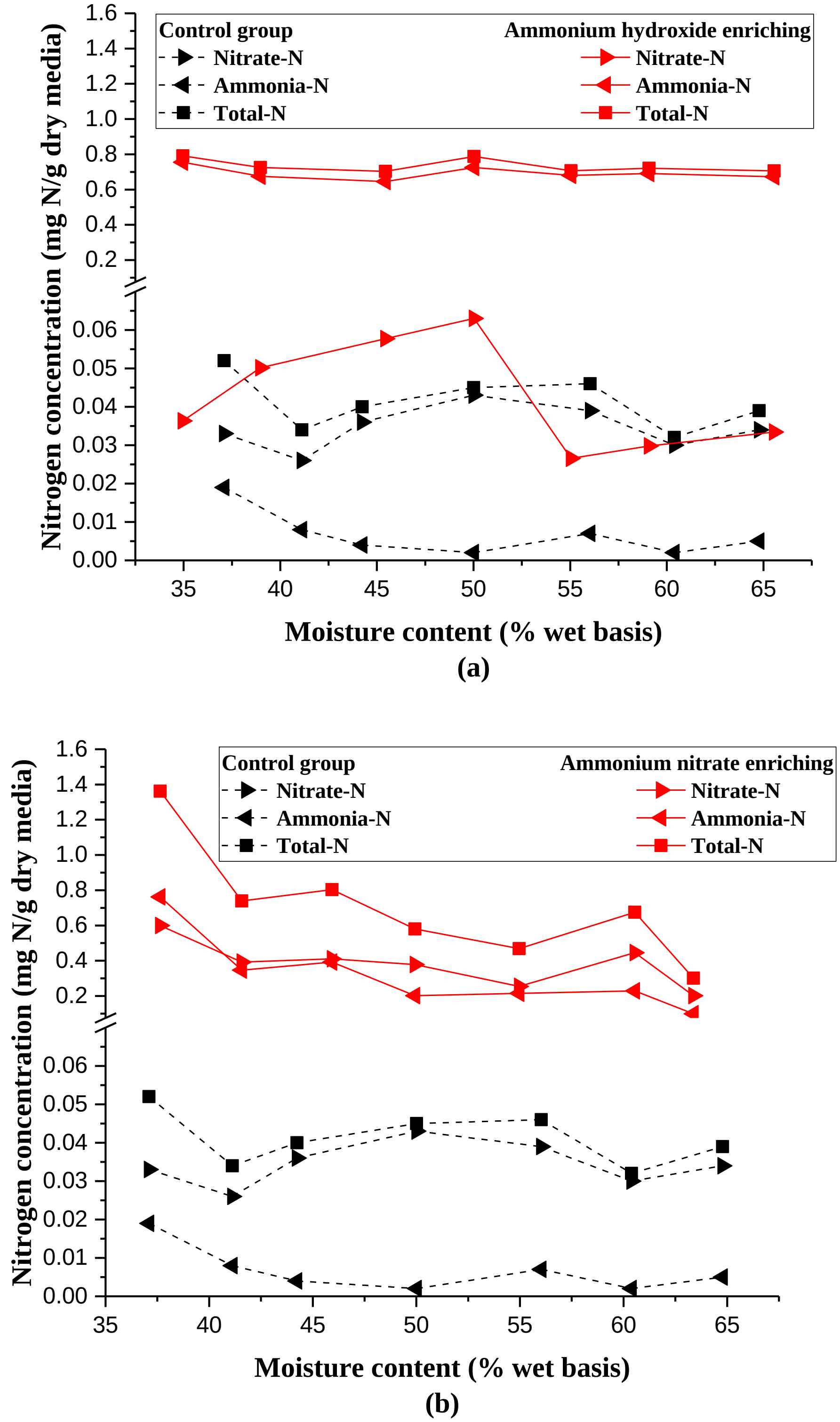


Figure 4-5. Profiles of nitrogen concentrations of different ways of nitrogen enrichment: (a) 0.75 mg ammonia-nitrogen per gram dry biofilter media of ammonium hydroxide enriching, and (b) 0.75 mg ammonia-nitrogen associated with 0.75 mg nitrate-nitrogen per gram.

4.3.2 Influence of ammonium-hydroxide and ammonium-nitrate

Figure 4-6 shows the sensor output for different batches as well as the profiles of different nitrogen along with the increasing moisture content treated by ammonium-hydroxide enriching. This figure shows a clear comparison between sensor readings (Figure 4-6a) as well as the concentration of ammonia-nitrogen and nitrate nitrogen (Figure 4-6b). It illustrates that ammonia-nitrogen was the dominant compound of nitrogen in all batches, and the concentration of ammonia-nitrogen remained stable. It can be assumed that the influence of nitrate-nitrogen is little in this test. According to the figure, a positive correlation between the sensor reading and the concentration of ammonia-nitrogen was found in this study. It can be seen from the chart that the higher ammonia-nitrogen concentration results in a higher sensor reading. The potential explanation for the trend of sensor output along with the ammonia-nitrogen concentration can be from the prospective of the impedance change. In biofilter, ammonia-nitrogen can be in forms of dissolved ammonia-nitrogen and free ammonia. The dissolved ammonia-nitrogen can be divided into two species: ionized ammonia (NH_4^+) and un-ionized ammonia ($NH_3{\cdot}H_2O$). Both the dominant component, free ammonia and un-ionized ammonia decreased the impedance of media and caused an increase in sensor reading. In conclusion, the introduction of ammonia-nitrogen decreased the impedance of the biofilter media, and lower impedance led to a higher sensor reading.

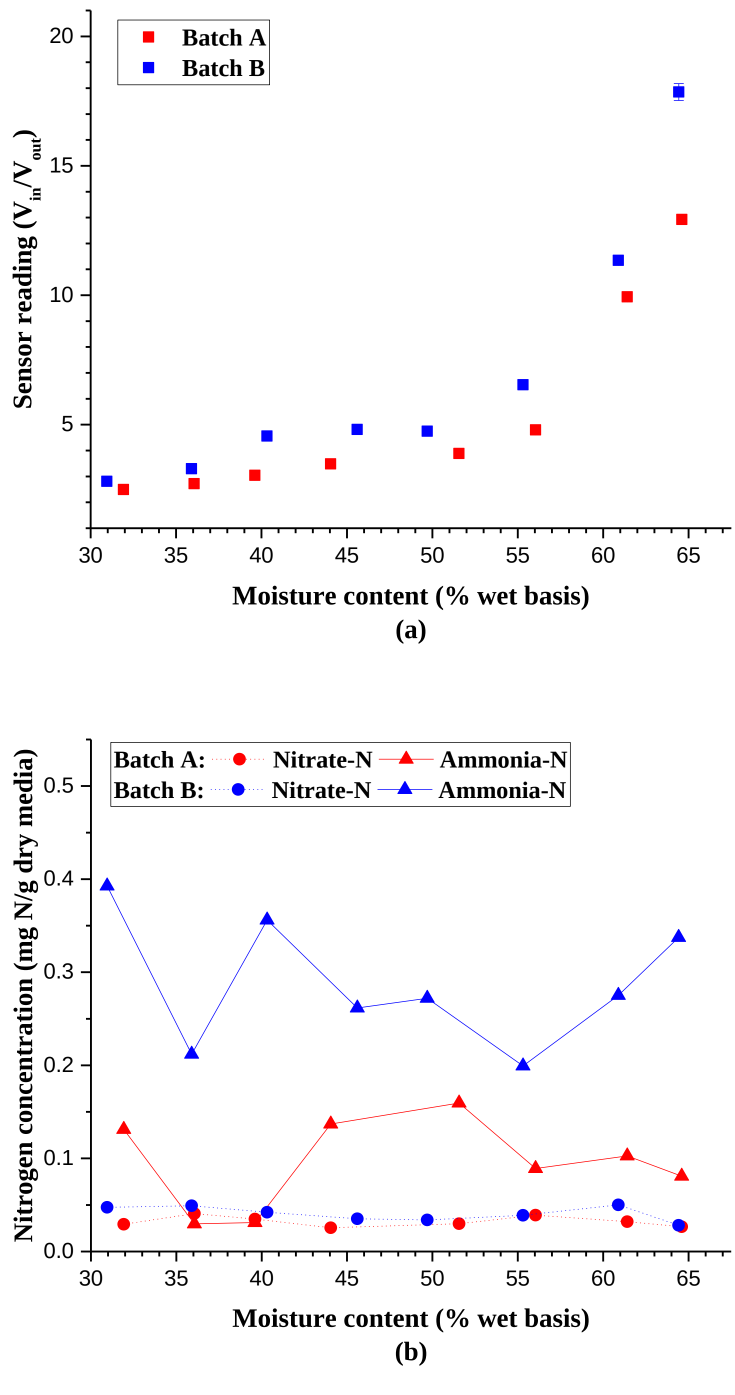
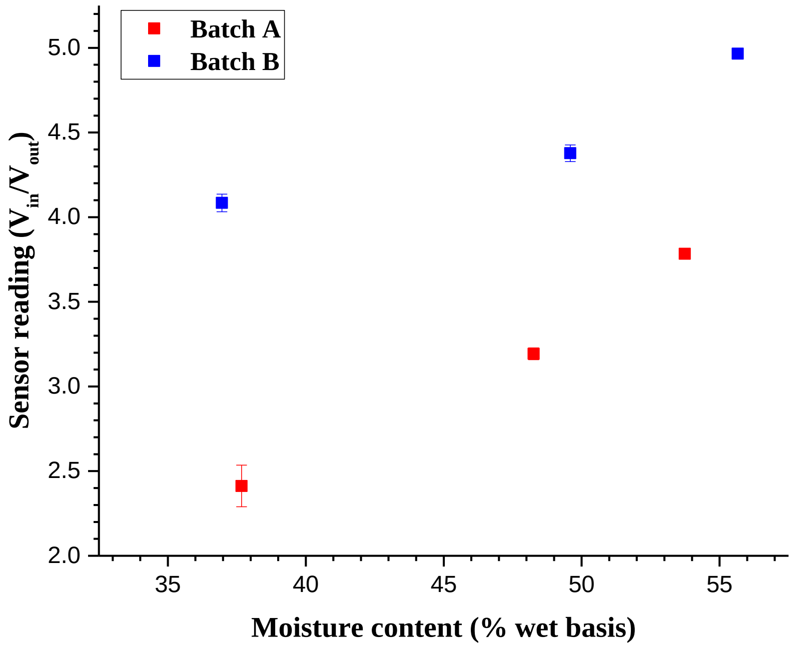


Figure 4-6. Results of ammonium-hydroxide enriching: (a) mean and standard deviation of sensor readings at varied moisture contents, and (b) nitrogen concentrations of biofilter media at varied moisture contents.

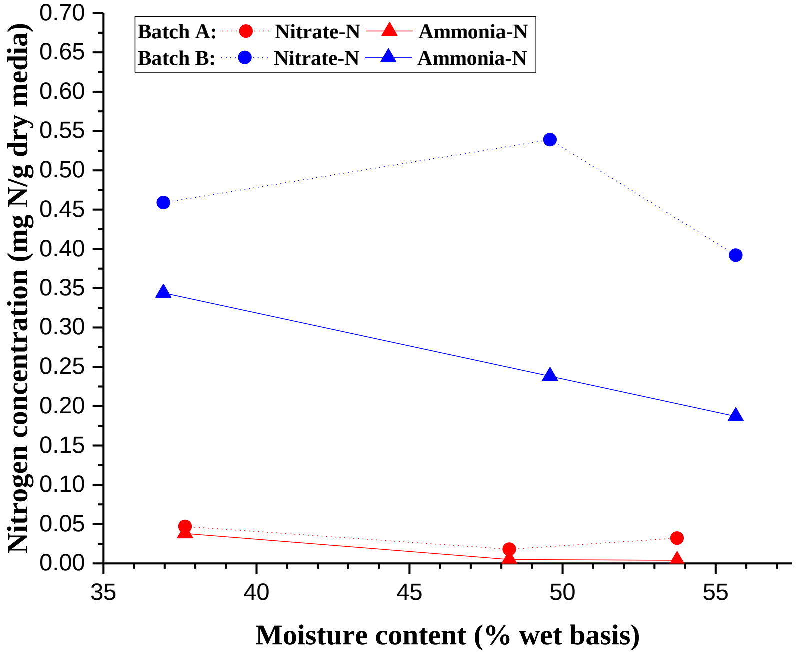
Note: Biofilter batches A and B were treated with 0.25 and 0.50 ammonia-nitrogen per gram dry biofilter media, respectively.

Figure 4-7 illustrates the sensor output of different biofilter batches and the profiles of different nitrogen treated by ammonium-nitrate enriching. Unlike the previous test, a slight adjustment was made in this test. The interval for moisture content between each operation was increased to diminish the influence of other variables (e.g., microbial activities, aging of the woodchips). The figure suggests a higher ammonium-nitrate concentration result in a higher sensor reading. It indicates that the impedance of the biofilter media was influenced by the combination effect of ammonia-nitrogen and nitrate-nitrogen. But at this point, it is not clear how to determine the individual contribution of ammonia-nitrogen and nitrate-nitrogen, which will be determined by subsequent statistical methods.

To consider the influence of high nitrogen concentration on the impedance of biofilter media, Figure 4-8 displays the sensor output and the profiles of different nitrogenous compounds in biofilter batches treated by ammonium-nitrate enriching at high concentrations. The sensor reading ranged from 4.6 to 28.6. The higher sensor reading found here compared with that in former studies could reflect the fact that the introduction of nitrogen at a high concentration have significant impact on the sensor reading. However, there is no great difference in sensor readings between batch A and batch B. This observation reinforces the importance of considering all the potential variables (moisture content, ammonia-nitrogen, and nitrate-nitrogen) for sensor reading. And there might be synergism between nitrogen concentration and moisture content.



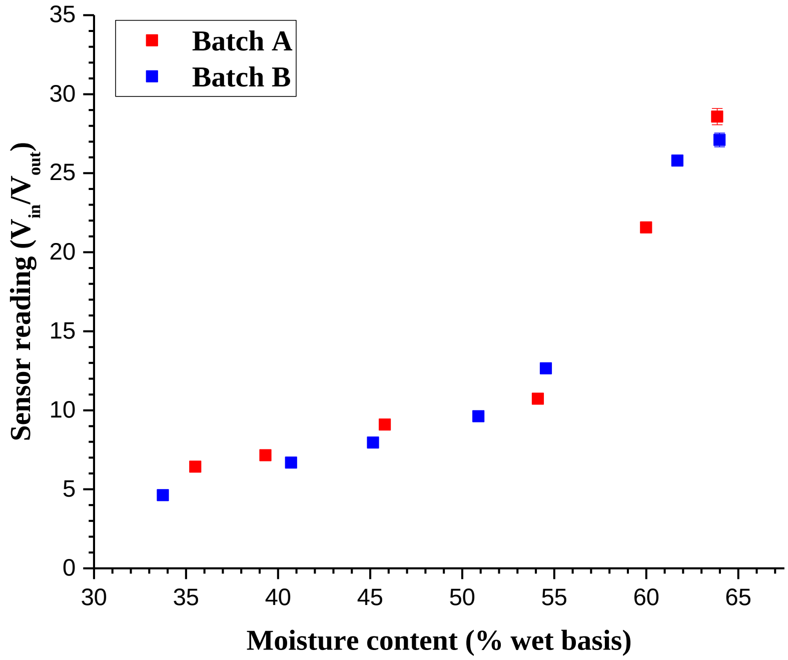
(a)



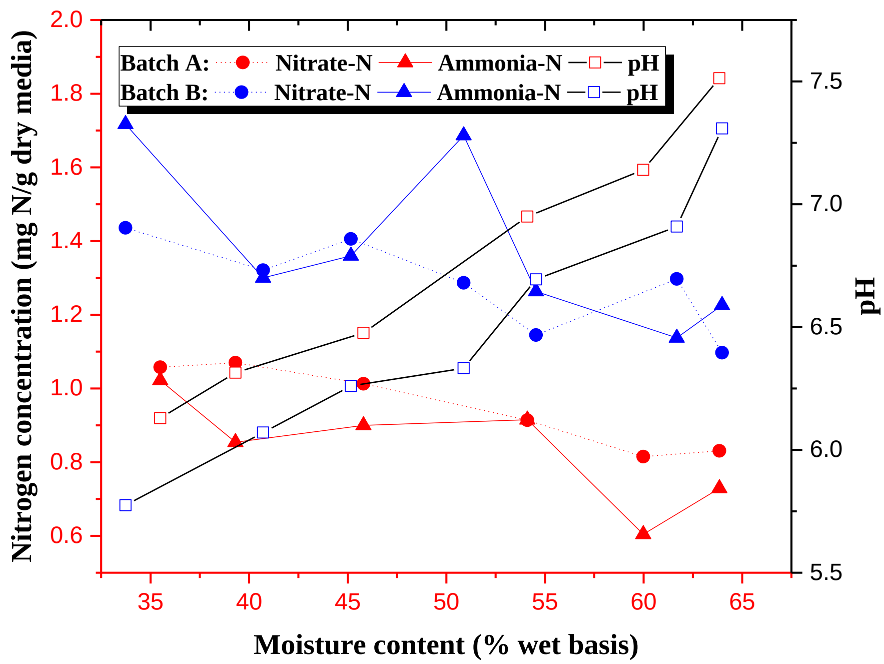
(b)

Figure 4-7. Results of ammonium-nitrate enriching: (a) mean and standard deviation of sensor readings at varied moisture contents, and (b) nitrogen concentrations of biofilter media at varied moisture contents.

Note: Biofilter batches A and B were treated with nitrate-nitrogen associated with the same ammonia-nitrogen at the concentration of 0.0625 and 0.5 mg per gram dry biofilter media, respectively).



(a)



(b)

Figure 4-8. Results of ammonium-nitrate enriching: (a) mean and standard deviation of sensor readings at varied moisture contents, and (b) nitrogen concentrations and pH of biofilter media at varied moisture contents.

Note: biofilter batches A, and B were treated with nitrate-nitrogen associated with the same ammonia-nitrogen at the concentration of 1.25, and 1.75 mg per gram dry biofilter media, respectively).

4.4 Mathematical model

The following mathematical model was established to demonstrate the relationship between sensor reading and moisture content, which is the precursor of the further statistical model.

4.4.1 Theoretical assumptions and validation

The following assumptions were made to simplify the modeling process.

1). Assume the fraction volume of nitrogen is negligible compared to water.

The unit of nitrogen concentration in this investigation was mg N per g dry media, and the powder dissolved in the water can be regarded as negligible.

2). Consider the impedance of biofilter media as a simple parallel connection of a resistor and a capacitor, the contribution of the resistor to the impedance is compared a little to the capacitor.

The validation was based on the results of experiments. The impedance of the reference capacitor $\left|Z_{ref}\right|$ *was*

$$\left|Z_{ref}\right| = \left|\frac{1}{j\omega C_{ref}}\right| = \left|\frac{1}{j2\pi f C_{ref}}\right| = \left|\frac{1}{2\pi \times 10^5 \, Hz \times 10^{-9} \, F}\right| = 1591.5\Omega \quad \textbf{(4.2)}$$

$$\left|Z_{35\%MC}\right| = \left|\frac{Z_{ref}}{reading - 1}\right| = \left|\frac{1591.5}{2-1}\right| = 1591.5\Omega \quad \textbf{(4.3)}$$

$$\left|Z_{55\%MC}\right| = \left|\frac{Z_{ref}}{reading - 1}\right| = \left|\frac{1591.5}{20-1}\right| = 83.7\Omega \quad \textbf{(4.4)}$$

The sensor readings ranged from 2 (35% MC) to 20 (55% MC), corresponding to the impedance of the biofilter media ranging from 1591.5 Ω to 83.7 Ω. While the resistance of the biofilter media was calculated based on the conductivity (Zelinka et al., 2008) and the equation (4.2).

$$R = \rho \frac{\ell}{A} \tag{4.5}$$

Where

R is the resistance, ℓ is the length of the conductor, A is the cross-sectional area of the conductor measured, and ρ is the electrical resistivity.

Based on calculation, the resistance of woodchips decreased with the increasing moisture content. However, for moisture content of 35%, the resistance was $10^4\,\Omega$; and for moisture content of 55%, the resistance was $10^3\,\Omega$. Both the value of resistances were larger than the impedance. Since the equivalent impedance of a parallel-connection circuit was determined by the contributor with the smaller value, the resistance can be negligible. It is appropriate to assume that:

$$Z_{biofilter} \approx \frac{1}{j\omega C_{biofilter}} \tag{4.6}$$

4.4.2 Mathematical formulation for correlation of sensor reading and moisture content

The following process attempts to build the relationship between sensor reading and moisture content. And this model only applies to moisture content of biofilter media from 35% to 65%.

$$reading = 1 + \frac{|Z_{ref}|}{|Z_{biofilter}|} = 1 + \frac{\left|\frac{1}{j\omega C_{ref}}\right|}{\left|\frac{1}{j\omega C_{biofilter}}\right|} = 1 + \left|\frac{C_{biofilter}}{C_{ref}}\right| \quad \textbf{(4.7)}$$

$$C_{biofilter} = \frac{\varepsilon_{biofilter} A}{d} = b\varepsilon_{biofilter} \quad \textbf{(4.8)}$$

$$reading = 1 + \left|\frac{C_{biofilter}}{C_{ref}}\right| = 1 + \left|\frac{b\varepsilon_{biofilter}}{C_{ref}}\right| = 1 + |c\varepsilon_{biofilter}| \quad \textbf{(4.9)}$$

Where,

C_{ref} : Capacitance of reference capacitor (constant)

$C_{biofilter}$: Capacitance of biofilter media in Farads,

ε : Dielectric constant (absolute, not relative)

A : Area of plate overlap (constant)

d : Distance between plates (constant)

$b = \frac{A}{d}$: Constant

$c = \frac{b}{C_{ref}}$: Constant

According to previous literature (Heimovaara et al., 1994),

$$\varepsilon_m^a = \Sigma \varepsilon_i^a v_i \quad \textbf{(4.10)}$$

Where,

ε_m is the dielectric constant of the medium, i represents each component (air, organic material, inorganic material, and water), v is the volume fraction of each component, and constant a is close to 0.5.

$$\varepsilon_{biofilter}^{0.5} = \varepsilon_{water}^{0.5} v_{water} + \varepsilon_{woodchips}^{0.5} v_{woodchips} + \varepsilon_{nitrogen}^{0.5} v_{nitrogen} + \varepsilon_{air}^{0.5} v_{air} \quad \textbf{(4.11)}$$

Based on the first assumption that the fraction volume of nitrogen is negligible compared to water, the terms regarding water and nitrogen can be combined as a term with the subscript "solution". Besides, the dielectric constant of air is 1, which is smaller than other components, the term regarding air can be omitted.

$$\varepsilon_{biofilter}^{0.5} = \varepsilon_{solution}^{0.5} v_{solution} + \varepsilon_{woodchips}^{0.5} v_{woodchips} \quad \textbf{(4.12)}$$

Rewrite the above equation, it can be converted as

$$\varepsilon_{biofilter} = \varepsilon_{solution} v_{solution}^2 + \varepsilon_{solution}^{0.5} \varepsilon_{woodchips}^{0.5} v_{woodchips} v_{solution} + \varepsilon_{woodchips} v_{woodchips}^2 \quad \textbf{(4.13)}$$

$$\varepsilon_{biofilter} = e v_{solution}^2 + f v_{solution} + g \quad \textbf{(4.14)}$$

Where,

e, f, g are constants.

Since

$$MC = \frac{m_{water}}{m_{total}} \quad \textbf{(4.15)}$$

$$v_{solution} = \frac{\frac{m_{solution}}{\rho_{solution}}}{\frac{m_{total}}{\rho_{total}}} = \frac{\frac{m_{water}}{\rho_{water}}}{\frac{m_{total}}{\rho_{total}}} = hMC\rho_{total} \quad \textbf{(4.16)}$$

Where,

ρ_{total} is the density of the biofilter media, which is a linear function of moisture content when the moisture content ranges from 35% to 65% (Simpson, 1993).

$$\rho_{total} = kMC \quad \textbf{(4.17)}$$

$$reading = 1 + \left| c\varepsilon_{biofilter} \right| = 1 + \left| cev_{solution}^2 + cfv_{solution} + cg \right| \quad \textbf{(4.18)}$$

$$reading = 1 + \left| ce(hkMC^2)^2 + cf(hkMC^2) + cg \right| = lMC^4 + mMC^2 + n + \varepsilon \quad \textbf{(4.19)}$$

Where,

h, k, l, m, and n are constants, ε refers to potential factors other than moisture content of the biofilter media.

Then the sensor reading can be expressed as a function of moisture content. This is the first demonstration of the relationship between sensor reading and moisture content. And it is a prerequisite to develop the statistical model.

4.5 Statistical and predictive model

4.5.1 Statistical model

The relationship between sensor reading and moisture content has been built, but the extent of the influence of different nitrogen as well as the concentration was far from uncertain. Limited research mentioned the correlation between the nitrogen concentration and impedance of the biofilter media.

With the measured data, a multiple linear regression was tried to determine the relationships among sensor reading, moisture content and different forms of nitrogen as well as their concentration. Two approaches were tried to explore the influence of ammonia-nitrogen and nitrate-nitrogen on sensor reading.

Approach 1:

The independent variables were ammonia-nitrogen concentration (ANC), nitrate-nitrogen concentration (NNC), the square of the moisture content (MC^2), and fourth order of the moisture content (MC^4), the dependent variable for the statistical was sensor reading (SR).

The relational expression for this statistical approach can be

$$SR = aANC + bNNC + cMC^2 + dMC^4 + e \qquad \textbf{(4.20)}$$

Where,

a, b, c, d, e are constants.

The rationale for this approach was to identify the respective influences of ammonia-nitrogen and nitrate-nitrogen. The coefficients of the fourth-degree polynomial function and the statistical significance of coefficients are shown in Table 4-3.

Table 4-3. Coefficients of polynomial and their statistics significance (approach 1).

Parameter	Estimate	Std. Error	t value	Pr (>|t|)	
e	3.398e+00	8.348e-01	4.070	0.000137	***
a	4.737e+00	7.401e-01	6.400	2.46e-08	***
b	-2.758e+00	1.203e+00	-2.293	0.025325	*
c	-2.867e-03	7.884e-04	-3.636	0.000570	***
d	1.196e-06	1.621e-07	7.380	5.15e-10	***

Multiple R-squared: 0.873

The coefficients show that both the increase of the ammonia-nitrogen and moisture content significantly results in a higher sensor reading, while the loading of nitrate-nitrogen decreases the sensor reading slightly. Based on the above function, the predicted and the observed sensor readings are plotted in Figure 4-9. For sensor readings less than 13, the predicted sensor reading agreed with the observed sensor reading, which indicated that this fourth-degree polynomial function was accurate and can represent the relationships among sensor reading, moisture content and different forms of nitrogen as well as their concentrations.

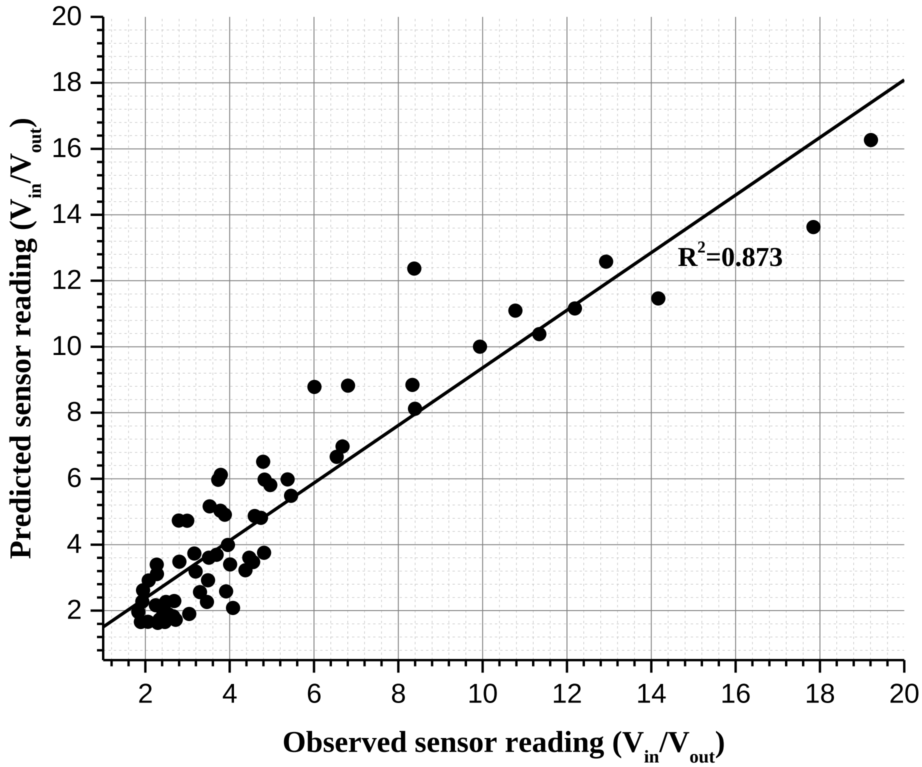


Figure 4-9. Comparison between predicted and observed sensor reading (approach 1).

Approach 2:

The independent variables are the sum of ammonia-nitrogen concentration and nitrate-nitrogen concentration (TNC), the ratio of nitrate-nitrogen to ammonia-nitrogen (RNC), the square of the moisture content (MC^2), and the fourth order of the moisture content (MC^4), the dependent variable for the statistical was sensor reading (SR).

The relational expression for this statistical approach can be

$$SR = aTNC + bRNC + cMC^2 + dMC^4 + e \qquad \textbf{(4.21)}$$

Where,

a, b, c, d, e are constants.

The rationale for this approach was to identify the influence of total nitrogen concentration as well as the dominant nitrogen form. The coefficients of the fourth-degree polynomial function and the statistical significance of coefficients are shown in Table 4-4.

Table 4-4. Coefficients of polynomial and their statistics significance (approach 2).

Parameter	Estimate	Std. Error	t value	Pr (>|t|)	
e	4.091e+00	9.314e-01	4.393	4.54e-05	***
a	1.692e+00	6.476e-01	2.612	0.011307	*
b	-6.431e-02	3.799e-02	-1.693	0.095551	.
c	-3.131e-03	9.044e-04	-3.462	0.000986	***
d	1.254e-06	1.852e-07	6.771	5.73e-09	***

Multiple R-squared: 0.837

The coefficients show that both the increase in moisture content and the total nitrogen concentration significantly result in a higher sensor reading, but the ratio of different nitrogen forms did not have any significant impact on the sensor reading. Figure 4-10 shows the predicted sensor reading and observed sensor reading based on the above regression.

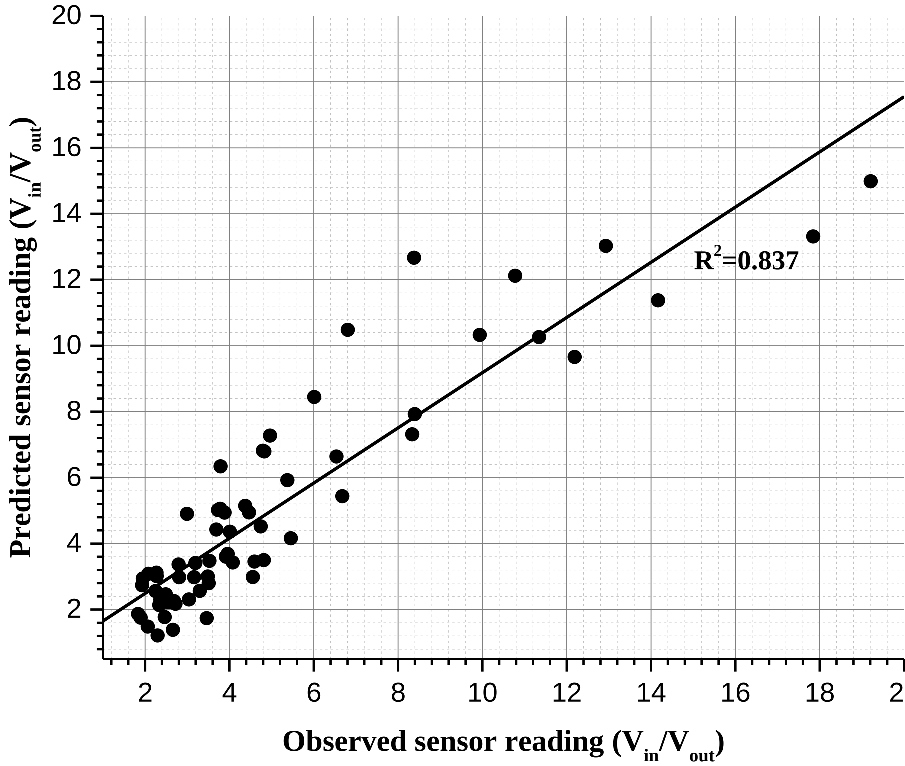


Figure 4-10. Comparison between predicted and observed sensor reading (approach 2).

The above results of statistical regression suggest that all the sensor readings in this investigation were influenced by moisture content and different forms of nitrogen. Approach 1, which correlated sensor reading with moisture content, ammonia-nitrogen concentration and nitrate-nitrogen concentration with high multiple R-squared (R^2 = 0.873), is best one to use to calibrate the sensor performance.

4.5.2 Predictive model

A predictive model based on the above statistical model was built to compute moisture content. Taking approach 1 as an example, the relational expression for this statistical approach is:

$$dMC^4 + cMC^2 + aANC + bNNC + e - SR = 0 \qquad \textbf{(4.22)}$$

To solve the moisture content (MC) of the biofilter media, the moisture content can be expressed as:

$$MC = \sqrt{\frac{-c \pm \sqrt{c^2 - 4d(aANC + bNNC + e - SR)}}{2d}} \qquad \textbf{(4.23)}$$

There will be one or two roots derived by the above equation, the suitable root of these two should be based on the anticipated moisture content and practical operation. Since all the above constants and all the input variables are known, the moisture content of the biofilter media can be easily derived with a pre-programmed user interface (UI) or software package.

Chapter 5 CONCLUSIONS AND FUTURE RESEARCH

5.1 Conclusions

The major conclusions from this experimental study are summarized as follows:

(1) Particle size distribution of the biofilter media has a direct effect on impedance-based moisture sensing. The change of impedance of the biofilter media can be attributed to 1) the contact area between the biofilter media and sensing unit, and 2) uneven moisture content distribution due to compaction. This means the sensor may require re-calibration based on the particle size distribution of the biofilter media caused by the compaction effect.

(2) The concentration of different forms of nitrogen (ammonia-nitrogen, nitrate-nitrogen) have a significant impact on the impedance of the biofilter media. The impedance of the biofilter media is negatively related to increased ammonia-nitrogen concentration but positively related to increased nitrate-nitrogen concentration.

(3) A mathematical model was established to couple the sensor reading with the moisture content. A statistical model verified that the moisture content, ammonia-nitrogen and nitrate-nitrogen determine the sensor reading of the impedance-based moisture sensor with acceptable predictive power (R^2 = 0.873). A predictive model was built for moisture content of the biofilter media based on a statistical model for future use.

5.2 Practical use of the results

These studies suggest that a feasible system is possible to monitor the moisture content from 35% to 65% for biofilters for agricultural operations. The nitrogen-concentration can be easily measured by commercial nitrogen sensors as well as regular commercial water quality services, thus could be used as an input of this moisture measurement system as well as an indicator for biofilter management. This study represents the first time that different methods of nitrogen enrichment in biofilters were considered in moisture content measurement. These results could assist in improving the performance of a biofilter operation and biofilter management, since the impedance-based moisture content sensor can be programmed based on the model. However, this methodology is only specific to woodchips-based gas-phase biofilters.

5.3 Future research

Further studies are foreseen to investigate the performance of sensors associated with microbial activities. Temperature and moisture content regimes on microbial activity, which provides a potential to increase the impedance among sensing units, and thus interfere with the sensor reading. Tests need to eliminate or calibrate interference from microbes.

Efforts should also be invested in improving the robust design of sensor, e.g., the circuit part. Sensors that have stable integrated circuits and are low-cost are preferred to work on a large scale in agricultural applications.

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