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CO_2 EMISSION AND O_2 UPTAKE OF SOIL UNDER DIFFERENT SYSTEMS

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TÍTULO DA TESE: CO_2 EMISSION AND O_2 UPTAKE OF SOIL UNDER DIFFERENT SYSTEMS

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AUTHOR'S CURRICULUM DATA

RISELY FERRAZ DE ALMEIDA – Daughter of Rhonda Graça Ferraz and Valternor Ferreira de Almeida, was born on September 29, 1986, in Vitória da Conquista, Bahia state, Brazil. She earned her Bachelor of Science Degree in Agronomy in February, 2012 at Universidade Estadual do Sudoeste da Bahia (UESB), Vitória da Conquista campus. At that same time, she attended the Instituto Federal de Tecnologia da Bahia (IFBA) where she got her technical degree in Environment Science. On March 2012, she entered the M.Sc. Program at the Universidade Federal de Uberlândia (UFU), Uberlândia campus, and received her M.Sc. Degree in Agronomy (Soil Science) in 2014. In March, 2014, she joined to the Graduate Program in Agronomy (Soil Science) at the São Paulo State University (UNESP/FCAV), Jaboticabal campus to get her doctorate degree. For that, she developed two soil projects at: the São Paulo State University (UNESP), Ilha Solteira campus; and University of Minnesota, St Paul campus - USA. These projects have been part of her doctoral thesis. Then she submitted the doctoral thesis to an examination panel, and received her Ph.D. Degree in Agronomy (Soil Science) from UNESP/FCAV on February 2017. As results of her background and expertise is associated with environmental and agricultural sciences, especially on the following research topics: sugarcane management, climate change, soil CO_2 emission, soil carbon, biochar, soil use and management.

I DEDICATE

To God for blessing me all the time.

To my beloved mother, Rhonda Graça Ferraz,
my greatest example of being a better person. This is for you!

I OFFER

To my family for being the base of my life

To my friends for being the family that I chose

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SUMMARY

CO_2 EMISSION AND O_2 UPTAKE OF SOIL UNDER DIFFERENT SYSTEMS

ABSTRACT - The soil O_2 and CO_2 concentration are the two most important gases related to soil microorganisms. Thus, this thesis was developed to observe the concentration and relationship between carbon dioxide (CO_2) and oxygen (O_2) under different residue systems. For that, we run two soil experiments in Brazil and the USA, respectively. The first experiment was developed to examine the relationship between CO2 and O2 using soil moisture and O2 as a soil respiration predictor in a sugarcane area under different managements of residues (mechanical harvesting - GH versus straw burning - BH). Therefore, the first experimental results are described in the Chapter 2 and entitled "Use of O_2 uptake as an index of CO_2 respiration in sugarcane areas under different managements". We run the second soil experiment measuring biochar's impact on CO_2 production or sorption and O_2 uptake in amended soils. Thus, we studied three soil types (Rosemount - RM; Potting soil Sunshine - PS; and UM) and five different biochars (Pine chip biochar - ICM; Royal Oak hardwood lump charcoal - RO; Accurel activated charcoal - AAC; Bamboo - B; and Macadamia nut - MC) and control treatment (Soil without biochar). Consequently, the results are described in the Chapter 3 and entitled "How O_2 uptake can help us understand the CO_2 sorption processes by biochar?". Thus, we can conclude with our results that the concentration and relationship between FCO_2 and FO_2 depend on different systems and soil conditions, for example: soil crop residue managements, soil moisture and use of biochar. The FO_2 is positively correlated with FCO_2 at biological condition with respiratory quotient (RQ) values close to 1.0. Moreover, we can observe that RQ values higher than 1 are results of soil–gas exchange fluxes after precipitation or higher available on O_2. Thus, the FO_2 can be used as an index for categorizing the source of FCO_2 respiration. To finish, we can observe that the biochar can be used to sequester CO_2 from the atmosphere by the absence of biological activities in a short period of time. However, we believe that more study should be developed to elucidate the CO_2 and O_2 sorption by biochars and their reactions (biological and/or chemical) when added biochar in soil.

Keywords: Biochar, Soil crop residue, Biological respiration, Respiratory quotient

EMISSÃO DE CO_2 E CAPTURA DE O_2 DO SOLO EM DIFERENTES SISTEMAS

RESUMO - O oxigênio (O2) e o dióxido de carbono (CO2) no solo são os dois principais gases relacionados com a atividade dos microorganismos no solo. Assim, esta tese foi desenvolvida para observar a concentração e a relação entre a concentração do CO2 e O2 sob diferentes sistemas de resíduos. Para isso, realizamos dois experimentos de solo no Brasil e nos EUA, respectivamente. O primeiro experimento foi desenvolvido para examinar a relação entre fluxo de CO2 (FCO2) e o fluxo de O2 (FO2) usando a umidade do solo e o O2 como um predictor da respiração do solo em uma área de cana-de-açúcar sob diferentes manejos de resíduos (colheita mecânica - GH versus colheita queimada – BH). Portanto, os resultados do primeiro experimento estão descritos no Capítulo 2 e sendo intitulado de "Uso da captura de O2 como índice de respiração de CO2 em áreas de cana-de-açúcar sob diferentes manejos". O segundo experimento do solo observou o impacto do biochar na emissão ou sorção de CO2 e O2 nos solos. Assim, foram estudados três tipos de solos (Rosemount - RM, Potting Sol Sunshine - PS e UM), cinco biochars diferentes (biochar de chip de pinho - ICM, biochar de Carvalho Oak Royal - RO, biochar Acurel ativado - AAC, biochar de Bambu - B; biochar de Macadâmia - MC) e o tratamento controle (solo sem biochar). Consequentemente, os resultados foram descritos no Capítulo 3 e intitulado "Como a captura de O2 pode nos ajudar a entender os processos de sorção de CO2 via biochar?". Assim, nós podemos concluir com os nossos resultados que a concentração e relação entre FCO2 e FO2 dependem dos diferentes sistemas e condições dos solos estudados, tais como: manejo de resíduos de culturas do solo, umidade do solo e uso de biochar. O FO2 está positivamente correlacionado com o FCO2 via atividade biológica e com valores de coeficientes respiratório (RQ) próximos de 1,0. Além disso, podemos observar que valores de RQ maiores que 1 são resultados dos fluxos de troca solo-gás após precipitação ou maior disponibilidade de O2 no meio. Assim, o FO2 pode ser utilizado como um índice para categorizar uma fonte de respiração de CO2. Para concluir, o biochar pode ser utilizado para sequestrar CO2 da atmosfera em curto período de tempo. No entanto, acreditamos que mais estudos devem ser desenvolvidos para elucidar a sorção de CO2 e O2 pelo biochar e suas reações (biológicas e/ou químicas) quando adicionado biochar no solo.

Palavras-chave: Biochar, Resíduo da cultura no solo, Respiração biológica, Quoeficiente respiratório

1. CHAPTER 1 – General considerations

1.1 Introduction and Justification

The soil air constituents as well as soil water content are important aspects controlling biological activities (plant and microorganism respiration) (MOREIRA; SIQUEIRA, 2006; LEPSCH, 2011; BRADY; WEIL, 2013). The soil air contains mainly nitrogen (N_2), oxygen (O_2), carbon dioxide (CO_2) and water vapor, and they are the most important gases in soil (GLINSKI; STEPNIEWSKI, 1985). Normally, the CO_2 concentration in soil is higher than in the atmosphere, while O_2 concentration is lower (LEPSCH, 2011; MARSCHNER, 2012).

The soil O_2 and CO_2 concentration are the two most important gases related to soil microorganisms, capture, and soils and root respiration, respectively, (GLINSKI; STEPNIEWSKI, 1985). Stepniewski et al. (2005) have mentioned the importance of a better understanding of the oxygen cycle, and they have called this cycle, that describes the stock, absorption, movement, functions and determination of O_2 concentration in the environment.

The CO_2 cycle also has been mentioned by other researchers, such as: La Scala Júnior et al. (2000), Xu and Qi (2001), Epron et al. (2006), Panosso et al (2009), Corrade et al. (2013), Almeida et al. (2014), Moitinho et al. (2014) and Almeida et al. (2015), who have explained the relationships among the CO_2 and soil attributes and characteristic under different systems, uses and managements.

Therefore, this thesis was developed with the hypotheses that the CO_2 emission has a close relationship with O_2 under residues systems. Moreover, to understand and clarify the concentration and relationship of O_2 and O_2 with RQ.

Thus, this thesis has the objectives of: (i) examine the relationship between FCO_2 and FO_2 using soil moisture, RQ and O_2 as a soil respiration predictor in a sugarcane area under different managements (mechanical harvesting versus straw burning) (Chapter 2); and (ii) measuring biochar's impact on CO_2 production or sorption and O_2 uptake in amended soils (Chapter 3).

1.2 Sugarcane production in Brazil

Brazil is currently the largest sugarcane producer and has an average stalks yield at 684.7 millions of tons in 2016/17 (CONAB, 2016) and an estimated 20 Mg ha^{-1} of residues on the soil surface after harvest (URQUIAGA et al., 1991; OLIVEIRA et al., 1999).

The management of sugarcane with mechanized harvesting without burning or straw removal from the soil contributes to a more sustainable management option for sugarcane production (FIGUEIREDO; LA SCALA, 2011). However, sugarcane harvesting continues to be undertaken using two distinct practices in Brazil: mechanical harvesting with straw burning (BH) and without burning (GH) (PANOSSO et al., 2011).

Some researchers, such as Panosso et al. (2011), Bicalho et al. (2014) and Moitinho et al. (2015) have been studying sugarcane area and looking at the relationship of soil attributes to CO_2 emission. They have found a higher relation of soil CO_2 emission with temperature and precipitation (PANOSSO et al., 2011), and soil physical attributes, such as: soil porosity (PANOSSO et al., 2011; PANOSSO et al., 2012; BICALHO et al., 2014; MOITINHO et al., 2015), texture (SIGNOR et al., 2014) soil bulk density (CHAVES; FARIAS, 2008; SIGNOR et al., 2014), soil moisture (SILVA et al., 2014; IAMAGUTI et al., 2015), and others, like soil mineralogy (LA SCALA et al., 2000).

Moreover, some soil chemical attributes under sugarcane also have been cited, such as: pH value (SIGNOR et al., 2014), cation exchange capacity (LA SCALA JÚNIOR et al., 2000), and available nutrients (SIGNOR et al., 2014). The organic components, the soil carbon stock (MENDONZA et al., 2000; CHAVES; FARIAS, 2008), and ratio of carbon and nitrogen (SIGNOR et al., 2014) and microbial activity (MENDONZA et al., 2000), also are important.

1.3 Soil Attributes related to CO_2 emission

The CO_2 is included in the global carbon cycle, and that cycle occurs in soil, plant and atmosphere where the CO_2 emission is an important component (RAICH; SCHLESINGER, 1992). The soil CO_2 emission is a result of root respiration and microorganism respiration via a biological process (MELILLO et al., 2002; LAL, 2009).

On the other hand, the soil CO_2 emission also is a result of soil chemical process (MARQUES et al., 2000; DELBEM, 2011 and ANGERT et al., 2015). According to ANGERT et al. (2015) the use of calcareous materials in the soil is an example of a chemical process that improves CO_2 emission. Furthermore, the use of nitrogenous fertilizer through the urea reaction another example (MARQUES et al., 2000; DELBEM, 2011). Therefore, the CO_2 emission is a result of biological and chemical reactions in soils. In addition, the climate and the physical, chemical and

biological soil attributes, as well as soil organic matter, are responsible for increasing or decreasing the CO_2 emissions (LAL, 2009).

The main climatic variables that directly influence the CO_2 emission from the soil into the atmosphere are temperature (soil and atmosphere) and precipitation (humidity) (Duiker; Lal, 2000; ALMEIDA et al., 2009). In fact, this occurs because these variables can help improve the soil microbiology, providing adequate conditions for soil organic matter degradation (DUIKER; LAL, 2000; SILVA-OLAYA et al., 2013)

The temperature is considered the most important factor for microbial activity (BENJAMIN et al., 2003; KYAW THA PAW et al., 2006; and LAL, 2009; CHEN et al., 2011). According to Fang and Moncrieff (2001) and Stanford et al. (1973) the temperature can improve the microbial activity with the exponential increase of soil respiration and consequently the CO_2 emission occurring at temperature between 5 and 35°C.

Among the soil physical attributes we can describe some soil properties and characteristic, such as soil texture (CAMPOS et al., 1999; DILUSTRO et al., 2005), soil porosity (CHEN et al., 2010; PANOSSO et al., 2011), bulk density (XU; QI, 2001), moisture (GARDINI et al, 1991; HOWARD; HOWARD, 1993; CHEN et al. 2011) and the soil mineralogy (LA SCALA et al., 2000; XU; QI, 2001; EPRON et al., 2006), that have the same ability to control the CO_2 emission from soils.

The soil porosity has been cited as the most significant soil physical attribute that influences the CO_2 diffusion into soil (EHLERS et al., 1969), and its relationships have been observed by Xu and Qi, (2001), Ranjard and Richaume (2001) Epron et al. (2006), Panosso et al. (2012), Bicalho et al. (2014) and Moitinho et al (2015). According to Ehlers et al. (1969) and Fang et al. (1998) the relationship is stronger because the CO_2 diffusion (movement) is lower in soils with a low amount of pores, mainly in micropores. Moreover, the soil pores are a natural habitat for microbial communities (RANJARD; RICHAUME, 2001).

The soil moisture is another important physical attribute related to CO_2 emission. Some researchers such as Gardini et al. (1991) and Howard and Howard (1993) consider the soil moisture the key abiotic factor that affects the CO_2 emission process. According to Chen et al. (2011) soils with higher soil moisture and air availability can promote up to an 80% increase in CO_2 emission. This occurs because the soil moisture can improve microorganism and root activity (LAL; KIMBLE, 1995; SMITH et al., 2003), and gas diffusion through the soil pores (HILLEL, 1998; IGNATIUS, 1999; SMITH et al., 2003; COSTA et al., 2008). However, conditions of

higher soil moisture values (>50%) happens increase of denitrification by anaerobic microorganisms and production of N_2O (nitrous oxide) (DALAL et al., 2003).

The more significant soil chemical attributes we can describe are the pH value (RETH et al., 2005; FUENTES et al., 2006), cation exchange capacity (LA SCALA JÚNIOR et al., 2000), and the nutrients available, such as phosphorus (DUAH-YENTUMI et al., 1998), magnesium (XU; QI, 2001), nitrogen (ALMEIDA, 2014). The organic components are also important, such as the carbon stock (LONGDOZ et al., 2000; AMADO et al., 2001; SÁ et al., 2001; ALMEIDA et al., 2015) and carbon and nitrogen ratio (TIAN et al, 1997; COSTA et al., 2008).

We can consider the soil organic matter, microorganism activity and root distribution and density as biological soil attributes. According to Longdoz et al. (2000) and Panosso et al. (2011) the biological soil attributes have short and high relationship with the CO_2 produced from the soil. Almeida et al (2015) have been observing that soil label carbon has the highest correlation with CO_2 because it is easily decomposed by soil microorganisms.

1.4 Soil Attributes related to O_2 uptake

The air in the soil has a function of suppling O_2 for a soil organism (micro fauna and aerobic microorganisms), and plant respiration (photosynthesis and chemosynthesis) (MALAVOLTA, 2006; CHEN et al., 2011). During plant respiration, the O_2 is used for chemosynthesis (VAN DOVER, 2000; SMITH, 2002) as the primary electron acceptor in aerobic conditions, whereas under anaerobic conditions the nitrate, carbon dioxide, sulphur and sulphate can be the primary electron acceptor (SMITH, 2002). In photorespiration process the O_2 is used as a substrate by rubisco activity (carboxylation enzyme) in C3 plants in oxygenase (O_2 absorption to CO_2 production in photorespiration) and carboxylase (CO_2 absorption at photosynthesis) (NAGANUMA, 1998; MARENCO et al., 2014).

The O_2 available in the soil depends on its fast and continuous exchange between the soil and the atmosphere (CHEN et al., 2011). According to soil available O_2 we can classify the soil as normoxic, hypoxia and anoxia (DREW, 1997; CHEN et al., 2011). The normoxic is when the soil has sufficient O_2 availability for root and microorganism activity. Hypoxia is considered the point between normoxic and anoxia, and anoxia is the total absence of soil O_2 (DREW, 1997). According to Chen et al. (2011) in hypoxia it is possible to observe the low exchange of gases between

the soil and the atmosphere, and it occurs with a decrease of root growth and microorganism activity.

There are some soil chemical, physical and biological attributes that can be used as available O_2 diagnostics in soil. For instance, soil color, water-filled pore space, air permeability, redox potential, available iron and enzymatic activities (GLINSKI; STEPNIEWSKI, 1985). Therefore, the soil conditions can be an indicator of O_2 availability, and it has become very important to understand the available soil O_2 conditions (BRADY; WEIL, 2013). Some researchers have been working to better understand the O_2 flux behavior and the relationship between the O_2 and soil attributes, researchers such as: Berry and Norris (1949), Currie (1965), Cary and Holder (1982), Cook (1995), Armstrong and Drew (2002), Cook and Knight (2003) Cook et al. (2007) and Cheng et al. (2012).

Some physical soil attributes have been described as having a close relationship with available O_2, such as: soil porosity (QUASTEL, 1965; HILLEL, 1998; BRADY; WEIL, 2013), macroporosity and microporosity (SIERRA et al., 1995, SCANLON et. al., 2002; BRADY; WEIL, 2013), bulk density (KYAW THA PAW et al., 2006) and soil aggregation (OUYANG; BOERSMA, 1992; SIERRA et al., 1995; KYAW THA PAW et al., 2006). According to Glinski and Stepniewski (1985) the soil porosity is the simplest and probably the oldest soil attribute that can be used as an available O_2 indicator.

The water filled pores have been cited as an important attribute to understand the soil O_2 available (DILLY 2001; COOK et al. 2007; ELBERLING et al., 2011; BRADY; WEIL, 2013) because both O_2 and water occupy soil pores (QUASTEL, 1965). In addition, O_2 has a low solubility in water (HILLEL, 1998), and the O_2 diffusion coefficient decreases when the soils become saturated with water (SCANLON et al., 2002). The soil microbiological activity is also other important soil attribute to help understand the available soil O_2 (BERRY; NORRIS, 1949; GLINSKI; STEPNIEWSKI, 1985; BRADY; WEIL, 2013). The activity takes place because the O_2 is consumed by soil organism and plant root respiration (BRADY; WEIL, 2013). Consequently, higher plant root concentrations and biological activity trigger higher O_2 consumption. This has been observed in soil layers close to soil surfaces (KYAW THA PAW et al., 2006).

1.5 Relationship between O_2 uptake and CO_2 emission

The relationship between O_2 and CO_2 concentration by respiration is described as inversely proportional and normally the CO_2 presents a higher concentration than O_2 (LESPCH, 2011). This occurs because of the respiration, where 1 mol of O_2 is consumed per 1 mol of CO_2 produced. That relation is called soil respiratory activity and can be determined by calculating the RQ (microbial respiratory quotient) according to Dilly (2001).

The balance and concentration of CO_2 and O_2 in soil depend on some factors, such as (1) The gas exchange facility between soil and atmosphere; (2) Biological process (STOBOVOI, 2001; LEPSCH, 2011), and (3) Chemical reactions (COOK et al. 2004).

In soil higher biological activity takes place with lower available O_2 and higher CO_2 produced by plant root respiration (GLINSKI; STEPNIEWSKI, 1985; COOK et al. 1998; COOK et al., 2007; LESPCH, 2011) and microorganism respiration (ORCHARD; COOK, 1983; STOBOVOI, 2001; DILLY, 2001). Some chemical reactions can consume O_2 (COOK et al. 2004) or produce CO_2 in soil (MARQUES et al., 2000; DELBEM, 2011).

Some soil CO_2 experiments have been developed with CO_2 results in different systems, managements and soil uses without inclusion of available O_2 or correlation between CO_2 and O_2 results, such as: Xu and Qi (2001), Epron et al. (2006), Panosso et al. (2009), Panosso et al. (2012), Bicalho et al. (2014), Almeida et al. (2015) and Moitinho et al. (2015). Indeed, the relationship between O_2 and CO_2 becomes very important when seeking to understand the biological or chemical soil system.

1.6 Use of Biochar in soil

Biochar is classified as a fine-grained and porous substance, similar in appearance to charcoal, that is produced by pyrolysis of biomass under oxygen-limited conditions (CHEN, 2011). The Biochar has a relatively structured carbon matrix with an extensive surface area and high degree of porosity (SOHI et al., 2009).

There are different kinds of biochars (SPOKAS; REICOSKY, 2009; SPOKAS et al., 2013), and they have been produced from different materials (animal manures and lignocellulosic feedstocks) (NOVAK et al., 2013), production processes (heating biomass and oxygen environment) (LAIRD et al., 2009) and aging (surface, oxidation and alteration) (SPOKAS; REICOSKY, 2009; SPOKAS, 2013). These processes and

process parameters, are particularly important to biochar quality, such as: temperature, and the nature of the feedstock (SOHI et al., 2009).

The use of biochar in soil has been cited as a technique to sequester carbon (GOLDBERG, 1985; LEHMANN, 2007; AMELOOT et al., 2013; THOMAZINI et al., 2015) and has received considerable interest as a material to improve crop production (LEHMANN et al., 2006; LEHMANN, 2007; LAIRD, 2008) and soil nutrient availability (soil fertility), such as nitrogen (PARK et al., 2011; LENTZ et al., 2014; AGEGNEHU et al., 2015) and other soil nutrients, such as phosphorus (LAIRD et al., 2010; AGEGNEHU et al., 2015), potassium, magnesium and calcium (LAIRD et al., 2010).

Moreover, the biochar in soil has been correlated with an increase in soil microbiological and enzymatic activity (LEHMANN et al., 2011), carbon degradation by microorganisms (HARRIS et al., 2003; BRUNN et al., 2008; LEHMANN et al., 2011; AGEGNEHU et al., 2015), and providing habitat for soil microbes (PIETIKAINEN et al., 2000), and increased organic carbon content (PENDERGAST-MILLER et al., 2011; LENTZ et al., 2014).

The relationship between the uses of biochar with CO_2 emission has also been observed by some researchers, such as Major et al. (2009), Zimmerman et al. (2011), Lehmann et al. (2011), Lentz et al. (2014) and Lin et al. (2014). However, their results depend on kinds of soil and biochar observed (NOVAK; BUSSCHER, 2009) and there is limited information regarding the associated underlying mechanisms (CHEN et al., 2011).

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2. CHAPTER 2: Use of O_2 uptake as an index of CO_2 respiration in sugarcane areas under different managements

ABSTRACT – The oxygen uptake (FO_2) and carbon dioxide emissions (FCO_2) in soils are important gases, and their concentrations can help understanding the relation of soil especially in environments where this is driven by aerobic microbial activity. This study was developed to examine the relationship and profile of flux of carbon dioxide (FCO_2) using the soil moisture and the flux of oxygen (O_2) as a soil respiration predictor in a sugarcane area under different residues managements (mechanical harvesting - GH versus straw burning - BH). We noticed that there was a lower and relatively constant FCO_2 and FO_2 when the soil moisture has a variation of 6.0 to 8.6% to both managements. However, the BH (87.07 ±19.45 g CO_2 m^{-2}) presented the higher cumulative CO_2 emission, which is 53.68% higher than GH (52.31 ± 15.41 g CO_2 m^{-2}). The relationship between FCO_2 and soil moisture was positive in both treatments, BH (r= 0.74) and GH (r= 0.69), whereas the soil moisture showed a negative correlation with O_2 uptake under GH (r=-0.46; P<0.00). Furthermore, there was a positive correlation between FCO_2 and FO_2 under BH (r = 0.74; P<0.00), and the respiration quotient (RQ) was constant and lower than 1 before the precipitation. So, the FCO_2 and FO_2 profiles and correlation depended on soil, crop residue managements and the FO_2 can be used as an index for categorizing the source of CO_2 respiration. The RQ values higher than 1 were results of soil–gas exchange fluxes after precipitation.

Keywords: Soil biological activity, Soil crop residue, Respiratory quotient, Mechanized planting-harvesting

2.1 Introduction

Measurement of oxygen uptake (FO_2) in soils is important, as it could help in understanding the relation to soil carbon dioxide emissions (FCO_2), especially in environments where this is driven by aerobic microbial activity (STERN et al. 1999). Therefore, this characterization can be useful in determining the impact of soil carbon losses under different agricultural managements. These gaseous exchange rates (FO_2 and FCO_2) are intimately related to the global carbon cycle, considering the FO_2 as a reflection of the carbon cycle (KEELING; SHERTZ 1992) and the relation between them can be a process key to the global carbon cycle (DILLY, 2003)

The soil respiration (CO_2 emission) occurs in soils driven by biochemical processes directly related to the respiration of roots and organic matter decomposition by microbial activity (LAL, 2009; MELILLO et al., 2002). These microbes are mainly aerobic, whose activity increases soil CO_2 gas concentrations to 2.0-3.0% (STERN et al., 1999).

In the soil atmosphere after CO_2 production and accumulation, pressure driven mass flow and diffusion are the main mechanisms responsible for FO_2 and FCO_2 transport in the soil (HILLEL, 1998), and resulting exchange of gases between the soil and the atmosphere (COOK et al., 2008). It must be remembered that the soil atmosphere composition is a balance between the metabolism and growth conditions of anaerobic and aerobic microorganisms (GARDINI et al., 1991).

The gaseous diffusion is primarily responsible for driving FO_2 and FCO_2 into the soil (BENJAMIN et al., 2003) and their transport is due to the concentration gradient between the atmosphere and soil gas phase (BALL; SMITH, 1991; PEREIRA; CRUCIANI, 2009). Since, these concentration gradients are in opposite directions for these two gases, their fluxes will be in opposite directions (CO2 outwards; and O2 inwards towards the soil).

The soil physical attributes directly influence the gaseous exchange with the atmosphere, among them are: the water filled pores - WFP (COOK et al., 2008), soil moisture (PANOSSO et al., 2011), soil depth (CAMPBELL, 1985; COOK, 1995), soil texture, soil water availability (CHEN et al., 2011) and the tillage management (BICALHO et al., 2014). Furthermore, available soil oxygen (CHEN et al., 2011), soil nitrogen and carbon and organic matter (ALMEIDA et al., 2015) also control microbial rates. Therefore, soil nutrients, such as: phosphor, magnesium and calcium, also have an influence on the gaseous exchange with the atmosphere (DUAH-YENTUMI

et al., 1998; XU; QI, 2001), by influencing the production or consumption rates of soil gases.

Soil management and use also are other factors that can influence the FCO_2 and FO_2. Brazil is currently the largest sugarcane producer and has an average an average stalks yield at 684.7 millions of tons in 2016/17 (CONAB, 2016) with an estimated 20 Mg ha^{-1} of residues on the soil surface after harvest (URQUIAGA et al., 1991; OLIVEIRA et al. 1999).

The management with mechanized harvesting of sugarcane without burning or removal of the straw on the soil contributes to a more sustainable management option for sugarcane production (FIGUEIREDO; LA SCALA, 2011). However, sugar cane harvesting continues to be undertaken using two distinct practices in some Brazilian states: mechanical harvesting with straw burning (BH) and without burning (GH) (PANOSSO et al., 2011).

To hypothesis that different residues managements can change the O_2, CO_2 concentrations and relationship with them in sugarcane area this study was run to examine the relationship between FCO_2 and FO_2 using soil moisture, RQ and O_2 as a soil respiration predictor in a sugarcane area under different managements (mechanical harvesting versus straw burning).

2.2 Material and methods

2.2.1 Characterization of the study area

The study was conducted in an area under sugarcane cultivation (*Saccharum* spp.) during July, 2014 (July 4-14th) in Mato Grosso do Sul state, near the municipality of Aparecida do Taboado (20º19'S and 51º13'W). The soil was classified as an oxisol according of Soil Survey Staff (2014), with sandy clay loam texture in the 0-0.2 m layer (Table 1 and Figure 2.1).

The region has a tropical humid climate classified as Aw (PEEL et al., 2007) which has a rainy summer season (Sept–Jun) and a dry winter (Jun-Aug), with an average annual rainfall of 1595 mm in 2014. During the field measurement in July, 2014, there was precipitation in the 13 th and the 14th day with a daily rainfall of 6.1 and 1.5 mm, respectively (Climate Channel at UNESP Ilha Solteira, http://clima.feis.unesp.br).

The effect of residue management was evaluated in two sections of the production field that were under different straw managements (Sections). Section 1: with mechanical harvesting and straw burning (BH); and Section 2: with mechanical

harvesting (GH) and maintenance of the straw. Normally the mechanical harvesting can add the average of 20 Mg of crop residue ha^{-1}. However, this quantity will depend on the variety used and harvest stage (CORREIA; DURIGAN, 2004; TOFOLI et al., 2009; ALMEIDA et al., 2014). Both sections were the same cultivation and harvest management with the sugarcane productivity of 63 and 46 Mg ha^{-1} in 2013 and 2014, respectively.

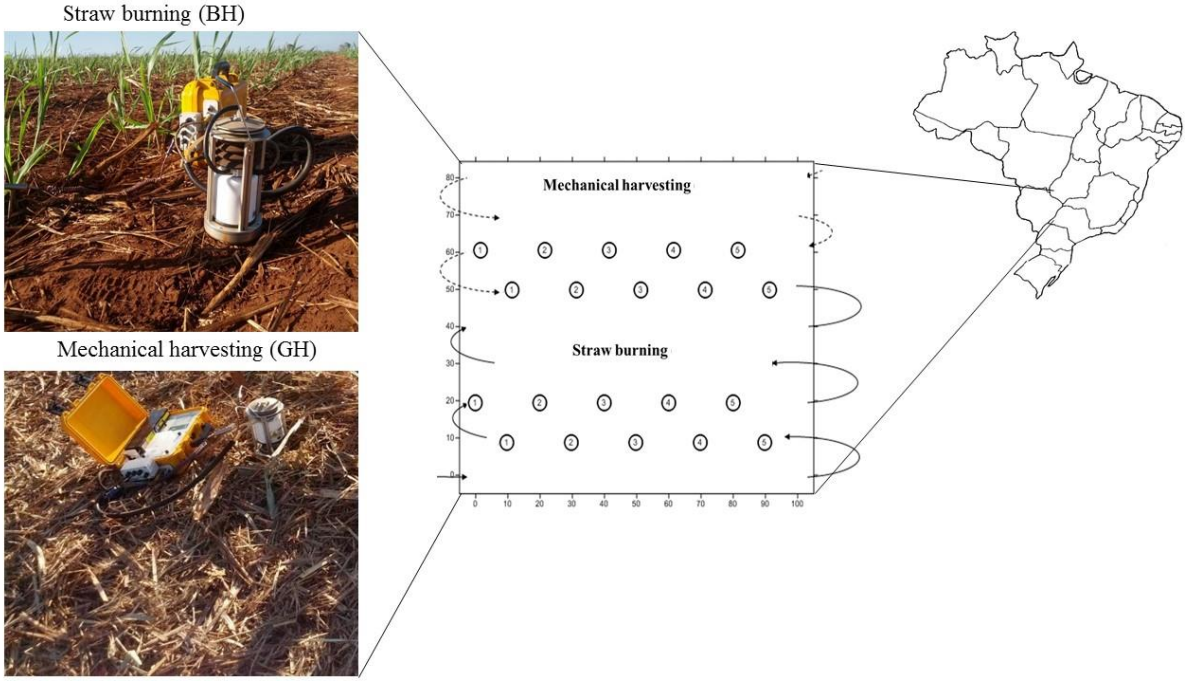

Figure 2.1. Areas under sugarcane cultivation (*Saccharum* spp.) with different straw managements: mechanical harvesting (GH) and straw burning (BH).

The study area was 21.77 ha cultivated with sugarcane, CTC variety, and a population of 60,000 plants per hectare. Historically, the study area has been used for sugarcane production (for 20+ years). The soil was prepared and sugarcane was planted using the conventional system (soil disturbance), application of dolomitic limestone (Dose of 1.5 t ha^{-1}), gypsum (Dose of 1.0 t ha^{-1}) and gypsum. The mechanized planting was performed with furrowing (average depth of 0.35 to 0.4 m), placing 18 buds per m^{-2}.

The fertilization was performed in the furrow with the distribution of 250 kg ha^{-1} of mono-ammonium phosphate (MAP), equivalent to 120 kg ha^{-1} of P_2O_5 and 27 kg ha^{-1} of $N\text{-}NH^{4+}$. Subsequently, topdressing was done with the liquid formula 05-00-13 + 0.3% Zn + 0.3% B, in the amount of 1,000 L ha^{-1}, equivalent to 50 kg ha^{-1} N, 130 kg ha^{-1} of K_2O, 3 kg ha^{-1} of Zn and B. After the first cutting, ratoon fertilization was

performed based on the best management practices applying 90 kg ha^{-1} N, 30 kg ha^{-1} of P_2O_5 and 110 kg ha^{-1} of K_2O.

The sugarcane harvest was done mechanically and with burning for BH in 2014. However, for GH the sugarcane harvest was done mechanically and without burning. In sampling time, the sugarcane plants were 20 cm high and it was approximately 2 weeks after their 2rd cutting (harvest) in 2014. Thus, the area did not have plants in higher growth stages.

Field sampling was conducted by selecting 10 points with at least 5 m spacing from each management sector. We used 10 points because the oxygen sensor takes about 15 minutes to analyze one simple. The results of the analyses of the soils from the two sections (0-0.2 m layer) are shown in Table 1. Physical and chemical attributes did not show differences between the systems observed.

This table presents the soil physical (Sand, Silt and Clay) and chemical properties (hydrogen ionic potential-pH; soil organic matter-SOM; phosphorus-P; sulfur-S; calcium-Ca^{+2}; potassium-K^{+}; magnesium-Mg^{+2} and aluminum-Al^{+3}) beside the soil porosity (Macro, micro porosity and Total porosity) and water full pores (WFP). These analytical results were analyzed through Embrapa methodology (1997).

Table. 1. Physical and chemical attributes of a Red-Yellow Latosol in use with sugarcane with mechanized harvesting with the presence of straw (GH) and straw burning (BH).

	Soil chemical attributes							
	pH	SOM	P	S	Ca^{+2}	K^{+}	Mg^{+2}	Al^{+3}
	-	g dm^{-3}	-------- mg dm^{-3} --------		----------$mmol_c$ dm^{-3} ----------			
BH	5.11±0.0	16.30±0.5	8.0±0.2	5.5±0.2	19.7±1.1	1.36±0.0	9.1±0.8	0.8±0.2
GH	5.18±0.0	15.90±0.2	8.2±0.2	5.3±0.2	22.0±0.8	1.26±0.1	8.7±0.5	0.9±0.31
	Soil chemical attributes				Soil physical attributes			
	Sand	Silt	Clay	Macro	Micro	TP		WFP
	---------- g Kg^{-1}----------				----------%----------			
BH	613±1.0	101.0±1.0	286.0±0.0	14.6±2.0	29.0±0.6	43.69±1.6		18.73±5.6
GH	602±0.3	111.5±0.3	286.0±0.0	11.1±1.3	31.58±1.0	42.75±1.0		18.49± 6.4

In the table: the hydrogen ionic potential is represented by pH; soil organic matter-SOM; phosphorus-P; sulfur-S; calcium-Ca^{+2}; potassium-K^{+}; magnesium-Mg^{+2}; aluminum-Al^{+3}; cation exchange capacity-CTC; Microporosity (Macro); Microporosity (Micro); Total pore (TP); water full pores (WFP). The physical and chemical attributes were compared using the Student's t test ($P \leq 0.05$)

The water-filled pores (WFP) was calculated using Equation 1 by Linn and Doran (1984). Where the soil moisture was the volumetric water content percent (%) and the total soil porosity percent (TP). To calculate the TP (TP= (1 - PB/PP)*100) we

used the soil particle density (PP) was assumed to be 2.65 Mg m^{-3} and soil bulk density (PB) in Mg m^{-3}.

$$\%WFP = \left(soil\ moisture / TP\right)100 \quad \text{Eq. (1)}$$

2.2.2 Soil sample and CO_2, O_2 soil moisture analyzed

PVC rings (polyvinyl chloride), 10 cm in diameter and 8.5 cm in height were previously installed and fixed at the sample points. After 24 hours, the CO_2 emissions (FCO_2), O_2 uptake (FO_2) and temperature in 6 separate measurement days (July 4th, 6th, 8th, 10th, 12th and 14th) totaling 10 observation days. We used this period of time because we wanted to observe the relationship of these variables to the soil without the confounding contribution from crop growth at higher stages. The soil measurements were collected in the morning between 7 and 8 am.

To collect FCO_2 we used an IRGA (LI-COR 8100A) which has a closed circulation system with an internal volume of 854 mL and a soil contact area of 84 cm^2 (LI-COR Inc. Linclon, NE, USA). The IRGA has an infrared (IR) system that measures the CO_2 concentration by optical infrared absorption spectroscopy (Figure 2.2a).

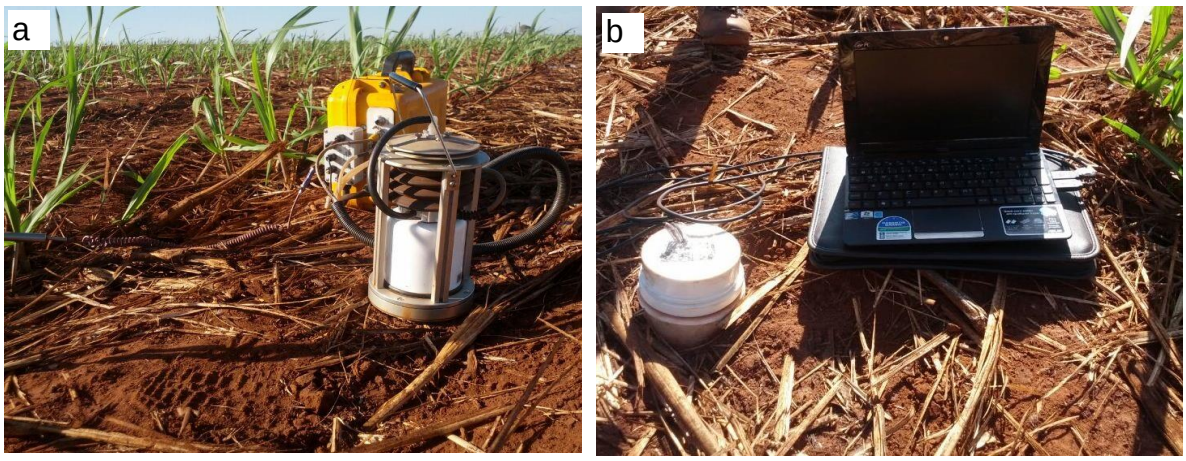

Figure 2.2 The IRGA system (a) and O_2 sensor used in this experiment.

The soil moisture was measured using a portable TDR system (Time Domain Reflectometry; Hydrosense™; Campbell Scientific, Australia) that determined the soil moisture according to the dielectric constant of the travel time of an electromagnetic pulse in the space between the two end points (2 rods, 12 cm high) inserted into the soil adjacent to the PVC collars (0-10 cm).

The soil FO_2 was monitored by an O_2 sensor (CM-021; CO_2 Meter, Inc., Ormond Beach, FL, USA) with a full scale span of 0–25% (v/v). This sensor is portable and utilizes ultraviolet light (UV) fluorescence to assess the oxygen concentration (Figure 2.2b). The sensor result was read using the software (Gaslab) to calculate the soil O_2 uptake rate.

With the CO_2 and O_2 results we calculated the respiratory quotient - RQ (mol mol^{-1}) according to Dilly (2001), Dilly (2003) and Wolinska (2011), where the RQ is a ratio of the CO_2 emission and O_2 uptake.

2.2.3 Calculus of soil FO_2

The soil O_2 uptake rate (dO_2/dt) was calculated by a linear interpolation of the concentration values as a function of time, during the first 300 seconds of sampling. Specifically, the soil FO_2 was calculated by Eq. (2) taking into account the pressure, temperature and volume of the gas trapped in the chamber. In Equation (1), $FO_2(t)$ is the amount of O_2 measured at time t, the dO_2 is the concentration change in relation to the unit of time (dt) in the area of the collar surface (A) (JASSAL et al., 2012; GIACOMO et al., 2014).

$$FO_2(t) \equiv {dO_2}/{dt \times A} \qquad \text{Eq. (2)}$$

The initial O_2 readings are in concentration units of parts per million (ppm). The PVC had a volume of 0.00066 m^3 and area 0.008 m^2. The volume measured by the sensor (ppm) was converted to moles of O_2 using the ideal gas law, Equation (3).

$$P(\Delta V) = (\Delta n)RT \qquad \text{Eq. (3)}$$

After Equation 2, we calculated the O_2 uptake by time (dO_2/dt). In Equation (4), the ΔV is the O_2 uptake (dO_2/dt), P is the pressure (Pa), T is the atmospheric temperature (K) and R is the universal constant of perfect gases (J mol^{-1} K^{-1}).

$$FO_2 \equiv {P\,\Delta V}/{\Delta nRT} \qquad \text{Eq. (4)}$$

2.2.4 Data processing and statistical analysis

The soil moisture, O_2 uptake, CO_2 emission and RQ daily mean were calculated using N (number) of 10 per day and compared by Student's t-test (P≤0.05) per each management sector. Consequently, the FCO_2, FO_2 and total RQ were calculated using all days observed with an N of 60 per treatment. Thus, an integration of the area under the FCO_2 and FO_2 curves were calculated using the Origin 7 program (Origin Lab Corporation 2002). Thereafter, the treatment results were compared by Student's t-test (P≤0.05).

The relationship between soil CO_2 emission and O_2 uptake with soil moisture was calculated using regression analysis and the Pearson correlation for both managements. The analysis of presuppositions was conducted using residuals analysis and identifying the outliers and influent values by Leverage statistics. The normality of residuals was verified by test Cramer-von-misses at 5% probability.

2.3 Results

2.3.1 Daily behavior of soil variables

The soil CO_2 emission on the 4^{th}, 6^{th}, 8^{th} and 10^{th} day were similar, especially in GH, with means of 0.95; 1.32; 0.76 and 0.83 µmol m^{-2} s^{-1} and 1.65; 1.54; 1.41 and 1.52 µmol m^{-2} s^{-1} for GH and BH, respectively (Figure 2.3d).

We can see that both treatments showed lower and relatively constant CO_2 emission when the soil moisture had 6.44 to 8.6% and 6.0 to 7.4% for BH and GH, respectively (Figure 2.3b). However, after the 10^{th} day the CO_2 emission and soil moisture increased with means of 4.87 and 2.26 µmol m^{-2} s^{-1} and 14.33 and 14.11% for BH and GH (Figure 2.3d, b). This maximum observed soil moisture value followed a precipitation event of 6.1 mm (Figure 2.3a).

The BH had higher CO_2 emission in all days observed with a statistical difference in GH according to student test (p≤0.05) on the 4^{th}, 8^{th}, 10^{th}, 12^{th} and 14^{th} day. The highest BH difference occurred on the 12^{th} day of study, with a CO_2 emission increase of 54.0% compared to the GH treatment (Figure 2.3d).

The temporal variability of soil O_2 uptake was inverse compared to the CO_2 emission in this study. The O_2 uptake was lower with variation of 0.22 to 0.46 and 0.20 to 0.40 mg m^{-2} s^{-1} on the first days (4^{th} and 10^{th}). 1110 However, after the precipitation event the O_2 uptake had a decrease of 73.0% for BH and GH treatments, respectively (Figure 2.3c).

When comparing the treatments we can also notice that the soil O_2 uptake did not present a difference between the treatments for all days observed according to the student t-test ($p \leq 0.05$). The BH and GH mean for all days observed were 0.24 and 0.25 mg m^{-2} s^{-1}, respectively (Figure 2.3c).

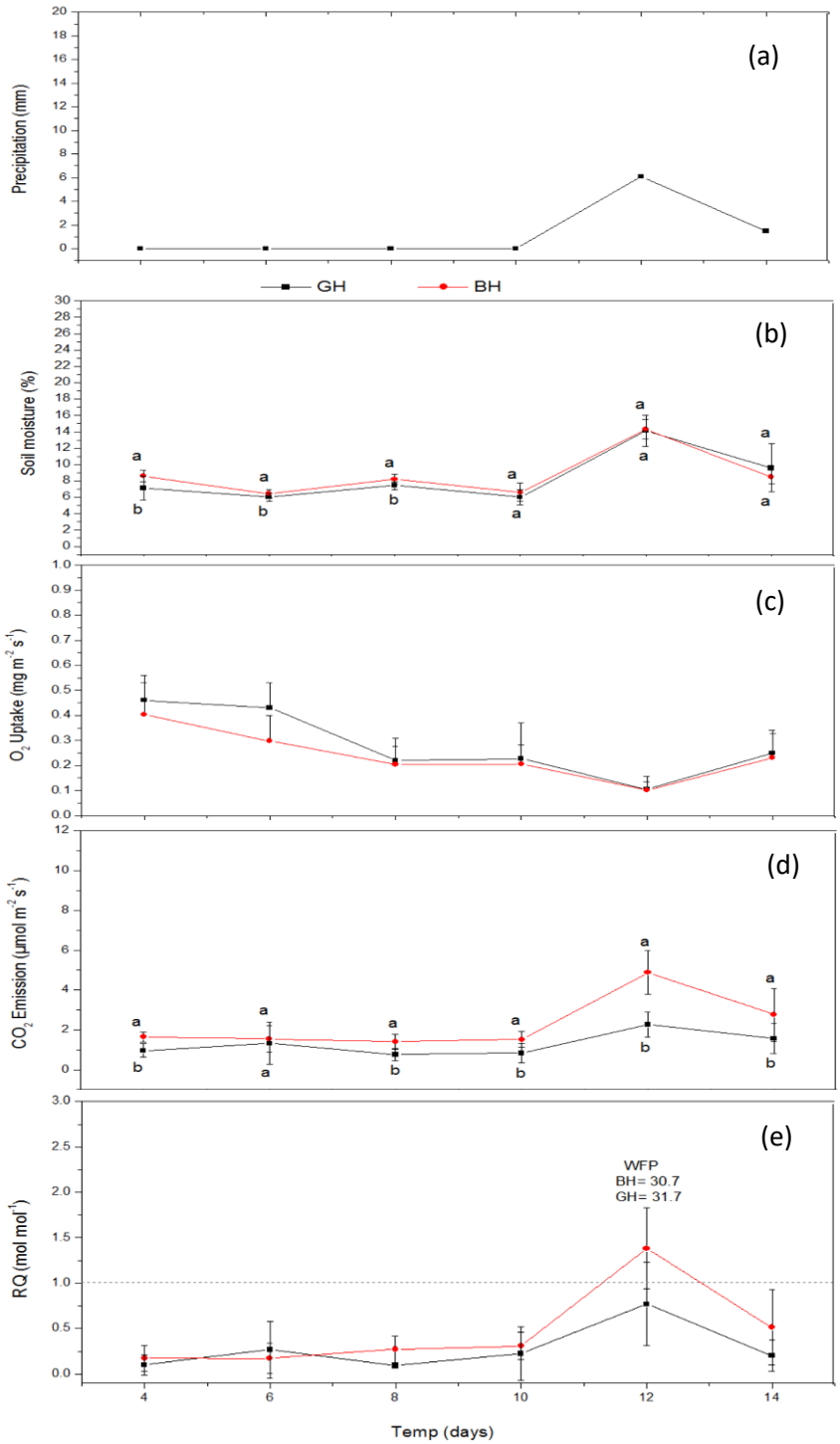

Figure 2.3. Precipitation (mm) (a), soil moisture (%) (b), O_2 uptake (mg O_2 m^{-2} s^{-1}) (c), CO_2 emission (µmol m^{-2} s^{-1}) (d) and respiratory quotient - RQ (mol mol^{-1}) (e) in soil with sugarcane managements with mechanized harvesting GH) and burned straw (BH). In the Figure 3b and d: days identified with lower-case when distinct, differ by the Student's t-test ($P \leq 0.05$).

The RQ behavior was very similar to the CO_2 emission, soil moisture and precipitation behaviors (Figure 2.3). In other words, the RQ was constant, lower than 1, and had a mean variation from 0.9 to 0.27 and 0.17 to 0.31 for GH and BH treatment from the 4th to 10th days, respectively. However, after precipitation (12th day) the RQ was higher than 1 and obtained the highest RQ mean in BH (1.38 ± 0.46 mol mol^{-1}), while in GH the mean was lower than 1 (0.77 ± 0.46 mol mol^{-1}).

2.3.2 Accumulated FCO_2, FO_2 and RQ

The cumulative FO_2 had means of 202.52 (±49.62) and 267.41 (±73.6) g O_2 m^{-2}, respectively, for the GH and BH managements (Figure 2.4a). When compared by student t-test (P≤0.05) we did not find a significant statistical difference between harvesting techniques on the FO_2 uptake.

The BH also had higher CO_2 emission cumulative (87.07 ±19.45 g CO_2 m^{-2}), when compared with GH (52.31 ± 15.41 g CO_2 m^{-2}). However, for CO_2 emission, the BH is significantly different (P≤0.05) with a 50.0% increase compared to the GH treatment (Figure 2.4a).

The cumulative ratio between CO_2 and O_2, represented by RQ total, was lower than 1 in both treatments with means of 0.59 (±0.18) and 0.43 (±0.13), respectively for BH and GH (Figure 2.4b). When comparing the RQ for both treatment we did not see a significant statistical difference between them by the Students t test.

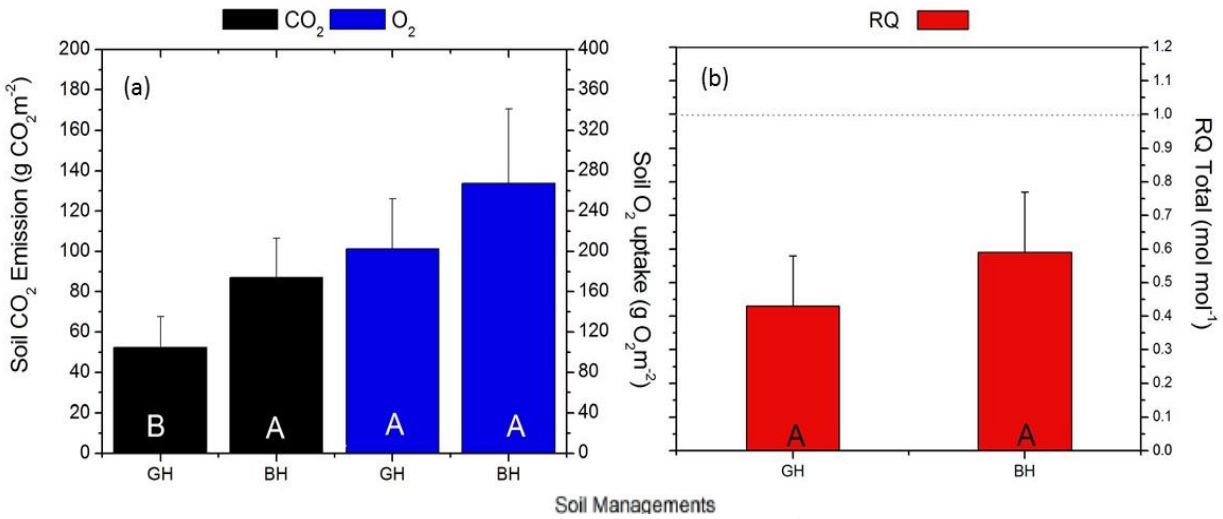

Figure. 2.4. Soil CO_2 emission cumulative (g CO_2 m^{-2}), soil O_2 uptake cumulative (g O_2 m^{-2}) (a) and RQ total (mol mol^{-1}) (b) in the ground with sugarcane managements with mechanized harvesting (GH) and burned harvesting (BH). In the figure: bars identified with upper-case letters when distinct, differ by the Student's t-test (P≤0.05).

2.3.3 Relationships between soil CO_2 emission and O_2 uptake with soil moisture Soil

The relationship between CO_2 emission and soil moisture was positive and significant in both treatments (Figure 2.5a, c). However, the BH had the higher correlation (r= 0.74) than GH (r= 0.69).

Despite the fact that the high correlation of soil moisture with CO_2, when we compared the soil moisture with O_2 uptake, was not significant for the BH treatment. However, the soil moisture for GH showed the negative correlation with O_2 uptake (r=-0.46), Figure 2.5b and d.

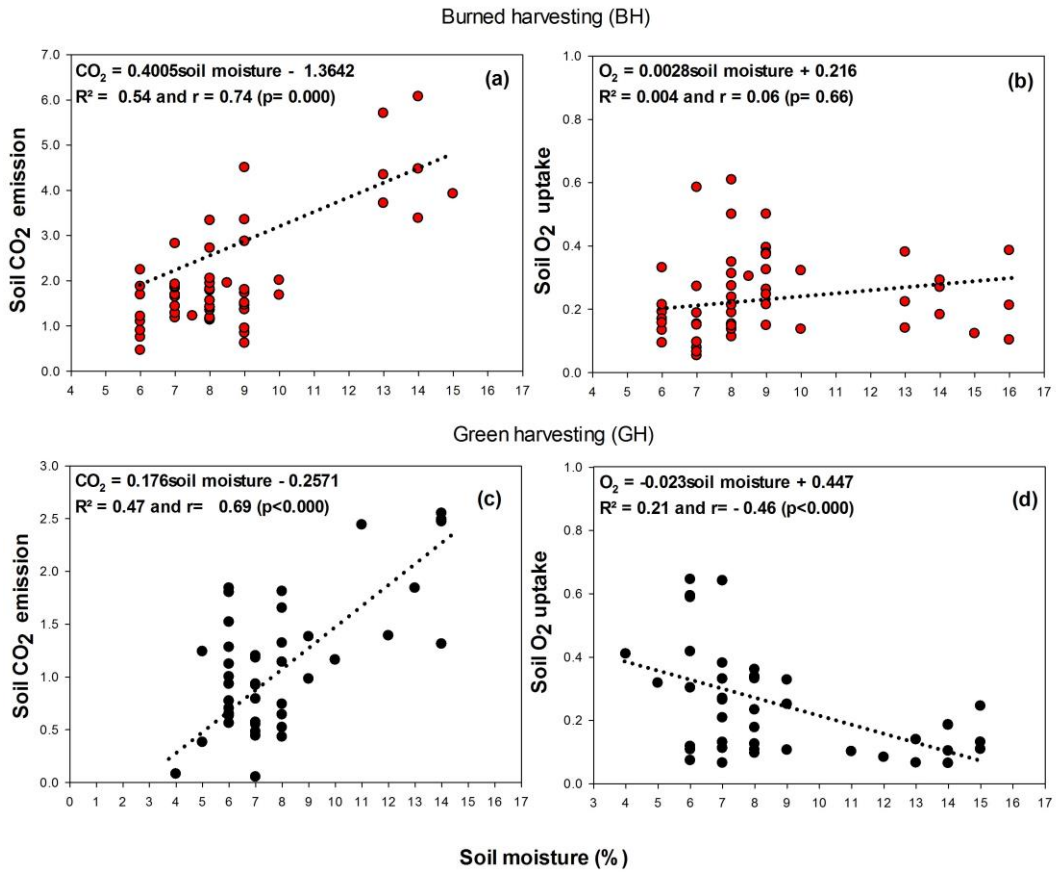

Figure. 2.5. Relationship between soil CO_2 emission (µmol m^2 s^{-1}) and O_2 uptake (mg of O_2 m^{-2} s^{-1}) (a and c), and CO_2 emission (µmol m^2 s^{-1}) and soil moisture (%) (b and d), in sugarcane soil with mechanized harvesting (GH) and burned harvesting (BH).

The relationship between CO_2 emission and O_2 uptake had opposite results for the BH and GH treatments. In BH, the correlation was negative and significant, but in GH there was no correlation between them (Figure 2.6). We also noticed that the GH (31.6%±1.05) had a higher microporosity distribution compared with BH (29.0%±0.60). However, GH had a lower macroporosity distribution with a decrease

of 23.9% (Table 1). Furthermore, the WFP was higher in BH (18.73%±5.6) compared to GH (18.49±6.4)

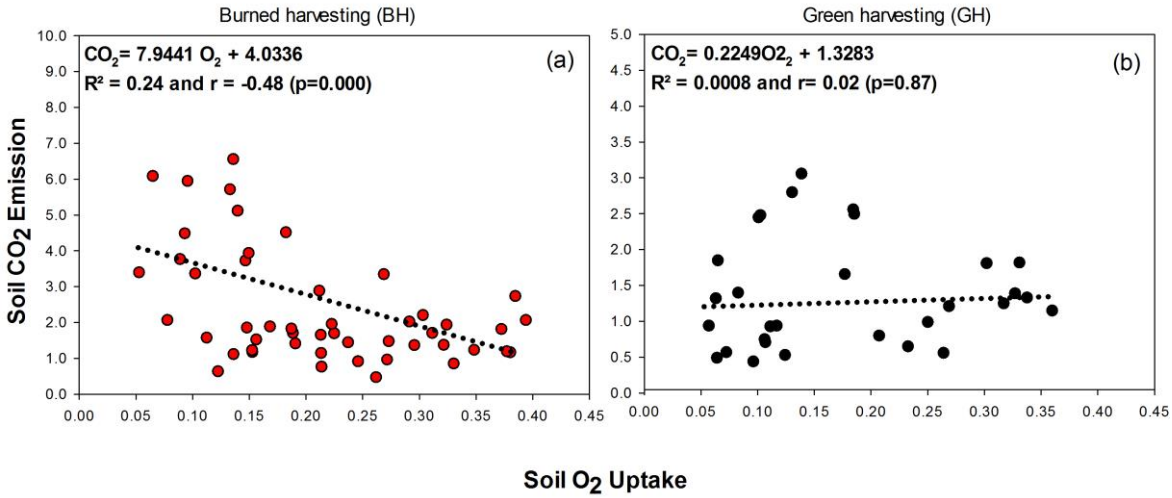

Figure. 2.6 Relationship between soil CO_2 emission (µmol m^{-2} s^{-1}) (a) and soil O_2 uptake (mg of O_2 m^{-2} s^{-1}) (b) in sugarcane soil with mechanized harvesting (GH) and burned harvesting (BH).

2.4 Discussion

2.4.1 CO_2 and O_2 results (Daily and accumulated)

We noticed a relative consistent magnitude of the FCO_2 on the 4th, 6th, 8th and 10th day for GH and BH which suggests a corresponding stability in soil microbial activity (Figure 2.3). The microbial stability occurs after the soil carbon mineralization of soil organic matter (CUNHA et al., 2011; BADÍA et al., 2013; KNICKER et al., 2013) and leads to higher emissions with higher microbial activity (LUO et al., 2006). However, we are not able to separate it into the components since the net emission comes from the biological activity of soil microbial respiration and root respiration as noticed by Stern et al. (1999), Lal (2009) and Melillo et al. (2002).

The higher CO_2 emission (Daily and Accumulated) in BH, compared to GH (Figure 2.3 and 2.4), has been observed in other studies, such as Panosso et al. (2009), Panosso et al. (2011) and Corradi et al. (2013) working with a similar Oxisol soil and BH and GH managements in the São Paulo state region. This difference could be explained by the higher nutrient availability in the BH treatment, due to burning, which has been mentioned by Marques et al. (2009) and Panosso et al. (2011). However, in our experiment we did not observe a significant difference in soil nutrients available in BH and GH. On the other hand, we noticed that the BH treatment presented higher macroporosity and PT, and lower microporosity

compared with GH (Table 1). The high porosity in BH could have been the result of burned sugarcane residue. Therefore, after the burned the porosity could have increased due to the opening of potentially charred root channels. Remembering that both the BH and GH had the same mechanical harvesting traffic and soil preparation.

The relationship between soil porosity and CO_2 emission has been reported by Xu and Qi (2001), Epron et al. (2006), Panosso et al. (2012) and Bicalho et al. (2014). It is so important that, according to Moitinho et al. (2015), when studying the FCO_2 variability (spatial and temporal) it is necessary to include soil porosity variability. Wick et al. (2012) also mentioned that the soil porosity can help to explain the soil CO_2 emission results.

The soil porosity is responsible for soil gaseous transport (XU; QI 2001; EPRON et al., 2006) and movement of organic and inorganic solutions throughout the soil (RANJARD; RICHAUME, 2001). Consequentially, a porosity with a higher macroporosity proportion facilitates soil oxygenation, microbiological activity and increases FCO_2 (FANG et al., 1998).

Additionally, a larger portion of macropores would result in potentially faster infiltration rates, since the water would preferentially fill larger pores first (HILLEL, 1980). Moreover, soil porosity represents the natural habitat for microbial communities (RANJARD; RICHAUME, 2001).

The lower FCO_2 in GH to be a result of the sugarcane residues on the soil surface. In addition, these residues have a high C/N ratio (83.63) (ALMEIDA et al., 2015), lignin (25.80%) (COSTA et al., 2013), cellulose (72.90%) (ALMEIDA et al., 2009), and lower crude protein concentration (2.50%) (PEREIRA et al., 2000). These characteristic are important parameters in nutrient dynamics (LAL, 2004) and consequently, there is reduced accumulated CO_2 due to slow residue decomposition (ALMEIDA et al., 2014).

Some researchers have been noticed that the surface residue is an additional barrier for diffusive transport. It also certainly contributes to increased water retention (OHASHI; GYOKUSEN, 2007; CONCILIO et al., 2009) and decreased evaporation, thus providing a buffer for abrupt temperature fluctuations (MARQUES et al., 2009). It has happened because of the mechanical sugarcane harvest added an average thickness between 10 and 12 cm of accumulated residues on the soil surface (OLIVEIRA et al., 1999; ALMEIDA et al., 2015). However, we did not notice the increased water retention (soil moisture) at GH in our experiment (Figure 2.3b).

Differently from CO_2, the O_2 uptake did not present a significant difference in the BH and GH treatments (for all days observed and O_2 accumulated), Figure 1 and 2. Concentration profiles of O_2 have a correlation with soil porosity (HILLEL, 1998; BRADY; WEIL, 2013), and the results, because the soil porosity is where the O_2 can be found (GLINSKI; STEPNIEWSKI, 1985; HILLEL, 1998). This relationship is so important to soil microbial activity that Glinski and Stepniewski (1985) mentioned that the soil porosity is the oldest and simplest soil attribute that can be used as soil O_2 indicator.

2.4.2 Relationships between soil CO_2 emission and O_2 uptake with soil moisture

The temporal variability of soil O_2 uptake was inverse when compared to CO_2 emission after precipitation, in our study (Figure 2.3). In the other words, after the precipitation, the O_2 uptake decreased and CO_2 increased in both treatments (Figure 2.3c). Thus, the soil moisture can be considered as the key abiotic factor that affects the CO_2 emission (GARDINI et al., 1991; HOWARD; HOWARD, 1993) and O_2 uptake processes (GARDINI et al., 1991).

Some researchers believe that soil moisture and soil aeration are the main factors that influence the CO_2 emission into the atmosphere (FANG; MONCRIEFF, 2001; COOK; ORCHARD, 2007; WEI et al., 2014). In fact, the soil temperature is another attribute that can also have an important influence (BENJAMIN et al., 2003; KYAW THA PAW et al., 2006; LAL, 2009; CHEN et al., 2011). Higher soil moisture has been observed to promote up to an 80% increase in FCO_2 (CHEN et al., 2011) as a result of higher microorganism and root activity (LAL; KIMBLE, 1995; SMITH et al., 2003) and lower diffusion of gases through the soil pores (HILLEL, 1998; IGNATIUS, 1999; SMITH et al., 2003).

That positive correlation between CO_2 and soil moisture is reported for BH and GH treatments (Figure 3). It has also been observed by Ignatius (1999), Epron et al. (2006), Lal (2009), Moitinho et al. (2014) and Wei et al. (2014). Furthermore, Corradi et al. (2013) hypothesized that this correlation was positive and linear in soil moisture variations of 38-47% for an Oxisol with high clay content (636 g kg^{-1}). According to Doran et al. (1990) and Chen et al. (2011) the highest soil respiration rates occur having between 40 and 70% water-filled pore (WFP) space in a majority of soils. In our experiment, the WFP was less than 70% of all treatments and days observed (Table 1).

The high correlation between FCO_2 and soil moisture in BH was correlated to higher macroporosity and lower microporosity, as we saw in our results (Figure 2.5 and Table 1). Moreover, the higher correlation coefficient in the BH, compared to GH, suggests that there is a high sensitivity of burned residue management to soil moisture variations. This results because of the connection between soil macroporosity and transport rates (SILVA et al., 2005). This concept is supported by the results of Ruser et al. (2006), in which they explained that the soil moisture did not influence the CO_2 emissions as much as the differences in porosity between compacted inter-row soils and the un-compacted inter-row, where compacted soil has fewer macrospores and more microspores at bulk densities of 1.24 and 1.65 g cm^{-3}, respectfully. Ceddia et al. (1999) noticed the water infiltration in sugarcane areas has a variation according to soil aggregations and porosity and normally the infiltration is faster in soil with higher macroporosity than microporosity.

Decreased O_2 uptake after precipitation events was also observed by Linn and Doran (1984) and Gardini et al. (1991). Water infiltration reduces the amount of O_2 in soil pores (COOK et al., 2007), and the water is a very effective at limiting the gas exchange (O_2) between the soil atmosphere and air atmosphere (ARMSTRONG; DREW, 2002; ELBERLING et al., 2011). Moreover, in that condition, there is a strong correlation between O_2 uptake and water filled pores (QUASTEL, 1965; COOK et al., 1998).

2.4.3 Relationships between soil CO_2 emission and O_2 uptake

The FCO_2 and FO_2 were inversely correlated in the BH treatment. However, we did not notice a relationship between them in the GH treatment. We also observed that the FO_2 was higher than FCO_2 in both treatments. The higher O_2 was also noticed by Angert et al. (2015) studying temperate and alpine forest ecosystems, and the relationship between FCO_2 and FO_2 in soil has also been noticed by Kyaw Tha Paw et al. (2006). With this result we can notice that there is a CO_2 increase as result of O_2 uptake in soil with a soil moisture variation of 6.44 to 14.5%. This relationship is more expressive in management without surface residue.

To understand and explain the FO_2 as a respiration predictor we used the calculated respiration coefficient (RQ), where RQ values close to 1, is a result of aerobic respiration in soil with WFP lower than 70% and, this aerobic reaction has a higher metabolic efficiency. It should be remembered that RQ is determined on the basis of the CO_2 emission and O_2 uptake rate (DILLY, 2001). Some researchers,

such as Stotzky (1960), Alef (1995) and Dilly (2003), utilize RQ to elucidate the relation between the FCO_2 and FO_2. The RQ index has also been used as a criteria of the soil microbial activity across different WFP conditions (STOTZKY, 1960).

We observed that the RQ was lower than 1 for BH and GH treatment before the precipitation on the days observed. Therefore, the RQ was higher than 1 after precipitation (12^{th} day), with soil moisture variations from 6.4 to 14.5% and WFP values lower than 32.0% for BH and GH, respectively. In other words, the RQ was higher than 1 under soil aerobic conditions.

According to Linn and Doran (1984) the increase of RQ values of 1.3 to 1.7 occurs with an increase of soil water and WFP value higher than 70%. Nevertheless, in our experiment on the 12^{th} day presented precipitation and the WFP value was not >70%, and the treatments had WFP values of 30.7% and 31.7% for BH and GH, respectively. Therefore, under this WFP condition, there was an aerobic respiratory predominance, as described by Franzluebbers et al. (1999), who noticed the maximum respiratory activity of soil microbial biomass at WFP levels ranging between 27% and 68%.

Values of RQ >1 are suggestive of chemical and physical soil processes (e.g., Soil–gas exchange fluxes). According to Angert et al. (2015), the variations in the RQ ratios in soil profiles can be a result of soil–gas exchange fluxes associated with biological respiration. Linn and Doran (1984) and Lal (2009) state that the physical process (soil–gas exchange fluxes) of water infiltration (percolation) triggers the expulsion of significant amounts of soil gases (e.g. CO_2). Chemical additions can also trigger CO_2 fluxes, such as liming (SILVA et al., 2015; ANGERT et al., 2015) and urea additions (MARQUES et al., 2000; DELBEM, 2011). However, the CO_2 emission in the Mediterranean climate calcareous soils can be described by a diffusion process alone (ANGERT et al., 2015).

High RQ values (> 1) can also occur with certain environmental conditions (ALEF, 1995), such as available soil or substrate compositions, the soil microbial community current nutritional conditions (DILLY, 2001; KUTZNER, 2013), carbon source (KUTZNER, 2013) or higher water contents (LINN; DORAN, 1984; GARDINI et al., 1991; IKEDA; NAKAMURA, 1996). Therefore, some environmental conditions may control the RQ ratio (DILLY, 2001).

We believe that our results support the use of RQ as a respiration predictor with RQ values lower than 1; a situation that helps to explain the alterations in soil O_2 and CO_2 fluxes. We also believe that our results can assist in the formulations of

mechanistic models for soil gas emissions (e.g. COOK, 1995; COOK et al., 2007; COOK; KNIGHT, 2003).

2.5 Conclusion

The FO_2 is inversely correlated with soil moisture in the GH treatment. There is a positive correlation between soil moisture and FCO_2.

The FCO_2 and FO_2 profiles and correlation depend on soil and crop residue managements.

The FO_2 can be used as an index for categorizing the source of CO_2 respiration. The RQ values higher than 1 are results of soil–gas exchange fluxes after precipitation.

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3. CHAPTER 3: How O_2 uptake can help us understand the CO_2 sorption processes by biochar?

ABSTRACT - The stocks of carbon in soil are the results of the balance between the carbon in soil and the atmosphere. This balance is a result of carbon inputs (Leaf and root detritus) and outputs (mainly carbon dioxide - CO_2 and methane - CH_4). The carbon dioxide (CO_2) sorption processes was investigated using the oxygen (O_2) uptake to monitor and understand biochar's impact in amended soils with three soils types (Rosemount - RM; Potting soil Sunshine - PS; and UM) and five biochars (Pine chip biochar - ICM; Royal Oak hardwood lump charcoal - RO; Accurel activated charcoal - AAC; Bamboo - B; and Macadamia nut - MC) and control treatment (Soil without biochar). We noticed that the RO, AAC and MC were able to sorption 100% of CO_2 from the atmosphere with significant difference to B and ICM, who absorbed 67.14 and 34.85%, respectively, without correlation between CO_2 and O_2 and respiratory quotient (RQ) values so much lower than 1 on the first days of incubation. The O_2 can also sorption by biochar without large difference between the biochars studied. When incubated the biochar+soil there was a CO_2 increase of 87.87% when added biochar in soil while to the O_2 happened a decrease of 11.58% after 57th day of incubation. Moreover, this temporal variability can be divided in 3 periods (P1, P2 and P3) with different behaviors and negative correlation between CO_2 and O_2 and RQ close to 1. Therefore, the biochars showed different results of soil and isolated experiments. Moreover, more study should be developed to elucidate the CO_2 and O_2 sorption by biochar and their reactions in soil.

Keywords: Biochar Activation; Charcoal; CO_2 sequestration; CO_2 and O_2 correlation; CO_2 and O_2 sorption

1.0 Introduction

The stocks of carbon in soil are the results of the balance between the carbon in soil and the atmosphere (DAVIDSON; JANSSENS, 2006). Consequently, this balance is a result of carbon inputs (Leaf and root detritus) and outputs (mainly carbon dioxide - CO_2 and methane - CH_4 (FEARNSIDE; BARBOSA, 1998), wherein the terrestrial biosphere can be a source or as a sink for CO_2 emission (GUO; GIFFORD, 2002).

The soil CO_2 emission is almost entirely by biochemical processes, and it is directly related to the respiration of roots and organic matter decomposition by microbial activity (MELILLO et al., 2002; DAVIDSON; JANSSENS, 2006; LAL, 2009; DILLY et al., 2010). However, the organic matter decomposition can be changed according some soil conditions, for instance: the temperature (CHEN et al., 2011), moisture (PANOSSO et al., 2011), oxygen (CHEN et al., 2011) and carbon available (CAYUELA et al., 2009; Guillou et al., 2011; ALMEIDA et al., 2014; 2015).

In the last years the CO_2 emission from soil to the atmosphere has been increasing as results of soil uses and managements (LEUNG et al., 2014; ESRL, 2016). However, this CO_2 can also be sequestrated from the atmosphere (BATJES, 1999) to the soils, sediments, vegetation (LAL, 2004a; LAL, 2004b; JACKSON et al. 2005) and oceans (LAL, 2004b). For these reasons, some researchers have been studying and explaining more about the CO_2 sequestration process from the atmosphere, for example: Effat et al. (2005) and Figueroa (2008), Escobedo et al. (2009), Huang et al. (2012).

The use of biochar in soil has been cited as a technical to sequester CO_2 (GOLDBERG, 1985; LEHMANN, 2007; AMELOOT et al., 2013; THOMAZINI et al., 2015) and sorption of NO_3^- (DEMPSTER et al., 2012; CLOUGH et al., 2013), fungicides as tricyclazole (GARCÍA-JARAMILLO et al., 2015) and soil organic pollutants (CHEN; CHEN, 2009; YU et al., 2010). However, there are different kinds of biochars (SPOKAS; REICOSKY, 2009; SPOKAS, 2013; HIGASHIKAWA et al., 2016), and they have made produced from different materials (animal manures, lignocellulosic feedstocks) (NOVAK et al., 2013), production processes (heating biomass, oxygen environment) (LAIRD et al. 2009) and aging (surface, oxidation, alteration) (SPOKAS; REICOSKY, 2009; SPOKAS, 2013). As consequence of that variety is important to understand more about the impact of applying biochar (CASE et al., 2012; CAYUELA et al., 2013) in soils.

To hypothesis that the CO_2 and O_2 concentrations and RQ values are different soils and biochars conditions. We have run an experiment measuring biochar's impact on CO_2 production or sorption and O_2 uptake in amended soils.

2.0 Materials and Methods

2.1 Soils and biochars sampling

Laboratory incubation experiments were performed at the University of Minnesota, campus Twin City, 2015. Initially, we divided the experiment in two experimental designs to study three soil types (Rosemount - RM; Potting soil Sunshine - PS; and UM) and five different biochars (Pine chip biochar - ICM; Royal Oak hardwood lump charcoal - RO; Accurel activated charcoal - AAC; Bamboo - B; and Macadamia nut - MC) and control treatment (Soil without biochar). In Figure 3.1, we are showing the biochars used in this experiment.

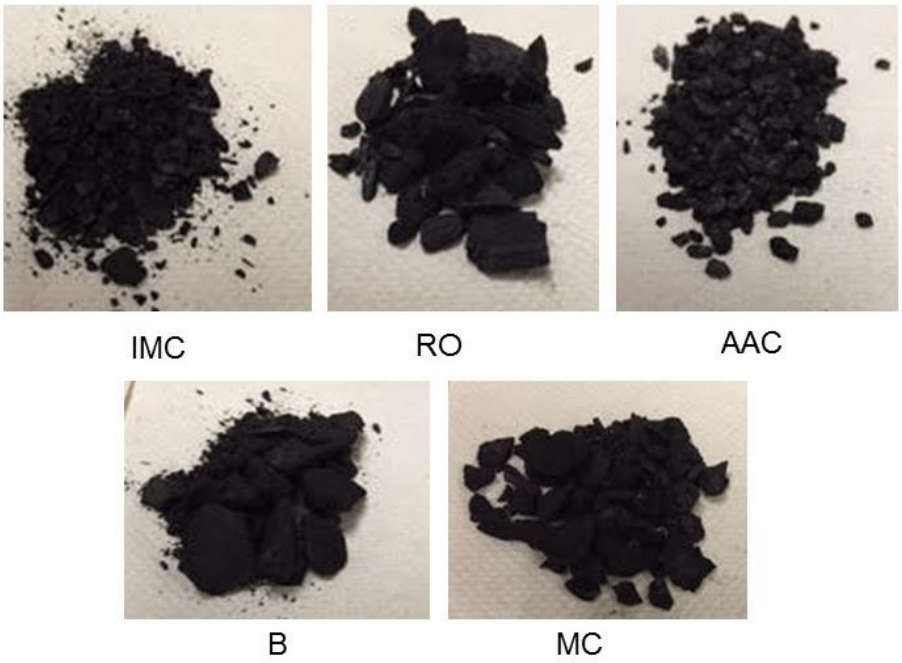

Figure. 3.1. Biochars used in this experiment.

The three soils used in this experiment and associated characteristics are summarized in Table 3.1. The two surface soils were collected (0-5 cm) and then air-dried until the start of the incubations to RM (Waukegan silt loam, collected at Rosemount, MN), UM (Chetek sandy loam, collected at St. Paul, MN). The synthetic potting soil mixture (PS) was used as received (Brand #1; Sun Gro Horticulture Distribution Inc, Agawam, MA). The five selected biochars also were subjected to

characterization. The feedstock and pyrolysis conditions of the biochar samples are given in Table 3.1.

Table 3.1 Soil and Biochar characterization.

Property	*Soils*		
	PS	*RM*	*MN*
pH (1:1 DI water)	6	6.5	6.2
OM* (g 100 g^{-1})	1.5	3.5	2.0

Biochars				
	Feedstock	*Pyrolysis[1]*	*Pyrolysis[2]*	*Activation steps*
ICM	50:50 of *Pinus ponderosa: Pinus banksiana*	550° C	2 hours	-
RO	Equal proportions of: *Quercus robur*; *Acer saccharum*; and *Fraxinus americana*,	550° C	22 hours	-
B	Mixed source of *Phyllostachys Aureosulcata*; *Phyllostachys Rubromarginata; Phyllostachys Bissetii;* and *Phyllostachys Aurea*	450-550° C	4 hours	-
MC	*Macadamia integrifolia* (nut shell)	500-550° C	<10 minutes	-
AAC	*Cocos nucifera* (nut shell)	550° C	12 hours	Post-production 1100° C with steam

* Organic matter; 1- Pyrolysis Temperature; 2- Pyrolysis Time;

2.2 Soils and biochars Incubation designs

The first experimental design was set up completely randomized to analyses the biochars incubation isolated in. Therefore, we incubated the biochars (ICM, RO, AAC, B and MC) with 4 replications. For that design, we added 1g biochar in 125 ml serum bottles, and consequently 0.5 ml of water were added per bottle. The bottles were sealed with red butyl rubber septa and left in an open laboratory environment at a controlled temperature of 25 °C for 22 days. The CO_2 and O_2 concentrations were measured on the 1th and the 27th day of incubation, respectively, these days were included in the first and second period of incubation.

The second experimental design was developed to analyze the CO_2 and O_2 concentrations from soils plus biochars incubated together. Therefore, we used the

factorial 3×5+1 with 3 replications represented for three soil (RM, PS and UM) and five biochars (ICM, RO, AAC, B and MC), and the control treatment.

Initially, were added 5 g of soil in 125 ml serum bottles (Diameter of 54 mm; Height of 107 mm), and posteriorly were added 0.1 g of biochar and 0.5 ml of water per bottle. Then, the soil and biochar were manually mixed and sealed with red butyl rubber septa (Grace, Deerfield,IL). After that, the setup was left in an open laboratory environment at a controlled temperature of 25 °C for 57 days. The CO_2 and O_2 concentrations were measured on 1th, 5th, 8th, 13th, 15th, 20th, 27th, 37th, 52th and the 57th day of incubation.

2.3 CO_2 and O_2 concentrations

We used the same methodology to analysis the CO_2 and O_2 concentrations for all the experiment designs. Initially, 5 ml of air (known composition) was injected into the sealed incubation vials using a syringe. To obtain the adequate mixing of the serum bottle headspace the syringe was flushed three times. Posteriorly, 5 ml of headspace was pulled back into the syringe, and it was injected in 10 ml headspace vial previously flushed with helium.

The CO_2 and O_2 headspace concentration were measured using the gas chromatography (Schimadzu GC) adapted to a mass spectrometer (GC-MS). To know more information about this system look Spokas and Reicosky (2009) on.

2.4 Data processing and statistical analysis

Initially, the observed days (1th, 5th, 8th, 13th, 15th, 20th, 27th, 37th, 52th and the 57th day) at the second experimental design were subjected to a multivariate exploratory analysis by hierarchical clustering to identify and expo the group structure contained within the data. For that, the similarity matrix was constructed with the Euclidean distance and the connections of the clusters were conducted by the Ward method (SNEATH; SOKAL, 1973). We used the distance between two groups using the sum of squares between the two groups through all variables (HAIR, 2007). The Euclidean distance among accesses to the set of variables was calculated, distinguishing between the studied factors (observed days).

We noticed with multivariate exploratory analysis results that there were three different kinds of group structure where they were divided at first period (1th and 5th), second (8th, 13th, 15th and 20th) and third period (27th, 37th, 52th and 57th) with similar characteristics and behavior in the group.

The CO_2 and O_2 concentrations rates (soil+biochar experiment) were calculated from the slope of the change in headspace concentration with time from gas samples taken at first, second and third period. Consequentially, the CO_2 and O_2 concentrations and rates from the first and second experiments were analyzed using the descriptive statistics (standard deviation, minimum, maximum and median) and statistically compared by Tukey test at 5% probability.

The relationship between CO_2 and O_2 concentrations was observed using the Pearson correlation. While, the respiratory coefficient - RQ (mol mol^{-1}) was calculated according to Dilly (2011). The RQ results also were analyzed using the descriptive statistics (standard deviation, minimum, maximum and median) and compared by Tukey test at 5% probability (Sisvar Inc., Brasil).

3.0 Results

3.1 CO_2 sorption by biochar

We noticed that the biochars incubated isolated had different reactions to CO_2 and O_2 concentrations and these results depend on the kinds of biochar and period of the time ($p<0.05$), Figure 3.2A and B.

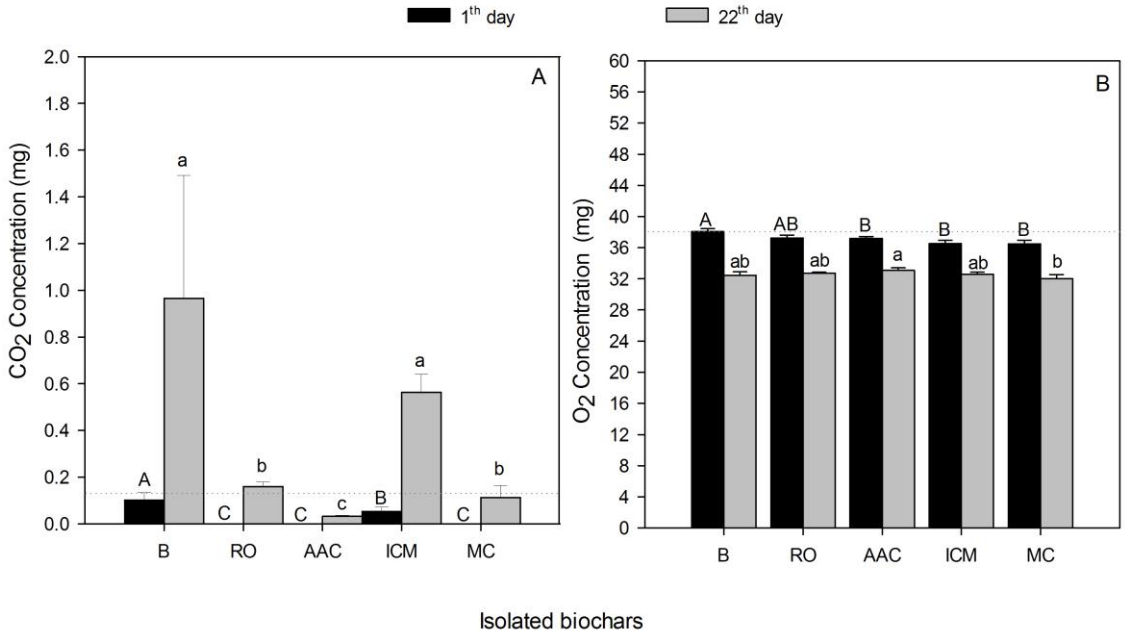

Figure 1. CO_2 and O_2 concentrations from the incubation of isolated biochars (Bamboo – B; Royal Oak hardwood lump charcoal – RO; Accurel activated charcoal – AAC; Pine chip biochar – ICM; and Macadamia nut - MC) on the first (Figure 1A) and second period (Figure 1B). In the figure, a bar with an uppercase letter compare the 1th day and lowercase the 27th day by Tukey Test ($p < 0.05$).

At the first day, the RO, AAC and MC were able to sorption 100% of CO_2 from the atmosphere (700 ppm or 0.15 mg of CO_2) with significant difference to B and ICM

($p<0.05$). The ICM and B also were able to sorption CO_2. However, they absorbed 67.14 and 34.85% of CO_2 from the atmosphere. The O_2 also had difference of concentration with a lower mean decrease of 2.03% from the atmosphere (23% or 37.7 mg of O_2) in this time. Even so, the B had the highest O_2 sorption with significant difference and mean increase of 3.175% to AAC, ICM and MC (Figure 3.2A).

The MC and AAC kept sequestering CO_2 on the 27th day. However, they were able to sequestrate 25 and 75% of CO_2 from the atmosphere, respectively. Moreover, the AAC had the highest O_2 sorption (33.08 mg) while MC was lowest (32.0 mg). The B and ICM became to emit CO_2 to the atmosphere by means of 0.97 and 0.56 mg of CO_2 produced and a decrease of 14.75 and 10.85 O_2 sorption from the first day, respectively (Figure 3.2B).

3.2 CO_2 and O_2 concentrations by biochar+Soil

The temporal variability of CO_2 at soil+biochar experiment had a mean increase of 87.87% for all treatments observed from the first until the last day (Figure 3.3A, B and C). The O_2 had the same behavior, however we noticed the mean decrease of 11.58% at the same period (Figure 3.3D, E and F).

The control treatment showed the lowest total CO_2 concentration in all soils observed by means of 2.43±0.50; 3.40±0.87; and 3.76±0.69 mg to RM, UM and PS, respectively. However, the control treatment had an increase of 85.36; 77.94 and 94.14% from the first until the last day to RM, UM and PS, respectively. The use of biochar in the soil help improving the CO_2 concentration with a mean increase of 87.02; 84.92; 89.07; 82.84; and 90.61% of CO_2 produced when compared with control treatment, respectively, to MC, ICM, B, RO and AAC (Figure 3.3 A, B and C).

The three periods obtained in soil+biochar experiment using the cluster analyses showed different characteristics and behaviors to CO_2 and O_2 concentrations (Figure 3.3). The CO_2 behaviors can be described like the growth, following the transition and stabilization, respectively.

In the first period, we noticed the highest CO_2 and a stability of O_2 concentration. The RM and UM soil had the highest CO_2 concentration with a mean of 0.38 and 0.30 mg CO_2 day^{-1} with significant difference and mean increased of 61.11% to PS in this period (Table 3.2). The same increment was observed to O_2 whereas the PS had the lowest mean (0.55 mg day^{-1}) with significant difference to RM (0.91 mg day^{-1}) and UM (0.86 mg day^{-1}) (Figure 3.3 and Table 3.2).

We also noticed that the PS soil took more time in this period to CO_2 concentration (behavior) with a mean of 12 days while the RM and UM took 5 days of activity time. To biochars, the control treatment had the lowest CO_2 (0.14 mg day^{-1}) while ICM had the highest mean with CO_2 increase of 61.11% compared to control treatment (Figure 3.3 and Table 3.2).

The second period also was characterized for high CO_2 and O_2 concentrations. However, it was 73.72% and 84.43% lower than first period, respectively to them. Moreover, the second period took more 19 days of activities when compared with the first period.

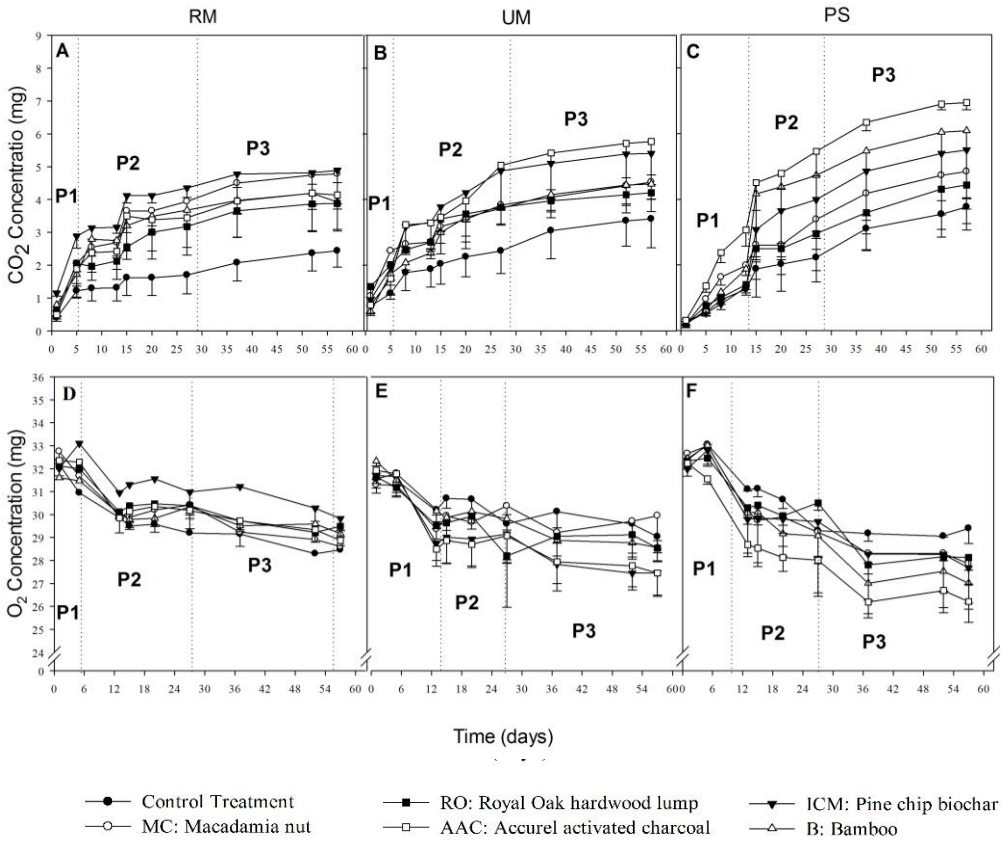

Figure. 3.3. CO_2 (mg) and O_2 concentrations (mg) from soils (RM, PS and UM) with biochars (Macadamia nut - MC, Pine chip biochar - ICM, Royal Oak hardwood lump charcoal - RO, Accurel activated charcoal – AAC, and Bamboo - B) and control treatment (Without biochar) on the first (P1), second (P2) and third period (P3).

In the first period, the PS (0.11 mg CO_2 day^{-1}) had the highest mean of CO_2 with significant difference to RM (0.05 mg CO_2 day^{-1}) and UM (0.07 mg CO_2 day^{-1}). The PS also had the highest mean of O_2 concentration with an increase of 50.0% and 38.88% and significant difference to RM and UM, respectively. Among the biochar, the highest CO_2 happened at soil + B (0.11 mg CO_2 day^{-1}) following the ICM (0.09 mg CO_2 day^{-1}), and MC, RO and ACC with similar mean of 0.07 mg CO_2 day^{-1}. These

biochars had a CO_2 accumulated decrease of 52.29; 53.86 and 74.0% compared to the first period.

The third and last period (from 27 to 57th day) was characterized for stabilization with lower CO_2 and O_2 concentrations (Figure 3.3). We did not difference to notice O_2 between the soils and biochar observed. While, the CO_2 the PS (0.03 mg CO_2 day^{-1}) had the highest mean of the significant difference to RM (0.01 mg CO_2 day^{-1}) and PS (0.01 mg CO_2 day^{-1}), Table 2.

The total accumulated CO_2 was higher than 4.5 mg of CO_2 for all biochars added at the PS soil with a mean of 6.94±0.21 mg CO_2 to AAC, following B (6.08±2.07 mg), ICM (5.50±2.23 mg), MC (4.84±0.79 mg) and RO (4.50±0.74 mg) 6.943±0.21 mg on the 57th day. The CO_2 results higher than 4.5 mg were also observed to ICM (4.88±0.36 mg), MC (4.78±1.69 mg) and ACC (4.13±0.43 mg) at RM, and to AAC (5.76±1.01 mg), ICM (5.40±1.39) and MC (4.53±0.07 mg) at UM (Figure 3.3 A, B and C).

Table 3.2. CO_2 (mg day^{-1}) and O_2 concentrations (mg day^{-1}) from soils (RM, PS and UM) with biochars (Macadamia nut - MC, Pine chip biochar - ICM, Royal Oak hardwood lump charcoal - RO, Accurel activated charcoal – AAC, and Bamboo - B) and control treatment (Without biochar) on 1th, 2th and 3th period of incubation.

	CO_2 concentration (mg day^{-1})			**O_2 concentration (mg day^{-1})**		
	1th Period	2th Period	3th Period	1th Period	2th Period	3th Period
			SOILS			
RM	0.38±0.17Aa	0.05±0.02Bb	0.01±0.00Bc	0.86±0.15Aa	0.09±0.02Bb	0.03±0.01Ac
UM	0.30±0.11Aa	0.07±0.03Bb	0.01±0.00Bc	0.91±0.09Aa	0.11±0.05Bb	0.02±0.03Ac
PS	0.14±0.11Ba	0.11±0.06Aa	0.03±0.00Ab	0.55±0.14Ba	0.18±0.08Ab	0.03±0.04Ac
			BIOCHARS			
Control	0.14±0.05Ba	0.04±0.02Bb	0.02±0.00Ac	0.69±0.29Aa	0.15±0.09Ab	0.01±0.01Ac
MC	0.32±0.13ABa	0.07±0.02ABb	0.02±0.00Ac	0.84±0.14Aa	0.10±0.05Ab	0.03±0.00Ac
ICM	0.36±0.21Aa	0.09±0.05ABb	0.018±0.00ABc	0.72±0.13Aa	0.10±0.04Ab	0.05±0.01Ac
B	0.29±0.23ABa	0.11±0.08Ab	0.019±0.00ABc	0.81±0.19Aa	0.14±0.07Ab	0.03±0.00Ac
RO	0.32±0.12ABa	0.07±0.02ABb	0.01±0.00Bc	0.88±0.12Aa	0.15±0.05Ab	0.01±0.05Ac
AAC	0.30±0.09ABa	0.07±0.05ABb	0.01±0.01Bc	0.87±0.13Aa	0.09±0.07Ab	0.04±0.02Ac

In the table, means with an uppercase letter compare CO_2 and O_2 concentrations per soils and Biochars, and the lowercase letter compares the periods by Tukey Test ($p < 0.05$).

3.3 Relationship between CO_2 and O_2

We noticed that the correlation between CO_2 and O_2 was different of according with kinds of soil and periods observed (Figure 3.4). In the first period, we did not see the correlation between CO_2 and O_2 at RM (r= -0.15; p= 0.39), UM (r= -0.31; p= 0.07) and PS (r: -0.08; p: 0.66), Figure 3.4A, D and G.

On the second period, that correlation also was not significant to RM (r= -0.04; p= 0.66). However, to UM (r= -0.64; p< 0.001) and PS (r= -0.67; p< 0.001) we noticed

a significant and negative correlation between them, Figure 3.4B, E and H. The PS (r=-0.91; p<0.001) and UM soil (r=-0.77; p<0.001) kept a negative and significant correlation on the third period, as well as RM did not have correlation between them. We can also observe that the correlation is strong in the third period, compared to other periods (Figure 3.4C, F and I).

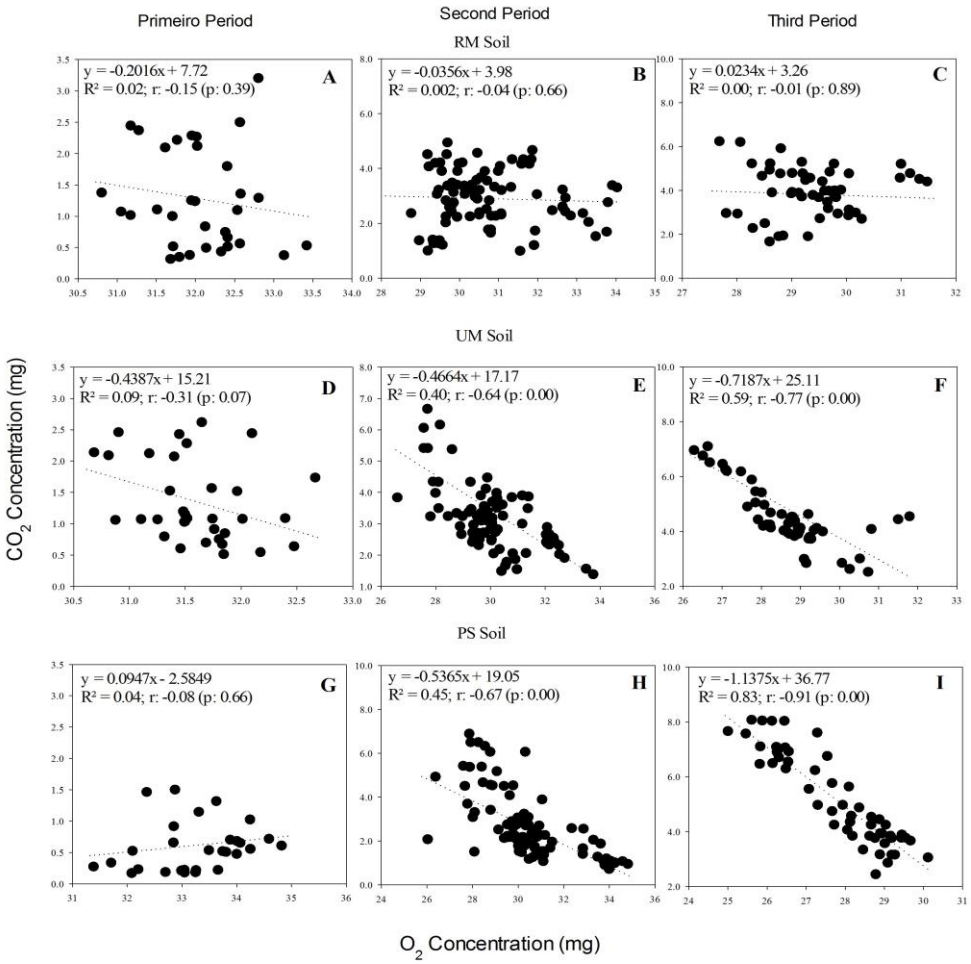

Figure 3.4. Relationship between CO_2 (mg) and O_2 concentration (mg) at soils (RM, PS and UM) incubated with biochars (Macadamia nut - MC, Pine chip biochar - ICM, Royal Oak hardwood lump charcoal - RO, Accurel activated charcoal – AAC, and Bamboo - B) and control treatment (Without biochar) on 1th, 2th and 3th period of incubation

The RQ values were frequently lower than 1 to both experiment design soil+Biochar and Isolated Biochar with RQ total mean of 0.36 and 0.05 moles moles^{-1}, respectively (Figure 3.5). The soil without biochar (control treatment) had the lowest values of RQ at all soils and periods observed by means of 0.13±0.03; 0.23±0.12 and 0.60±0.07 moles moles^{-1} on the first, second and third period respectively (Figure 3.5 A, B and C). While, to soil + biochar experiment the first

period had the lowest RQ values (0.24±0.13 moles moles^{-1}), following the third and second period (Figure 3.5).

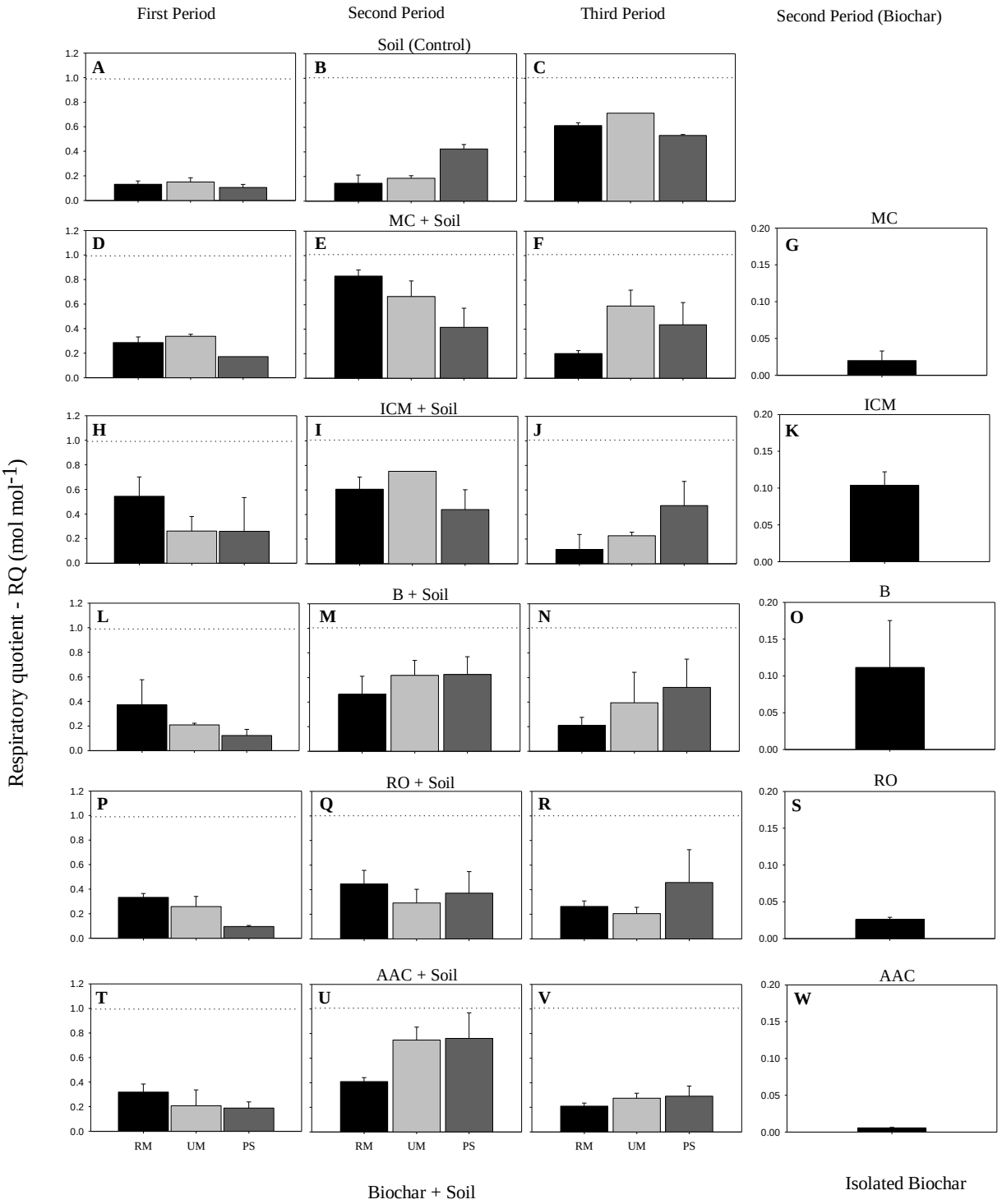

Figure 3.5. Respiratory Quotient (RQ) at isolated biochars design and biochar+soil design with different kinds of soils (RM, UM and PS) and biochars (Macadamia nut - MC, Pine chip biochar - ICM, Royal Oak hardwood lump charcoal - RO, Accurel activated charcoal – AAC, and Bamboo – B) incubated on first, second and third period of incubation.

When added the biochar in the soil, we can see a different behavior. The highest RQ values happened on the second period by means of 0.60±0.20; 0.57±0.13; 0.56±0.13; 0.36±0.12 and 0.65±0.18 mol mol^{-1} to MC, ICM, B, RO and AAC, respectively (Figure 3.5E, I, M, Q and U), with an RQ increase mean of 58.18% and 90.62% compared to soil without biochar (Figure 3.5A, B and C) and isolated biochars (Figure 3.5G, K, O, S and W).

The much lower RQ value in the isolated biochars experiment is linked with higher O_2 available and lower CO_2 available, without significant correlation between them (r= -0.14; p= 0.57). So, the lowest RQ value was noticed at AAC (0.005 mol mol^{-1}), following MC (0.05 mol mol^{-1}) and RO (0.02 mol mol^{-1}), Figure 3.5G, S and W. While, the B and ICM had the higher RQ values with mean increase mean of 74.8 and 70.11% to them (Figure 3.5 O and K).

4.0 Discussion

4.1 Isolated biochar (CO_2 and O_2 sorption by biochar)

The ability to sorption CO_2 from the atmosphere by the RO, AAC and MC (100% sequestered) and ICM and Bamboo (67.14 and 34.85% sequestrated) is a great result in our experiment. We were able to notice that the biochar can sequester the carbon from the atmosphere by biochar sorption. In fact, this ability depends on the kind of biochar observed and time of incubation with better results to MC, RO and AAC. While, the ICM and B sorption had the lower ability and they began emitting CO_2 on the 22 days after the incubation.

The MC may contain a different aromatic structure (DEENIK et al., 2011), as well as AAC and RO. So, biochar can have a better ability to sorption CO_2 from the atmosphere when has a higher carbon distribution (KEILUWEIT et al., 2010), the low O/C ratios (GLASER et al., 2002; KIMETU; LEHMANN, 2010), and contain oxygen-containing functional groups on biochars, such as: hydroxyl (–OH) and carboxylate (–COOH) (CHUN et al., 2004; YUAN et al., 2011) and a net negative charge on their surfaces due to the dissociation of oxygen-containing functional groups (INYANG et al., 2010; YUAN et al., 2011). Moreover, the higher presence of micropores (smaller than 0.5 nm) and surface area values can improve this ability (YARGICOGLU et al., 2015).

The high biochar microporosity can be obtained using the activation treatment and temperature (BROWN et al., 2006) how used to make the AAC in this

experiment. That biochar activation process can obtain during the cooling using the water or exposing the hot biochar to atmospheric oxygen (PURI et al., 1958; CHENG et al., 2006). Consequently, this activation process alters the surface chemistry at biochar (AZARGOHAR; DALAI, 2006; NUITHITIKUL et al., 2010), favoring the biological activities and CO_2 sorption.

We can also notice that happen the O_2 sorption by biochar process. However, this process did not have correlation with CO_2 sorption and with RQ values (RQ<1) so many lower than 1. These results showed an imbalance between the CO_2 and O_2 concentration as a result of absence of biological activities and probably the predominance of chemical reactions. The RQ has been used as criteria of the soil microbial activity (DILLY, 2011) and according of Angert et al. (2015) the variations in the CO_2 /O_2 concentration in soil profiles can be a result of soil–gas exchange fluxes and biological respiration.

The use of biochar in soil has also been related to adsorb the NO_3^- (DEMPSTER et al., 2012; CLOUGH et al., 2013), retention of fungicides as tricyclazole (GARCÍA-JARAMILLO et al., 2015), stabilizing heavy metals (WULFSBERG, 2000; UCHIMIYA et al., 2011; HIGASHIKAWA et al., 2016) and soil organic pollutants (CHEN; CHEN, 2009; YU et al., 2010). However, there is limited information associated underlying mechanisms (CHEN et al., 2011) and CO_2 and O_2 sorption.

4.2 Biochar+soil (CO_2 and O_2 concentrations)

The higher CO_2 concentration in soil with biochars added has also been observed, for some researcher, such as: Major et al. (2009), Zimmerman et al. (2011), Lehmann et al. (2011), Lentz et al. (2014) and Lin et al. (2014) when compared with control treatment.

This happened because the use of biochar in soil has been correlated with an increase the activity, soil microbiological and enzymatic (LEHMANN et al., 2011), and the carbon degradation by microorganisms (LEHMANN et al., 2011; HARRIS et al., 2013; AGEGNEHU et al., 2015). Moreover, the biochar can also provide habitat for soil microbes (PIETIKAINEN et al., 2000), and increase organic carbon (PENDERGAST-MILLER et al., 2011; LENTZ et al., 2014), nitrogen (PARK et al., 2011; LENTZ et al., 2014; AGEGNEHU et al., 2015) and other soil nutrients, such as phosphorus (LAIRD et al., 2010; AGEGNEHU et al., 2015), potassium, magnesium and calcium (LAIRD et al., 2010).

Some physical attributes also can be changed when adds biochar in soil, and they can improve the activity soil microbiological and enzymatic. The soil aeration (KINNEY et al., 2012) and bulk density (OGUNTUNDE et al., 2008; JONES et al., 2010; PEAKE et al., 2014) are some examples. Thomazini et al. (2015) noticed the increase of these attributes with biochar added in different kinds of soil from Minnesota, Wisconsin, California, Florida, South Carolina, Idaho, Illinois, Michigan and Pennsylvania.

Moreover, the biochar has the ability to create an additional external soil porosity, and it does not alter the packing of soil particles (LIMM et al., 2016). This higher porosity happens with biochar fragmentation, and it normally happens by microbial activities (SIGUA et al., 2014) or the biochar can disintegrate themselves naturally (SPOKAS et al., 2014; NAISSE et al., 2015). This fragmentations occur more readily in the sandy soils and less evident on the silty (PEAKE et al., 2014; SPOKAS et al., 2014). Moreover, the biochar particle size is not constant (SPOKAS et al., 2014), and as a result alters soil physical properties (SPOKAS et al., 2009) favorably in agricultural productivity (PEAKE et al., 2014).

We also noticed that the CO_2 concentration difference between the biochars and soils used in this experiment. Novak and Busscher (2009), Sigua et al. (2014), Agegnehu et al. (2015), García-Jaramillo et al (2015) also observed similar results.

The highest CO_2 total accumulated when added AAC, B and ICM at PS soil, ICM and at RM, and ACC and ICM at UM happened because of the feedstock and pyrolysis to theses biochars conditions (GARCÍA-JARAMILLO et al., 2015), and this difference is commonly found between different kinds of biochars (JASSAL et al., 2015; SIGUA et al., 2016). The ability to CO_2 sorption on AAC, MC and RO are not enough to keep the lower CO_2 concentration when added biochar in soil, mainly in PS soil. While, the highest total accumulated in B and ICM is perfectly explained by lower ability to CO_2 sorption how we noticed in the biochar isolated experiment.

The similar MC results (low CO_2 concentration) have been noticed by Harris et al. (2013) and Lin et al (2014), however, they did not explain this result as a consequence of CO_2 sorption. Lin et al. (2014) explained that lower CO_2 concentration in soil with MC is correlated with soil microbial biomass (SMB), and SMB reductions (from 30% to 37%) in three different kinds of soils (Prairie, Agricultural and Forest Soil).

5.0 Conclusion

The RO, AAC and MC are able to sorption 100% of CO_2 from the atmosphere. The O_2 can also sorption by biochar without large difference between the biochars studied.

There is a CO_2 increase of 87.87% when added all biochars tested in soil while to the O_2 happen a decrease of 11.58% on the 57th day of incubation.

The biochars show different behaviors at soil and isolated experiment. More study should be developed to elucidate the CO_2 and O_2 sorption by biochar and their reactions in soil.

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4. CHAPTER 4 – GENERAL CONCLUSIONS

We can conclude that the concentration and relationship between CO_2 and O_2 depend on different systems and soil conditions, for example: soil, crop residue managements, soil moisture and use of biochar.

The FO_2 is positively correlated with CO_2 at biological condition with RQ values close to 1.0. Moreover, we can notice that RQ values higher than 1 are results of soil–gas exchange fluxes after precipitation or higher O_2 available. Thus, the RQ and FO_2 can be used as an index for categorizing the CO_2 respiration.

The biochar can be used to sequester CO_2 from the atmosphere on short time. We did not know how the biochar is able to sequester CO_2 and which process (biological and/or chemical) the biochar uses.

We believe that more study should be developed to elucidate the CO_2 and O_2 relation in soil with different uses and managements, such as: sugarcane and biochar added. The use of sensor to analyze CO_2 emission and O_2 uptake should be tested in the laboratory first to choose the best methodology and experimental design.