DO PRESCRIBED BURNS YIELD ENHANCED CARBON STORAGE IN SALT MARSHES? A COMPARATIVE ASSESSMENT IN DELAWARE SALT MARSHES WITH VARIED BURN HISTORIES

by

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A thesis submitted to the Faculty of the University of Delaware in partial fulfillment of the requirements for the degree of Master of Science in Marine Studies

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ABSTRACT

Salt marshes and other tidal wetland ecosystems play crucial roles in the environment and provide a suite of ecosystem services including erosion control, providing breeding and nursery habitat for birds and fisheries species, and improvement of water quality. In the context of carbon cycling and climate change, tidal wetlands are of recent increased interest for a carbon sequestration ecosystem service. It has been estimated that these environments can sequester up to 244.7 ± 26.1 g C m^{-2} yr^{-1}. Tidal marsh habitat has also been lost at alarming rates over recent centuries due in part to the invasion of *Phragmites australis*. *Phragmites* invasions have been associated with altered hydrology and reductions in native biodiversity in Delaware marshes and are frequent targets for restorations to reestablish native marsh habitat. Several tactics are commonly used by land managers for the removal of *Phragmites* such as mowing, application of herbicides, and prescribed burns. *Phragmites* may actually store organic carbon more efficiently than native species meaning its removal may unintentionally result in reduced carbon sequestration. Prescribed burns, however, are known to produce biochar, a recalcitrant form of organic carbon that is efficiently buried in soils and may recoup some of the lost carbon sequestration. With this thesis, I utilize core collections at three tidal marsh sites for the purpose of studying whether prescribed burns improve carbon storage in tidal Delaware marshes: a site with a known burn history (Roberts Farm), an adjacent site with no recent (<20 years) burn history (Rocks Tract), and a site with no history of burns (St. Jones). Cores (15 cm, n=10) were collected from each site and analyzed for total organic carbon (TOC) and Black Carbon (BC), the incompletely combusted remains of biomass burning.

Two methodologies were utilized for BC analysis, the Benzene Polycarboxylic Acid (BPCA; BC_{BPCA}) and Chemo-Thermal Oxidation at 375º (CTO-375; BC_{CTO}) methods in order to assess the implications of methodology on BC quantification. The BPCA method generally reported a higher concentration of BC (mean = 4.97 ± 1.06 BC_{BPCA} g^{-1} soil) compared to the CTO-375 method (mean = 1.84 ± 1.19 mg BC_{CTO} g^{-1} soil) as has been found for terrestrial soil but in contrast to findings for marine sediments. Charring was observed in our organic-rich marsh soils using the CTO-375 method artificially inflating the BC_{CTO} results in the St. Jones marsh in particular. A dilution test was conducted on a subset of samples using blank sand and found an average decrease in BC_{CTO} of 1.6 ± 0.9 mg BC_{CTO} g^{-1} soil. Future studies using BC_{CTO} are encouraged to evaluate their data for potential charring by performing such dilution quality assurance procedures.

The Roberts Farm marsh showed higher TOC (58.4 ± 14.4 mg TOC g^{-1} soil) and BC_{BPCA} (5.80 ± 1.06 mg BC_{BPCA} g^{-1} soil) concentrations than the adjacent unburned Rocks Tract marsh (47.2 ± 12.1 mg TOC g^{-1} soil; 4.76 ± 0.77 mg BC_{BPCA} g^{-1} soil) suggesting that recent burns may have increased carbon storage in the Roberts Farm soils. Using the BC_{CTO} method, however, Roberts Farm BC_{CTO} concentrations (1.44 ± 0.26 mg BC_{CTO} g^{-1} soil) were similar to Rocks Tract (1.31 ± 0.57 mg BC_{CTO} g^{-1} soil) indicating that the soot-like portion of the BC pool to be similar at the two sites, and char materials may be contributing the differences observed in the BC_{BPCA} data. TOC concentrations at the mesohaline St. Jones (81.2 ± 44.2 mg TOC g^{-1} soil) site were higher than at Roberts Farm and Rocks Tract demonstrating alternate controls on C storage than the presence or absence of burning. BC_{BPCA} B6:B5 ratios were similar at the Roberts Farm and Rocks Tract marshes suggesting similar BC sources, and I

speculate that the BC derived from the prescribed burns at Roberts Farm is transported atmospherically and hydrologically to the Rocks Tract marsh. All three marshes have measurable BC in their soils reflecting its ubiquitous delivery from sources other than the prescribed burns.

This thesis demonstrates the utility of using multiple BC characterization methods for studying its quantities and characteristics and provides insight into the impacts of prescribed burns on soil C storage. The data suggest a potential C sequestration benefit from these prescribed burns. Additional studies are needed to assess the ubiquity of this benefit as well as to compare it quantitatively to C storage in *Phragmites* soils. Tidal marsh managers can use the C storage information found in this work within their salt marsh restoration decision-making strategies.

Chapter 1

INTRODUCTION

1.1 Salt Marsh Environments

Tidal marshes, hereafter referred to as salt marshes, represent critical habitats for a diverse array of organisms whose composition varies both within and across sites (Fischer et al., 2000). Acting as a transitional zone between terrestrial and aquatic ecosystems, salt marshes provide refuge, feeding grounds, and spawning areas for a wide range of organisms, including fish and crustaceans (Deegan et al., 2000). Across sites, variations in geologic and chemical properties, such as geomorphology, salinity, and tidal regime, result in species composition shifts and vegetation zonation, affecting the functioning of these ecosystems (Fischer et al., 2000; Silvestri et al., 2005; Moffett et al., 2010). Alterations in the abiotic properties and the local vegetation also influence the presence and abundance of avian populations at these sites. Salt marshes serve as crucial areas for nesting, feeding, refuge, and migration for a variety of bird species, including shorebirds, perching birds, wading birds, and gulls and terns (Craig and Beal, 1992; Darnell and Smith, 2004; Teixeira et al., 2014).

Beyond the critical habitat provided for organisms, there are a suite of economic and recreational services that salt marshes generate. These services are of significant interest to policy makers and researchers as they are often how the public interacts with these wetlands. Salt marshes offer direct services to the public through consumptive uses such as fisheries and water supply, as well as non-consumptive uses like recreation, transportation, aesthetics, and more (Barbier et al., 2011). According to

Antoni and Scarborough (2017), a survey conducted in 2015 revealed that 68% of Delaware residents interacted recreationally with salt marshes. Residents engaged in activities such as fishing/shellfishing, hiking, birdwatching, kayaking, and hunting, among others. The survey also estimated that DE residents spent a substantial amount of money while utilizing saltmarshes, including $38 million on fishing, $15 million on wildlife viewing, and $9 million on hunting. Despite these significant benefits, the survey indicated that residents were more aware of direct, tangible services and less cognizant of services such as water quality, coastal erosion, and carbon storage. This may be due to lack of public awareness and media attention compared to other coastal ecosystems (Duarte et al., 2008). Still, these less visible ecosystem services generate a large portion of the estimated value of these sites (Barbier et al., 2011; Kauffman, 2016; Kutcher et al., 2018).

Salt marsh ecosystems also provide an array of physical and chemical services that are critical to both ecological and human health. In terms of physical ecosystem services, salt marshes serve as a natural defense against storm surges by attenuating wave energy during extreme weather events, thereby protecting nearby infrastructure (Barbier et al., 2011; Shepard et al., 2011; Gittman et al., 2014). Moreover, saltmarsh vegetation has been found to have a positive effect on shoreline stabilization and reduction of lateral erosion (Broome et al., 1988; Shepard et al., 2011). Soil accretion within marshes is a key component in maintaining coastal resilience in the face of sea level rise (Reed, 1990; Craft et al., 2008; Schuerch et al., 2013; Weston et al., 2023). However, there is evidence that sea level rise is occurring at a rate that may outpace marsh accretion (Crosby et al., 2016), which would result in marsh migration and loss. Salt marshes also play a significant role in local biogeochemistry by facilitating

complex and valuable nutrient fluxes (Tobias and Neubauer, 2019). The cycling of nitrogen, phosphorus, and sulfur through these ecosystems is vital for supporting trophic interactions, soil accretion, and water purification by stimulating primary production (Vernberg, 1993). Marshes effectively remove nutrients from surface water via processes including denitrification (Caffrey et al. 2007), and filter pollutants and soil originating from surrounding landscapes (Bertness, 1999). Carbon cycling and its role in salt marsh ecosystem services are of particular interest to researchers.

Of particular interest to this thesis, salt marshes are increasingly recognized as providing a valuable carbon sequestration ecosystem service because of their ability to store carbon in biomass and soils (Chmura et al., 2003; Mcleod et al., 2011; McOwen et al., 2017). The high primary productivity in salt marshes yields correspondingly high rates of nitrogen and carbon incorporation into salt marsh biomass and soils (Vernberg, 1993). Primary productivity in *Spartina alterniflora* marshes, ranging from 100-2500 g C m^{-2} $year^{-1}$, has been documented, as have comparable rates for other species found in salt marshes, such as *Spartina patens* (669-1147 g C m^{-2} $year^{-1}$) and *Distichlis spicata* (63-785g C m^{-2} $year^{-1}$) (Gabriel and Cruz, 1974; Roman and Daiber, 1984). These high rates of primary productivity result in substantial quantities of carbon stored in biomass and salt marsh soils, rendering salt marshes valuable "blue carbon" ecosystems for reducing atmospheric carbon dioxide concentrations. The burial of carbon and nitrogen in salt marsh soils can significantly reduce the quantity released from the marsh to the atmosphere and other ecosystems. It is estimated that salt marshes cover an area of 22,000-400,000 km^2 globally and bury 0.5-87.2 Tg C yr^{-1} (Chmura et al., 2003; Mcleod et al., 2011; McOwen et al., 2017). This is noteworthy when compared to temperate forests, which cover an area of 10,400,000 km^2 and bury

53.0 Tg C yr^{-1} (Mcleod et al., 2011). Despite covering a smaller land area, salt marshes bury carbon at significantly higher rates than terrestrial ecosystems (218 ± 24 g C m^{-2} yr^{-1} for salt marshes and 5.1 ± 1.0 g C m^{-2} yr^{-1} for temperate forests).

Managing salt marshes to maximize blue carbon storage presents a complex challenge, as managers must balance this ecosystem service with the full range of services that the marsh provides in the context of stakeholder interests. A range of restoration activities offer both benefits and limitations with respect to potential restoration goals, with economic considerations being a crucial factor. With limited funding available for salt marsh projects, local, state, and federal officials must make informed decisions regarding which ecosystem services they consider the most important. Prioritizing among potentially conflicting goals can be a challenging aspect of management, and marsh management strategies will vary depending on stakeholder priorities (Gedan et al., 2009; Adam, 2019).

Phragmites australis, an invasive common reed originally from Eurasia, has rapidly spread throughout wetlands in North America (Hazelton et al., 2014) and is a frequent target for removal and replacement with native vegetation with salt marsh restoration efforts. In the Delaware estuary, the initial establishment of *Phragmites* dates back to the 1950s (Philipp and Field, 2005). Since its introduction, *Phragmites* has continued to encroach upon salt marshes along the East Coast of the United States, leading to declines in native vegetation, degradation of habitats for native wildlife, and hydrologic alteration (Marks et al., 1994; Benoit and Askins, 1999; Chambers et al., 1999; Kimball and Able, 2007). Managers have employed various methods to remove *Phragmites* and restore native vegetation including mowing or using cattle to graze the *Phragmites* (Tesauro and Ehrenfeld, 2007; Brundage, 2010). The most widespread

practice for the removal of the species is the application of herbicides (Hazelton et al, 2014). In Delaware, herbicides have been used in conjunction with prescribed burns to remove non-native species, although data on the effectiveness of prescribed burns for restoring native vegetation and other ecosystem services are sparse.

Despite the clear negatives posed by *Phragmites* invasions, it offers biogeochemical benefits by storing more carbon in its biomass and soils and increasing denitrification relative to native vegetation with associated benefits for climate and water quality (Duke et al., 2015; Gu et al., 2020; Yacano et al., 2022). Therefore, the removal of *Phragmites* may result in a loss of carbon and nitrogen ecosystem services. However, prescribed burns may yield additional biogeochemical services in the form of black carbon (BC), a material of interest to researchers and land managers due to its unique physicochemical properties.

1.2 Black Carbon & the Environment

Black carbon (BC) is derived primarily from the incomplete combustion of biomass, wood, and fossil fuels, resulting in pyrolyzed organic matter (Goldberg, 1985; Jones and Chaloner, 1991; Coppola and Druffel, 2016). It exists along a combustion continuum, ranging from charcoals to soot-like and graphitic material, and is predominantly composed of condensed polycyclic aromatic compounds (Hedges et al., 2000; Masiello, 2004). The diverse range of BC products and location along the combustion continuum can be attributed to factors such as formation temperatures, burn duration, fuel material, and oxygen availability (Keiluweit et al., 2010; Wozniak et al., 2020).

Black carbon (BC) exhibits a wide range of physicochemical properties due to its existence along this combustion continuum. These properties influence its ease of

transport from terrestrial to marine environments through aquatic or atmospheric pathways (Schmidt and Noack, 2000). The global storage and flux dynamics of BC are of significant interest to researchers due to its implications for the carbon cycle and climate change (Coppola et al., 2022). Geochemists are particularly intrigued by the storage of BC because of its apparent refractory nature in the environment (Hedges et al., 2000; Masiello, 2004). This refractory nature, characterized by its resistance to biological and chemical conversion to carbon dioxide, is likely linked to its aromatic structure or physical protection from binding with minerals and organic compounds (Hammes et al., 2007). Moreover, the condensed aromatic structure of BC contributes to its resistance to degradation by microbes (Schmidt et al., 2011).

Overall, BC's recalcitrant nature classifies it as part of the slow carbon cycle, with residence times spanning hundreds to thousands of years (Schmidt and Noack, 2000; Masiello, 2004; Hammes et al., 2007; Coppola and Druffel, 2016). This extended residence time in soils provides a climate benefit by adding recalcitrant carbon to soils thereby sequestering carbon from the atmosphere. Leonard et al. (2010) also observed that specific burning regimes in Chesapeake Bay tidal marshes led to increased above and below-ground vegetation growth. Furthermore, BC may sorb and stabilize dissolved organic matter components cycling in salt marshes, preventing their remineralization or export to the estuary (Lian and Xing, 2017), thus ensuring their preservation over climate-relevant timescales (> 100 years). Consequently, BC deposited from prescribed burns can potentially sequester carbon from the atmosphere through multiple mechanisms and serve as a valuable ecosystem service offered by salt marshes during the restoration process (Coppola et al., 2022), an idea that has

been acknowledged for burns in terrestrial forests (Myers-Pigg, 2016; Coppola et al., 2022) but has not been fully explored in salt marshes.

The quantification of black carbon (BC) presents a challenge due to its inherent heterogeneity (Schmidt et al., 2001). Consequently, various methods have been developed to measure different components of the BC pool, each carrying its own set of biases and none of which characterizes the full spectrum of the BC pool quantitatively (Masiello, 2004; Hammes et al., 2007). When utilizing these BC methods, it is crucial to fully comprehend which portion of the BC pool is being quantified to account for potential positive artifacts as well as to understand portions of the BC pool that go unquantified or uncharacterized in a measurement (negative artifacts). Several non-pyrogenic sources of interference, such as humic acids, lignin, and carbonaceous rocks like coal, have been shown to produce false positives for BC (Masiello, 2004; Hammes et al., 2007; Bostick et al., 2018; Gerke, 2018). Furthermore, the choice of methodology often depends on the specific environment and medium of interest. For instance, optical methods may be employed for atmospheric BC studies, whereas the benzene polycarboxylic acid (BPCA) or chemo-thermal oxidation method (CTO) may be more suitable for open ocean or coastal soil-related investigations (Hammes et al., 2007; Coppola et al., 2022). Because of the various drawbacks of individual methods, it is valuable to utilize complementary measurements to gain a fuller picture of the sources, quantities, and character of BC.

1.3 Research Goals & Hypotheses

The primary objective of this research is to enhance our comprehension of how prescribed burn history influences the storage of total organic carbon (TOC) and BC in salt marsh soils, specifically comparing sites with known burn histories to those

without burn histories. Additionally, we aim to evaluate how the use of different BC quantification methodologies impacts our evaluations of the quantity and quality of BC in the soil. To accomplish these goals, we collected and sectioned 10 cores (3 sections per core) from each of three marsh field locations with varying burn histories. These sites were assessed for their TOC content and their BC content using two analytical methods, namely BPCA and CTO-375, to gather data on the overall concentrations of BC in the soil as well as the degree of condensation in BC aromatic compounds.

I hypothesize that sites with recent burn histories will exhibit higher BC and TOC storage in their soils. I further hypothesize that the two BC methodologies will yield different BC concentrations, as the BPCA method reports a larger proportion of the BC pool compared to the CTO-375 method (Maisello, 2004). Finally, the BC in soils with recent and more extensive burn histories are expected to show BPCA ratios reflecting more condensed aromatic compounds compared to unburned salt marshes.

The outcomes of this study will be relevant to land managers interested in the various ecosystem services offered by salt marshes, especially carbon sequestration. Additionally, biogeochemists investigating the application of BC analytical methods on salt marsh soils will find these results valuable.

Chapter 2

METHODS

2.1 Site Description

For the purposes of this study, three sampling locations were selected based on their varied burn histories. These sites are: 1) St. Jones Reserve marsh in Dover, DE (Figure 1b) and two salt marsh tracts located adjacent to one another along Blackbird Creek in Townsend, DE: 2) Roberts farm (Fig. 1a), and 3) Rocks Tract (Fig. 1a).

The St. Jones marsh is located along the St. Jones River at approximately 39°05'17.0"N, 75°26'11.6"W and is situated within a National Estuarine Research Reserve. The site is a mesohaline salt marsh with an average salinity, calculated from the Scotton Landing water quality station, of 10.8 ± 7.0 (Central Data Management Office 1995-2023 (https://cdmo.baruch.sc.edu//); McKenna et al., 2018). The site's mean elevation is 0.909 ± 0.108 m, and it has a tidal range mean of 1.5 m with a mean spring tide range of 1.7 m (DNERR Estuarine Profile, 1999). The dominant vegetation is *Spartina alterniflora*, occupying approximately 62% of the reserve site, with other present species including *Spartina paten*, *Spartina cyanosuriodes*, *Distichlis spicata*, and *P. australis* (DNERR Estuarine Profile, 1999; St. Laurent et al., 2020). The St. Jones site has no history of prescribed burns and acts as a control compared to the other two sampling locations.

The two remaining sites, Roberts Farm and Rocks Tract, are situated along Blackbird Creek near Townsend, DE at approximately 39°25'55.2"N, 75°36'06.7"W situated in between the Blackbird Creek Reserve and the Appoquinimink Wildlife Area. The two sites are adjacent to one another separated by a boat launch and constitute an oligohaline marsh with an average salinity, calculated from the Blackbird

Landing water quality station, of 2.0 ± 2.1 and a range of 0.0-10.0 (Central Data Management Office 1995-2023 (https://cdmo.baruch.sc.edu//); McKenna et al., 2018). Roberts Farm has an average elevation of 0.658 ± 0.185 m while Rocks Tract has an average elevation of 0.820 ± 0.409 m. The mean tidal range for the Lower Blackbird Creek is 1.7 m with the average spring tide range being 1.92 m. *S. alterniflora* is also the most abundant vegetation at Roberts Farm and Rocks Tract but covers only 29% of the Blackbird Reserve. There is a more diverse array of vegetation species present at these sites reflecting the lower salinity. While there are species that were present at St. Jones, such as *S. patens, D. Spicata,* and *P. australis*; There are also numerous tidal freshwater species such as *Hibiscus palustris, Cephalanthus occidentalis, Pontederia cordata, Peltandra virignica, Nuphar lutea,* and *Polygonum hydropiper*. The burn history of Rocks Tract is unknown, but no burn has been conducted over the past several years. The recent burn history of Roberts Farm is known with the location of several burns dating back to 2017 recorded in Fig. 2 (H. Small, personal communication).

a)

b)

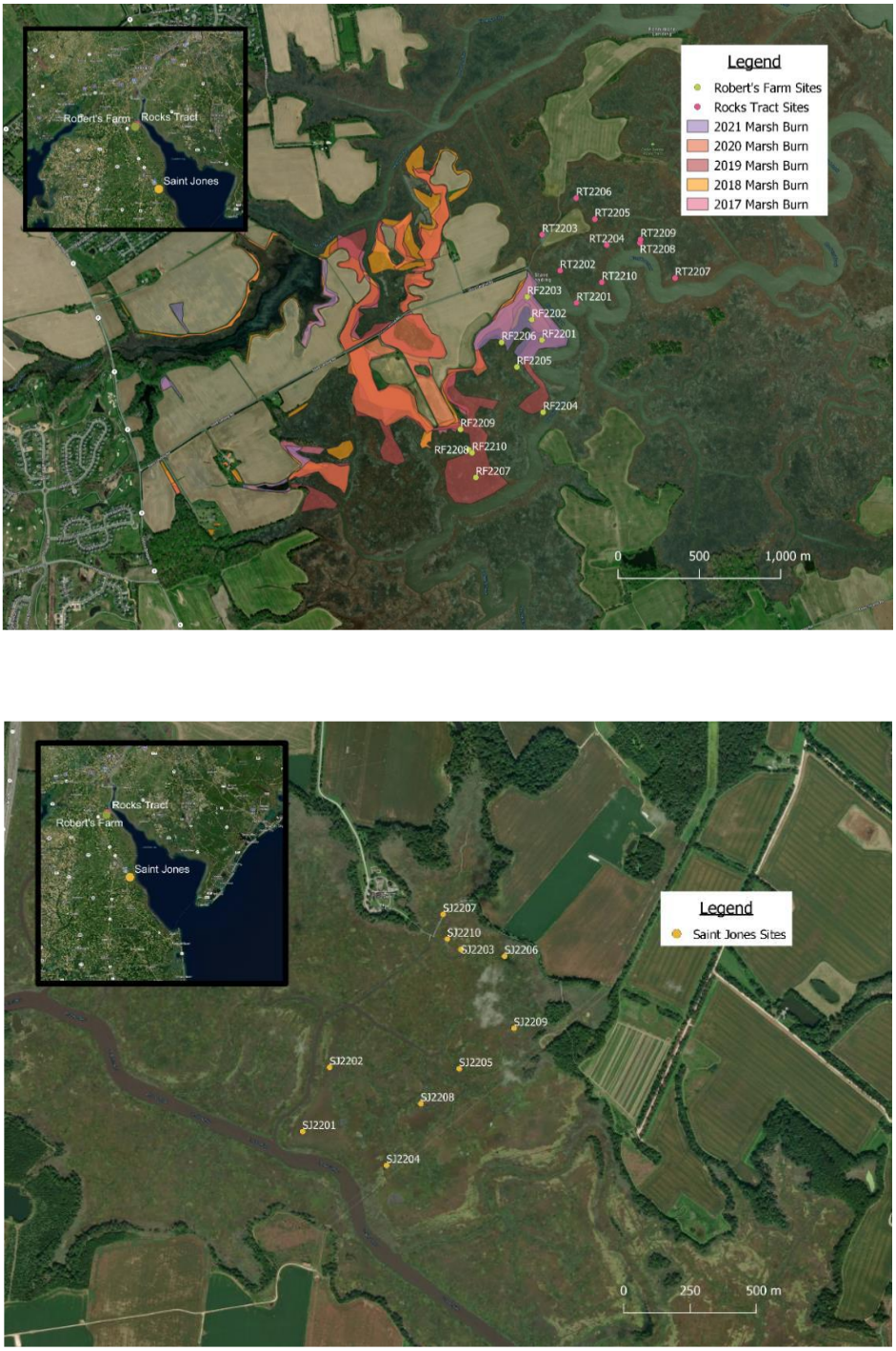

Figure 1: Map showing the sampling locations for a) the Roberts farm and Rocks Tract sampling sites and b) the St. Jones Reserve site. The locations of burns conducted in Roberts Farm between the years 2017-2021 are denoted by the shaded regions in panel a.

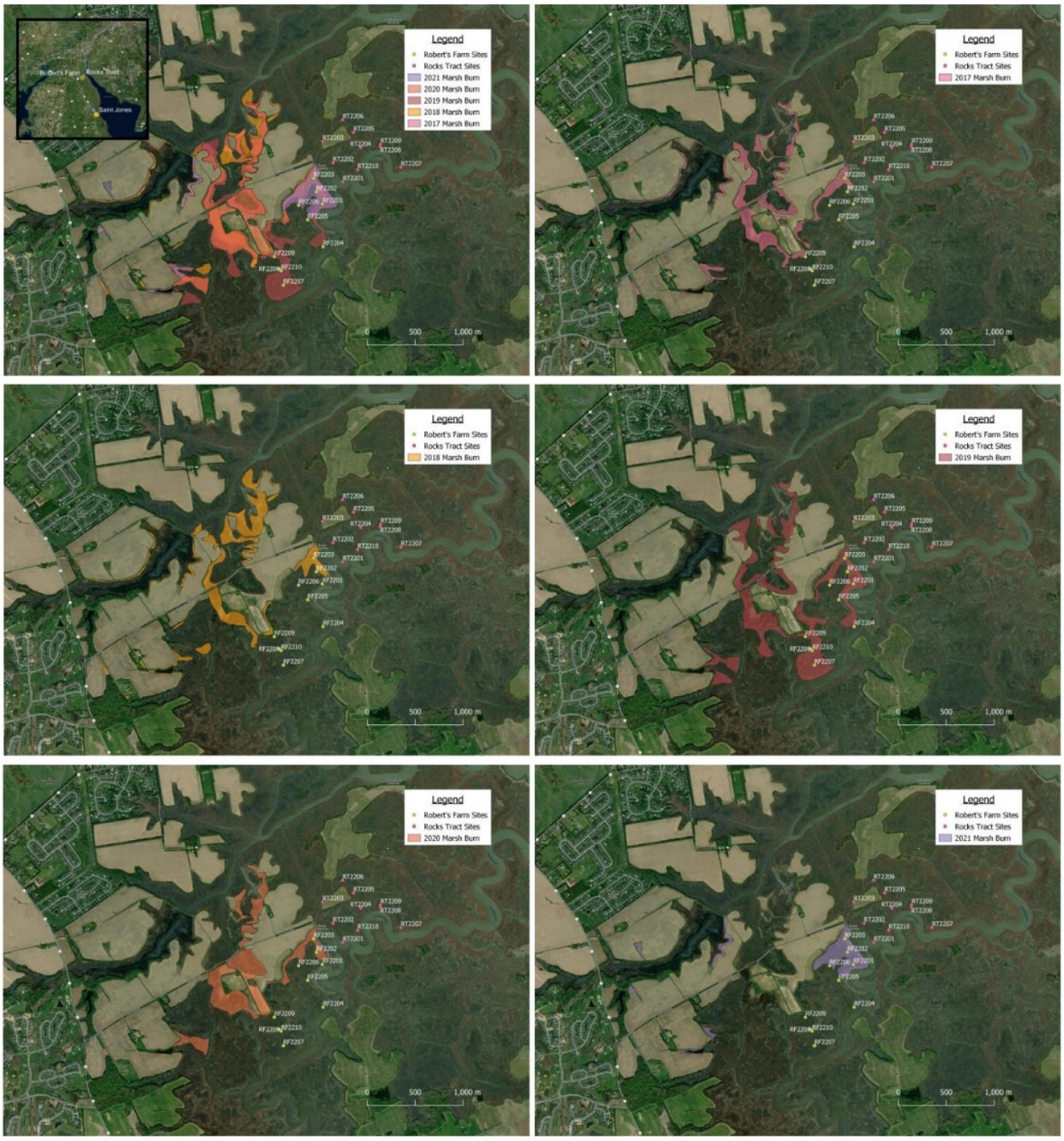

Figure 2: History of prescribed burns at Roberts farm between the years 2017-2021 broken down by year. The locations of sampling sites are denoted by the dots and sample labels.

2.2 Sample Collection

Cores (n=10, 10 cm diameter, 30 cm length) were collected from each site utilizing a push pull method for extraction. The length of the core was chosen due to the history of burns spanning back only a few years and accounting for accretion rates at each location (Tucker, 2016). Specifically, accretion rates for St. Jones ranged between 0.43-1.10 cm yr^{-1}, and for Blackbird Creek, an area near to Roberts Farm and Rocks Tract, it ranged from 0.38-0.56 cm yr^{-1}. This accretion rate would cover the number of known fires that have occurred at Roberts Farm. Nine sampling sites were selected within each location with the purpose of creating three 3-core transects leading away from the major creek (Blackbird creek for Roberts Farm and Rocks Tract and the St. Jones River for St. Jones) at each site. The tenth sampling site was reserved for an area of the study site dominated by *P. australis*.

Marshes were accessed by foot or boat over the course of several days. The collection dates and access were determined by site location, weather, tidal cycle, and hunting restrictions. The goal was to collect samples primarily during low tide to remain consistent and provide ease of access. Gaia GPS application was utilized to locate the proposed site coordinates, and the actual site coordinates, which may have changed slightly due to on the ground marsh conditions (e.g., unexpected marsh hummocks or water, inaccessible marsh areas), were recorded once a core was collected. The dominant vegetation at each site was recorded before clearing away vegetation for core extraction via a polycarbonate push core (10 cm diameter, 30cm length). Core tubes were driven to a depth > 15 cm then measured for compaction by recording three different distances from the top of the core to the soil for both the inner and outer portions of the core tube. These measurements were averaged, and the outer distance is subtracted from the inner distance and divided by the length of the

core to gauge compaction (Carr et al., 2020). Upon extrusion, the cores were capped then brought to the lab for storage in a freezer (-20°C) until they were ready to be processed.

Cores were collected over two sampling periods including January 6th-February 11th of 2022 and November 1st-30th of 2022. A total of sixty cores were collected, but only the thirty cores from the first sampling period will be utilized for the purpose of this thesis.

2.3 Sample Processing

Samples are removed from the freezer and allowed to thaw overnight prior to processing to allow for the soil to be removed from the core. After the soils have thawed, they are attached to a table using a worm gear clamp. A glove bag is then taped around the top of the core. The glove bag is deoxygenated using ultrahigh purity N_2 (g) before being sealed to keep the soil anoxic. The top cap is removed from the core so sectioning within the glove bag can begin. A hammer and PVC rod are utilized to tap the bottom cap of the core and push the soil up through the top opening. The soil within the core is then sectioned between 0-5cm, 5-10cm, and 10-15cm. These sections are placed into plastic bags (also kept anoxic with N_2 (g)) before being returned to the freezer. The remaining soil within the core is then discarded.

Core sections are freeze-dried in 1000ml freeze drying flasks. Each flask is attached to a VerTis Sentry freeze drier operated at -40ºC and 500 millitorr for at least a day to remove all moisture. The freeze-dried samples are broken up by hand over 3 ASTM E-11 specification sieves with decreasing mesh sizes (4mm, 1mm, and 0.5mm) to allow for the separation of soil from plant matter within the sections. The soil is

transferred to a pre-cleaned (450 °C, 4 h) 150 ml jar that is then deoxygenated with N_2 (g) before being sealed with the jar lid and stored frozen (-20 °C) until analysis.

2.4 Sample Analysis

2.4.1 Total Organic Carbon

CHNS elemental analysis (EA) was conducted using a COSTECH ECS4010 Elemental Analyzer following the methods outlined by Menzel and Vaccaro (1964) and Ehrhardt and Koeve (1999). Soil samples were acidified (1 M HCL) to convert inorganic carbon to CO_2, leaving behind the organic carbon pool. After overnight drying in desiccators, the samples were weighed and transferred to solvent-rinsed tins (dichloromethane and acetone) then placed in well plates until analysis. Samples were combusted in the elemental analyzer at 980º C in the presence of oxygen. The generated CO_2 proceeded to a thermal conductivity detector with the detector response correlating with its concentration within the sample as determined by an ethylenediaminetetraacetic acid standard curve following (eq. 1). This concentration was then related to the sample weight to obtain the total organic carbon (TOC) value reported as mg TOC g^{-1} Soil (eq. 2).

$$C_{TOC} = \frac{PA_{TOC} - b_{TOC}}{m_{TOC}} \quad (1)$$

C_{TOC} = concentration of sample (in mg)
PA_{TOC} = peak area of sample
B_{TOC} = intercept of calibration curve
M_{TOC} = slope of calibration curve

$$TOC = \frac{C_{TOC}}{W} \quad (2)$$

TOC = final POC concentration of sample (mg C/g soil)

C_{TOC} = concentration of sample from previous equation
W = weight of sample (in grams)

2.4.2 Black Carbon Analysis

2.4.2.1 Chemo-Thermal Oxidation at 375º C Black Carbon

The CTO-375 method employed in this study adheres to the procedure outlined by Gustafsson et al. (2001). Homogenized and freeze-dried soil samples were weighed into 10ml crucibles, and 1 M hydrochloric acid was added to remove inorganic carbon. Afterward, the samples were placed in a desiccator for 24 hours to ensure complete drying. The dried and acidified samples were transferred to 5ml crucibles and subjected to a 24-hour treatment at 375 °C in a Thermolyne Furnace to eliminate non-pyrogenic organic carbon (OC). To prevent charring, the oven was equipped with a tube and aquarium pump to maintain constant airflow, facilitating the supply of oxygen.

The combusted and non-combusted samples were then weighed and analyzed for carbon content using a Costech CHNS Elemental Analyzer (as described above in 2.4.1). The results for $BC_{CTO\text{-}375}$ were reported as mg BC_{CTO} g^{-1} soil. To verify the accuracy of BC recovery, a set of standards was prepared and analyzed alongside the samples. These standards included 1) a blank sand standard, 2) commercially available biochar (Wakefield), 3) standard reference material (SRM) 1941b (marine soil collected from Baltimore Harbor, purchased from the National Institute of Standards and Technology [NIST]), and 4) SRM2975 (diesel particulate matter, NIST).

2.4.2.2 Benzene Polycarboxylic Acid Method Black Carbon

Benzene polycarboxylic acid (BPCA) analyses were conducted following the methods outlined by Wagner et al. (2017) and Barton and Wagner (2022). Dried and homogenized soil samples from the cores were sent to Dr. Sasha Wagner's laboratory at Rensselaer Polytechnic

Institute in Troy, NY. An amount of soil containing less than 5mg of organic carbon (OC) was placed into 20ml ampules along with concentrated nitric acid. The ampules were sealed and subjected to 6 hours of heating at 150 °C in an oven. This thermal oxidation with HNO_3 digested the pyrogenic aromatic compounds, converting them into their carboxylated constituent building blocks (BPCAs). To ensure complete preservation of the oxidation products, the nitric acid and reaction products were carefully transferred to 12 mL vials.

A drying apparatus involving inert gas and a hot sand bath was used to evaporate the nitric acid, allowing the BPCAs to be prepared for separation and quantification. The samples were then dissolved using 0.75 mL of nitric acid and transferred to 2 mL vials before undergoing a second round of nitric acid evaporation. Subsequently, the samples were transferred to HPLC vials using mobile phase A (a dilute phosphoric acid solution (0.6 M; pH1)) and kept ready for analysis using high-performance liquid chromatography.

BC content was quantified using a calibration curve established using standard compounds and reported as BC_{BPCA}. The number of carboxylic acids present on a BPCA, as determined by this technique, positively correlates with the relative abundance of aromatic rings in a given compound. Highly condensed B5CA and B6CA compounds have been shown to be predominantly produced by pyrogenic processes, while B3CA and B4CA compounds have been demonstrated to originate from both pyrogenic and non-pyrogenic processes (Bostick et al., 2018). B5CA and B6CA concentrations were calculated by integrating peaks from HPLC occurring at approximate retention times. The concentrations of B5CA and B6CA were summed, and a conversion factor from Stubbins et al. (2015) was applied to obtain the BC_{BPCA} concentration (eq. 3). This value was further converted to report BC_{BPCA} as mg BC_{BPCA} g^{-1} soil. It is important to note that this estimate provides a conservatively low estimation of the quantity of BC produced by burning, as B3CA and B4CA compounds are

also produced during burning. The B3CA and B4CA compounds have also been found to have non-pyrogenic sources (Bostick et al., 2018) and thus are not used to quantify BC. Additionally, the ratio of B6CA to B5CA serves as a measure of the relative degree of condensed aromaticity in the sample.

$$[BC] = 0.0891\ x([B6CA] + [B5CA])\ 0.9175 \quad (3)$$

2.5 Statistical Analysis

Statistical significance was determined among sites via one-way Analysis of Variance (ANOVA). When statistical differences were detected by ANOVA, post Hoc Tukey tests were conducted to further elucidate the differences between the sites. Correlations were conducted to measure the potential effects of other variables such as elevation and loss on ignition within and among sites on the results. All statistical tests were conducted using Rstudio version 4.2.2.

Chapter 3

RESULTS

3.1 Total Organic Carbon

Total Organic Carbon (TOC) varied significantly among the sites with a mean of 62.27 ± 31.12 mg TOC g^{-1} soil and a range of 17.8-209.6 mg TOC g^{-1} soil (n=90). St. Jones contained the highest mean concentration at 81.24 ± 44.18 mg TOC g^{-1} soil (n=30). This site encompassed a wide scale of TOC spanning 35.18-209.62 mg TOC g^{-1} soil, a range of 174.44 mg TOC g^{-1} soil (Table 1) (Fig.3). The site with the next highest concentration was Roberts Farm with a mean of 58.36 ± 14.37 mg TOC g^{-1} soil (n=30) and a range of 37.8-86.4 mg TOC g^{-1} soil. Rocks Tract contains the lowest TOC of the three sites with a mean of 47.22 ± 12.06 mg TOC g^{-1} soil (n=30) and a range of 62.3 mg TOC g^{-1} soil.

Table 1: Mean, minimum, maximum, and range of soil TOC concentrations (mg TOC g^{-1} soil) for core sections at each site as well as the total across every site. Mean values for a site are calculated for every core section (n=30 per site).

Site Name	Average	Minimum	Maximum	Range
Roberts Farm	58.4 ± 14.4	37.8	86.4	48.6
Rocks Tract	47.2 ± 12.1	17.8	80.0	62.3
St. Jones	81.2 ± 44.2	35.2	209.6	174.4
Total	62.3 ± 31.1	17.8	209.6	191.9

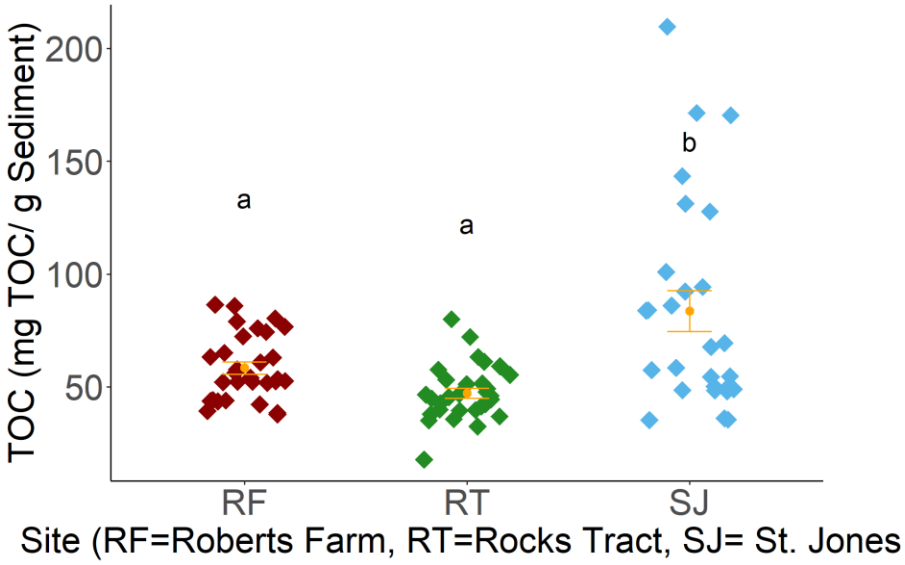

Figure 3: TOC concentrations (mg TOC g^{-1} soil) for the core sections collected at each site. Mean concentrations and one standard deviation are denoted by the orange diamond and error bars. The a and b notations denote which sites are or are not statistically different from one another (ANOVA with Tukey HSD, $p < 0.001$).

ANOVA analysis identified a statistical difference in TOC concentration among the sites ($F(2)= 12.18$, $p < 0.001$). A Tukey Post Hoc Test revealed a significant difference between St. Jones and Roberts Farm (difference of 25.27 mg TOC/ g soil, $p <0.01$) as well as St. Jones and Rocks Tract (36.41 mg TOC/ g soil, $p < 0.001$). TOC at Roberts Farm and Rocks Tract were found to be statistically indistinguishable ($p > 0.05$). No significant differences were observed with depth over the entire dataset and within a site (ANOVA, $F(2)= 0.249$, $p > 0.05$) (Fig. 4).

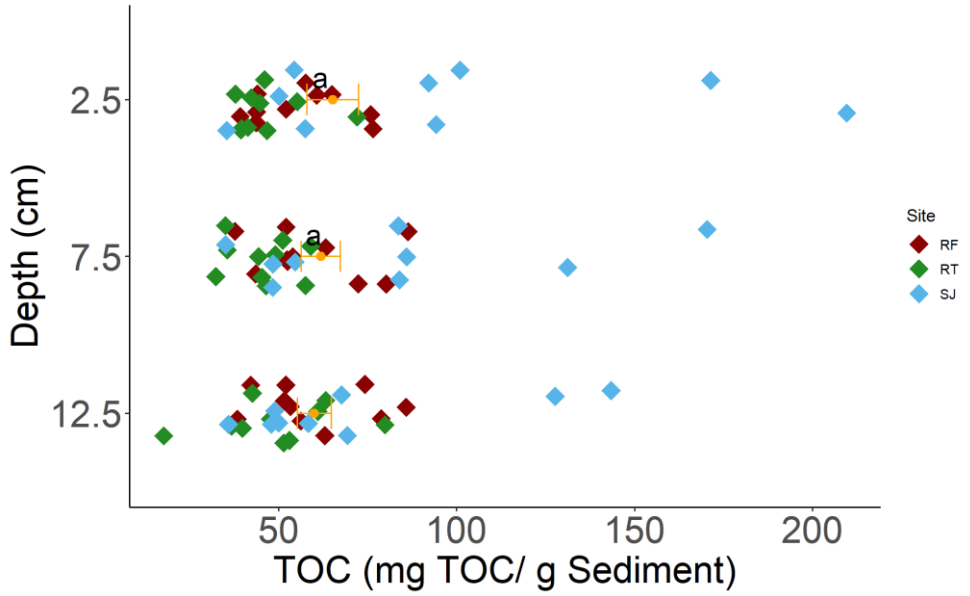

Figure 4: TOC concentrations (mg TOC g^{-1} soil) plotted versus depth of core section.

3.2 Black Carbon via Chemo-Thermal Oxidation at 375° C

Black Carbon values obtained from this method (BC_{CTO}) exhibited similar trends to the TOC values (Figure 5). Although the trends are similar to TOC, the average value of BC_{CTO} (1.84 ± 1.19 mg BC_{CTO} g^{-1} soil with a range of 0.43-7.19 mg BC_{CTO} g^{-1} soil (n=90)) is much lower than TOC. St. Jones contained the highest value for black carbon with a mean of 2.78 ± 1.58 mg BC_{CTO} g^{-1} soil and ranged from 0.75-7.19 mg BC_{CTO} g^{-1} Soil (n=30) (Table 2). This again was followed by Roberts Farm at a mean of 1.44 ± 0.26 mg BC_{CTO} g^{-1} soil and a range of 0.80-2.06 mg BC_{CTO} g^{-1} soil (n=30). The lowest values obtained for BC_{CTO} was at Rocks Tract which contained a mean of 1.31 ± 0.57 mg BC_{CTO} g^{-1} soil with a range of 0.43-3.45 mg BC_{CTO} g^{-1} Soil.

Table 2: Mean, minimum, maximum, and range of soil BC_{CTO} concentrations (mg BC_{CTO} g^{-1} soil) for core sections at each site as well as the total across every site. Mean values for a site are calculated for every core section (n=30 per site).

Site Name	Average	Minimum	Maximum	Range
Roberts Farm	1.44 ± 0.26	0.80	2.06	1.26
Rocks Tract	1.31 ± 0.57	0.43	3.45	3.02
St. Jones	2.78 ± 1.58	0.75	7.19	6.44
Total	1.84 ± 1.19	0.43	7.19	6.76

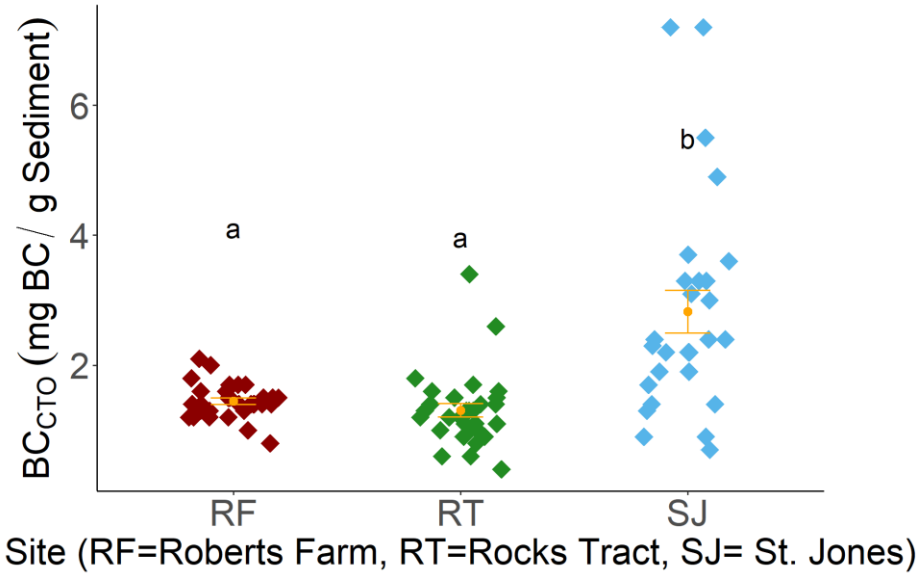

Figure 5: BC_{CTO} concentrations (mg BC_{CTO} g^{-1} soil) for the core sections collected at each site. Mean concentrations and one standard deviation are denoted by the orange diamond and error bars. The a and b notations denote which sites are or are not statistically different from one another (ANOVA with Tukey HSD, $p < 0.001$).

ANOVA analysis identified a statistical difference in BC_{CTO} concentration found among the sites ($F(2)= 19.07$, $p < 0.001$). A Tukey Post Hoc Test revealed a significant difference between St. Jones and Roberts Farm (difference of 1.38 mg BC_{CTO} g^{-1} soil, $p <0.001$) as well as St. Jones and Rocks Tract (1.52 mg BC_{CTO} g^{-1} soil, $p < 0.001$). No significant difference was observed for BC_{CTO} between Roberts Farm and Rocks Tract ($p > 0.05$). A further ANOVA test reveals there is no significant difference across depth among all the results ($F(2)= 0.249$, $p > 0.05$) (Fig. 4).

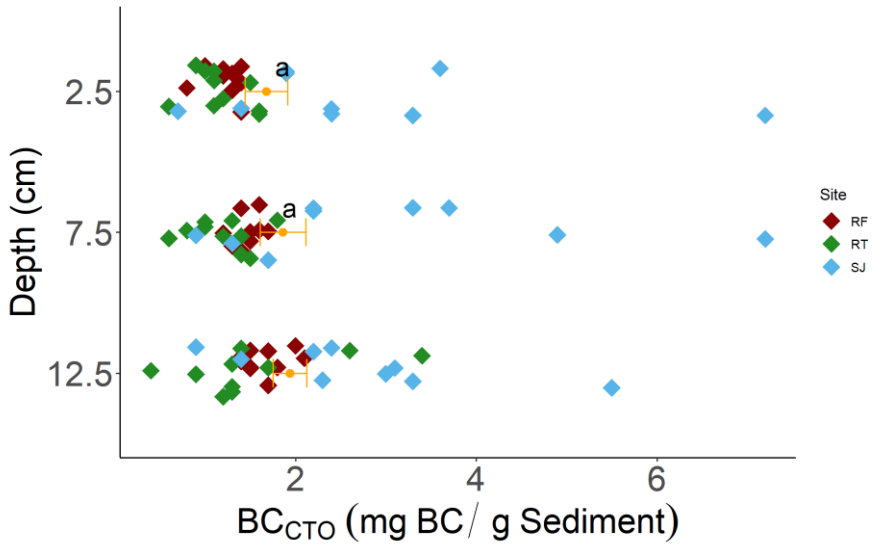

Figure 6: BC_{CTO} concentrations (mg BC_{CTO} g^{-1} soil) plotted versus depth of core section.

3.3 Black Carbon via Benzene Polycarboxylic Acid Method

The values obtained for BC generated using the BPCA method (BC_{BPCA}) differed in concentration trends when compared to TOC and BC_{CTO}. The mean concentration applying this method was higher than the BC_{CTO} (4.97 ± 1.06 mg BC_{BPCA} g^{-1} soil; range = 2.214-8.400 mg BC_{BPCA} g^{-1} soil (n=88)). Roberts Farm soils contained the highest values for BC_{BPCA} (5.802 ± 1.063 mg BC_{BPCA} g^{-1} soil; range = 4.22-8.40 mg BC_{BPCA} g^{-1} Soil (n=30) (Table 3) (Fig. 7)). Rocks Tract followed Roberts Farm in terms of concentration with a mean of 4.76 ± 0.77 mg BC_{BPCA} g^{-1} soil and a range of 2.40-6.18 mg BC_{BPCA} g^{-1} soil. BC_{BPCA} concentrations were lowest at St. Jones (4.28 ± 0.65 mg BC_{BPCA} g^{-1} soil; range = 2.21-5.61 mg BC_{BPCA} g^{-1} soil).

Table 3: Mean, minimum, maximum, and range of soil BC_{BPCA} concentrations (mg BC_{BPCA} g^{-1} soil) for core sections at each site as well as the total across every site. Mean values for a site are calculated for every core section (RF and RT n=30, SJ n=28).

Site Name	Average	Minimum	Maximum	Range
Roberts Farm	5.80 ± 1.06	4.22	8.40	4.18
Rocks Tract	4.76 ± 0.77	2.40	6.18	3.78
St. Jones	4.28 ± 0.65	2.21	5.61	3.39
Total	4.97 ± 1.06	2.21	8.40	6.19

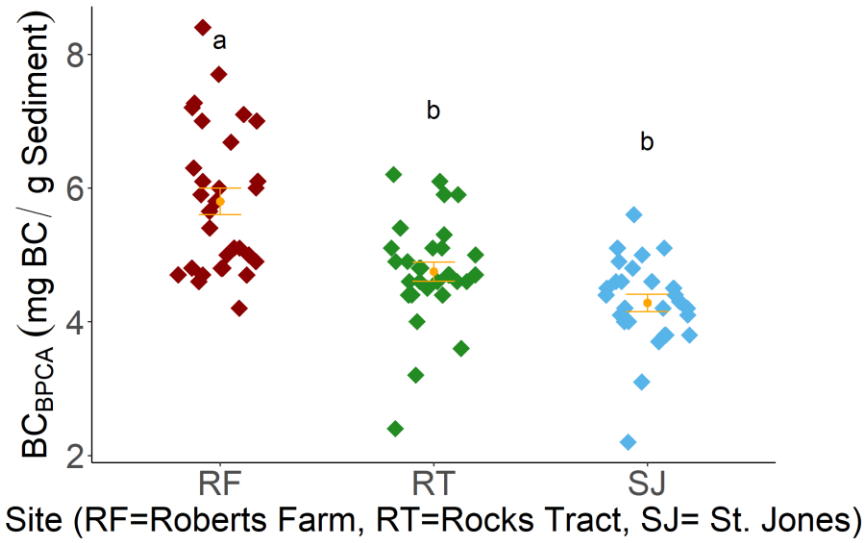

Figure 7: BC_{BPCA} concentrations (mg BC_{BPCA} g^{-1} soil) for the core sections collected at each site. Mean concentrations and one standard deviation are denoted by the orange diamond and error bars. The a and b notations denote which sites are or are not statistically different from one another (ANOVA with Tukey HSD, $p < 0.001$)

Along with the concentration of BC_{BPCA}, the BPCA method supplied data on the relative condensation of aromatic compounds at each site. This data is presented as a ratio of the B6:B5 components present in a sample. The mean across all sites is 1.19 ± 0.25 with a range of 0.54-1.75 (n=88) (Table 4) (Fig. 8). The B6:B5 ratio was highest at Roberts Farm at with a mean of 1.29 ± 0.14 and range of 1.06-1.75 (n=30). Rocks Tract followed Roberts Farm with a mean of 1.23 ± 0.23 encompassing a range of 0.54-1.57 (n=30). St. Jones had the lowest B6:B5 ratio of the sites at 1.04 ± 0.29 and (range = 0.54-1.71 (n=28)).

Table 4: Mean, minimum, maximum, and range of soil B6:B5 ratio for core sections at each site as well as the total across every site. Mean values for a site are calculated for every core section (RF and RT n=30, SJ n=28).

Site Name	Average	Minimum	Maximum	Range
Roberts Farm	1.29 ± 0.14	1.06	1.75	0.69
Rocks Tract	1.23 ± 0.23	0.54	1.57	1.03
St. Jones	1.04 ± 0.29	0.54	1.71	1.17
Total	1.19 ± 0.25	0.54	1.75	1.22

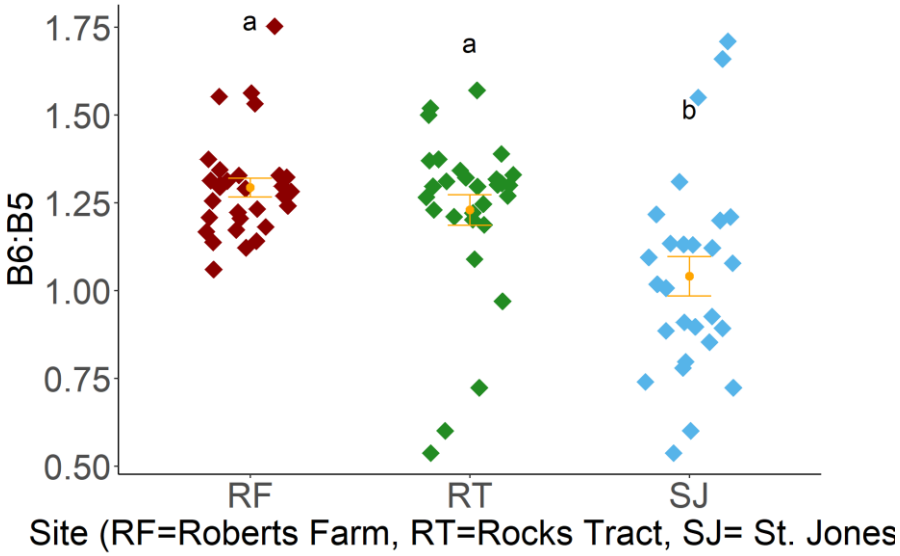

Figure 8: B6:B5 ratios for the core sections collected at each site. Mean ratios and one standard deviation are denoted by the orange diamond and error bars. The a and b notations denote which sites are or are not statistically different from one another (ANOVA with Tukey HSD, $p < 0.001$).

ANOVA analysis identified a statistical difference in BC_{BPCA} concentration among the sites ($F(2)= 22.9$, $p < 0.001$). A Tukey Post Hoc Test revealed a significant difference between Roberts Farm and St. Jones (difference of 1.52 mg BC_{BPCA} g^{-1} soil, $p <0.001$) as well as Roberts Farm and Rocks Tract (1.05 mg BC_{BPCA} g^{-1} soil, $p < 0.001$). No significant difference was observed for BC_{BPCA} between St. Jones and Rocks Tract ($p > 0.05$). A further ANOVA test reveals there is no significant difference across depth among all the results ($F(2)= 1.681$, $p > 0.05$) (Fig. 9).

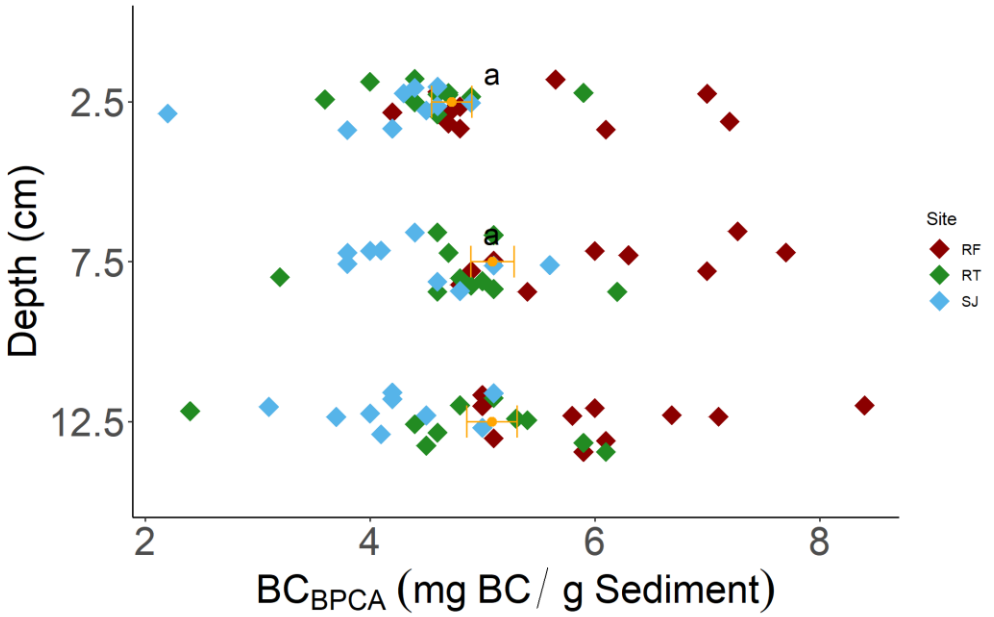

Figure 9: BC_{BPCA} concentrations (mg BC_{BPCA} g^{-1} soil) for all sample core sections color-coded for site and plotted versus depth of core section.

Similar to the BC_{BPCA} concentrations, there were significant differences between the three sampling sites for the B6:B5 ratio (ANOVA, $F(2)= 9.13$, $p < 0.001$). A Tukey Post Hoc Test revealed a significant difference between Roberts Farm and St. Jones (difference of 0.25, $p <0.001$) as well as Rocks Tract and St. Jones (difference of 0.19, $p < 0.01$). No significant difference for B6:B5 ratio was observed between Rocks Tract and Roberts Farm ($p > 0.05$). A further ANOVA test reveals there is no significant difference across depth among all the results ($F(2)= 0.53$, $p > 0.05$) (Fig. 10).

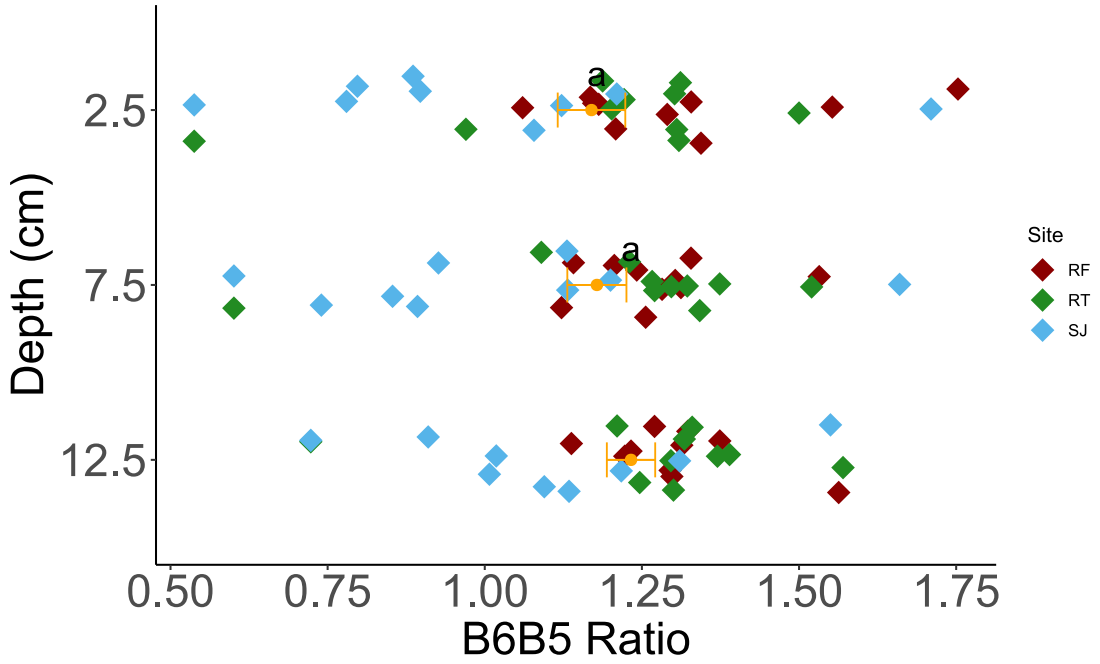

Figure 10: B6:B5 ratios for all sample core sections color-coded for site and plotted versus depth of core section.

Chapter 4

DISCUSSION

4.1 Comparison of Black Carbon Methodology for Tidal Marsh Soils

Several studies have explored the role of black carbon in organic carbon pools within tidal wetlands (Wang et al., 2014; Guo et al., 2018; St. Laurent et al., 2020; Gallagher et al., 2021). However, the majority of the research examining soil and sedimentary BC pools has focused on terrestrial soils (Schmidt et al., 2001; Czimczik et al., 2004; Liang et al., 2006; Czimczik and Masiello, 2007; Hammes et al., 2007) and marine sediments (Suman et al., 1997; Gustafsson et al., 2001; Gélinas et al., 2001; Middleburg et al., 1999; Coppola et al., 2014). Limited studies employed the BC_{CTO} method to investigate tidal wetland, specifically marsh wetland, BC pools (St. Laurent et al.; Gallagher et al., 2021). Consequently, questions remain regarding the applicability of different methods in tidal wetlands and the resulting variations in concentrations among these methods.

4.1.1 Potential Charring using the CTO-375 Method due to TOC Rich Samples

One of the known drawbacks associated with the CTO-375 method is the potential occurrence of charring, which can lead to artificially inflated BC_{CTO} concentrations, particularly in OC-rich samples. Gustafsson et al. (2004) noted that samples rich in organic carbon are more prone to the charring artifact. Since sample TOC is charred to produce BC_{CTO}, evidence for an artifactual BC_{CTO} measurement can be found in linear correlations between BC_{CTO} and TOC concentration. Unlike Roberts Farm and Rocks Tract soils, the St. Jones soils show a strong positive correlation between BC_{CTO} and TOC (Fig. 12; r^2 =0.8). The St. Jones soils exhibit significantly

higher TOC compared to Roberts Farm and Rocks Tract soils (Fig. 3) which may explain why clear evidence for charring is only observed at that site.

A dilution test was conducted to further validate the charring observation. The four cores from St. Jones with the highest TOC concentrations (all three sections of SJ01, SJ02, SJ06, SJ09, n=12) were diluted using a blank sand sample (Thermo Fisher Scientific S2510) at ratios of 1:1, 1:2, and 1:3. The results of the dilution test revealed a significant decrease in BC_{CTO} concentration, with a mean decrease of 1.6 ± 0.9 mg BC_{CTO} g^{-1} Soil per sample. The mean concentration decreased for each ratio, with values of 1.5 ± 1.0 mg BC_{CTO} g^{-1} Soil for 1:1, 1.5 ± 0.7 mg BC_{CTO} g^{-1} Soil for 1:2, and 1.9 ± 0.9 mg BC_{CTO} g^{-1} Soil for 1:3 ($F(3)=4.932$, $p < 0.01$) (Fig. 16). All individual cores exhibited a significant decrease in BC_{CTO} concentration for at least one dilution, except for SJ06 ($p < 0.05$) (Fig. 17). This decrease in BC_{CTO} concentration observed in the dilution test serves as conclusive evidence confirming the occurrence of charring during the CTO-375 method.

The lack of a strong correlation between TOC and BC_{CTO} for Roberts Farm and Rocks Tract suggests that TOC concentrations were low enough to prevent charring and avoid any impact on the quantity of BC though further sand dilution tests are needed to confirm the accuracy of all BC_{CTO} measurements. Artifactual charring of the St. Jones soils but not the Roberts Farm or Rocks Tract site provides an explanation for why the St. Jones soils showed the lowest BC_{BPCA} but the highest BC_{CTO} concentrations.

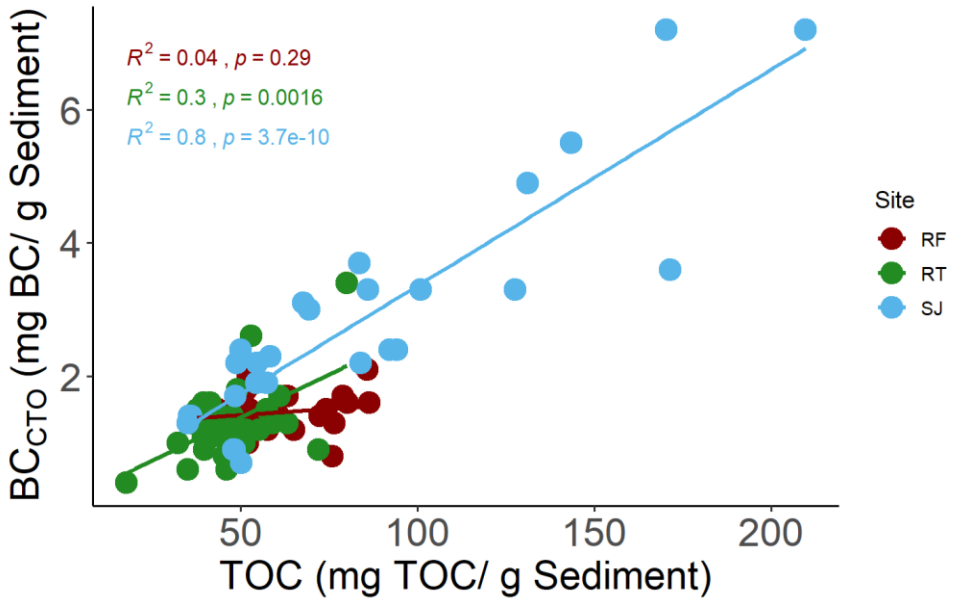

Figure 11: Linear regression for each site show a correlation between TOC and BC_{CTO}

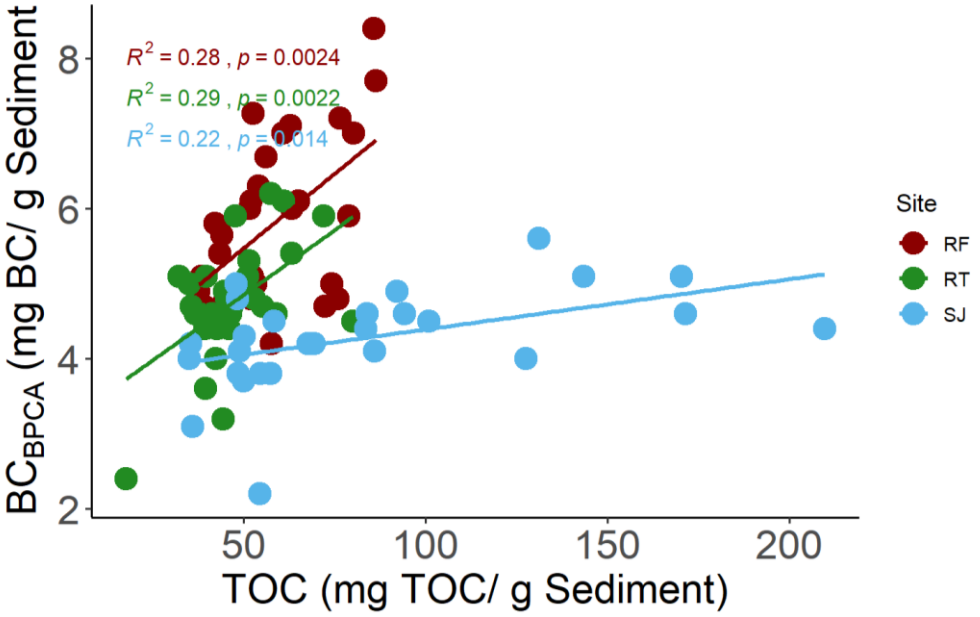

Figure 12: Linear regression for each site show a correlation between TOC and BC_{BPCA}

Linear regression analyses were conducted for TOC, BC_{CTO}, and BC_{BPCA}, along with selected parameters, to assess their influence on the reported concentrations (Figs. 14 and 15). In the context of methods, it is noteworthy that TOC and BC_{CTO} exhibit similar trends concerning elevation and the B6:B5 ratio (R^2= 0.55 and 0.56 for TOC versus elevation, and BC_{CTO} versus elevation, respectively; R^2= 0.7 and 0.98 for TOC versus B6:B5 ratio, and BC_{CTO} versus B6:B5 ratio, respectively). These similar correlations provide further evidence that, specifically for St. Jones, BC_{CTO} is more of a reflection of high TOC concentrations on sites as opposed to being representative of BC_{soot} actually present.

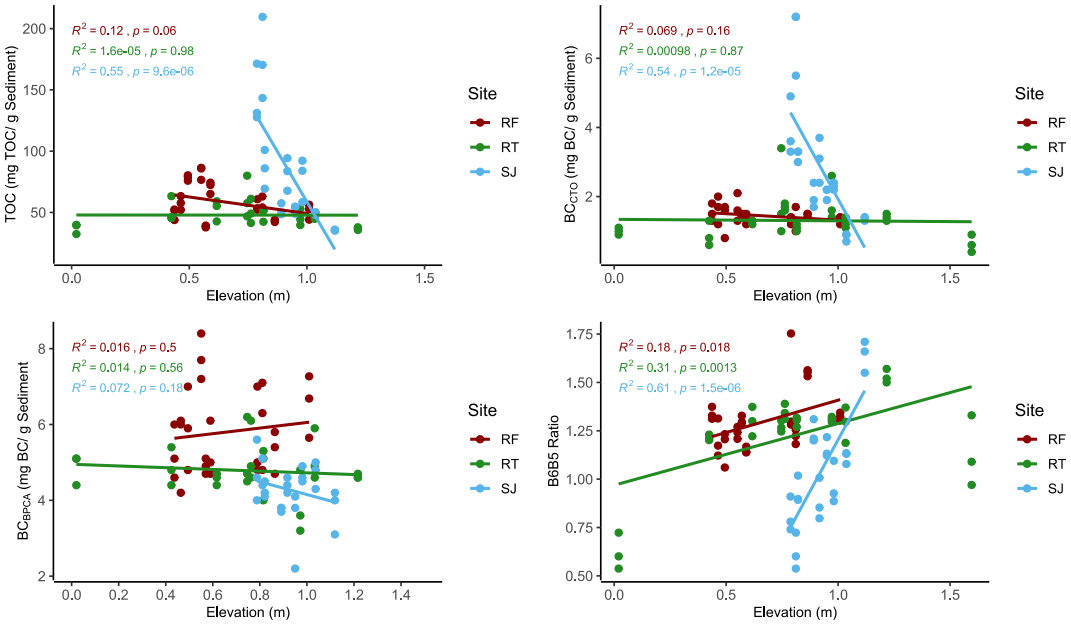

Figure 13: Linear regressions conducted for TOC, BC_{CTO}, BC_{BPCA}, and B6B5 ratio for each site versus elevation (m)

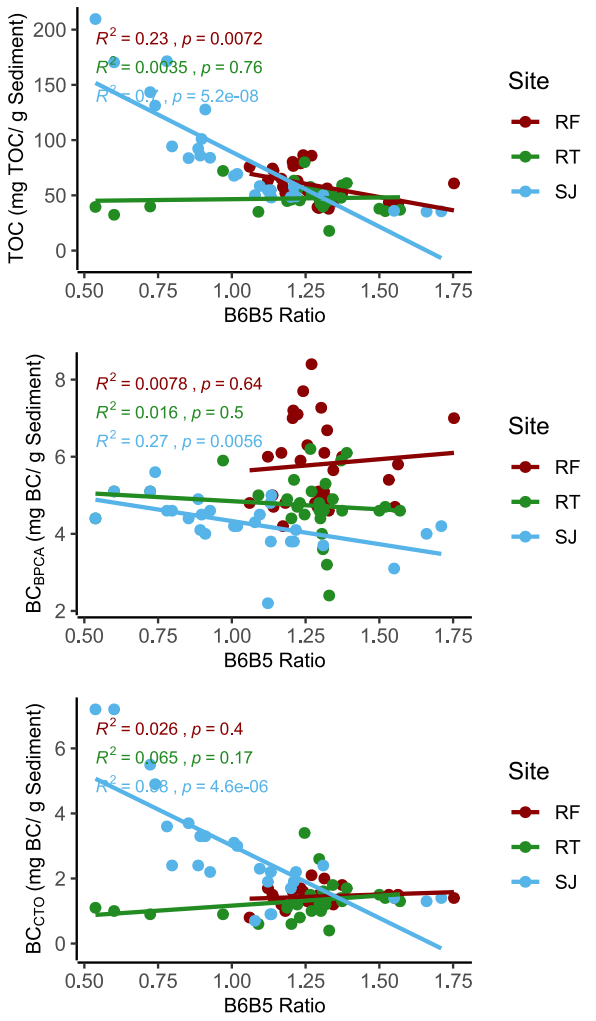

Figure 14: Linear regressions conducted for TOC, BC_{CTO}, and BC_{BPCA} ratio for each site versus ethe B6:B5 ratio

The presence of charring is particularly significant for tidal wetland soils due to their generally high concentration of organic carbon compared to adjacent marine and terrestrial environments (McLeod et al., 2011). It could pose a consistent issue for researchers investigating these environments, as it may lead to inflated BC

values. Given the potential inflation of BC_{CTO} values due to charring in this study, the subsequent discussion comparing BC across sites will exclude the St. Jones CTO-375 results.

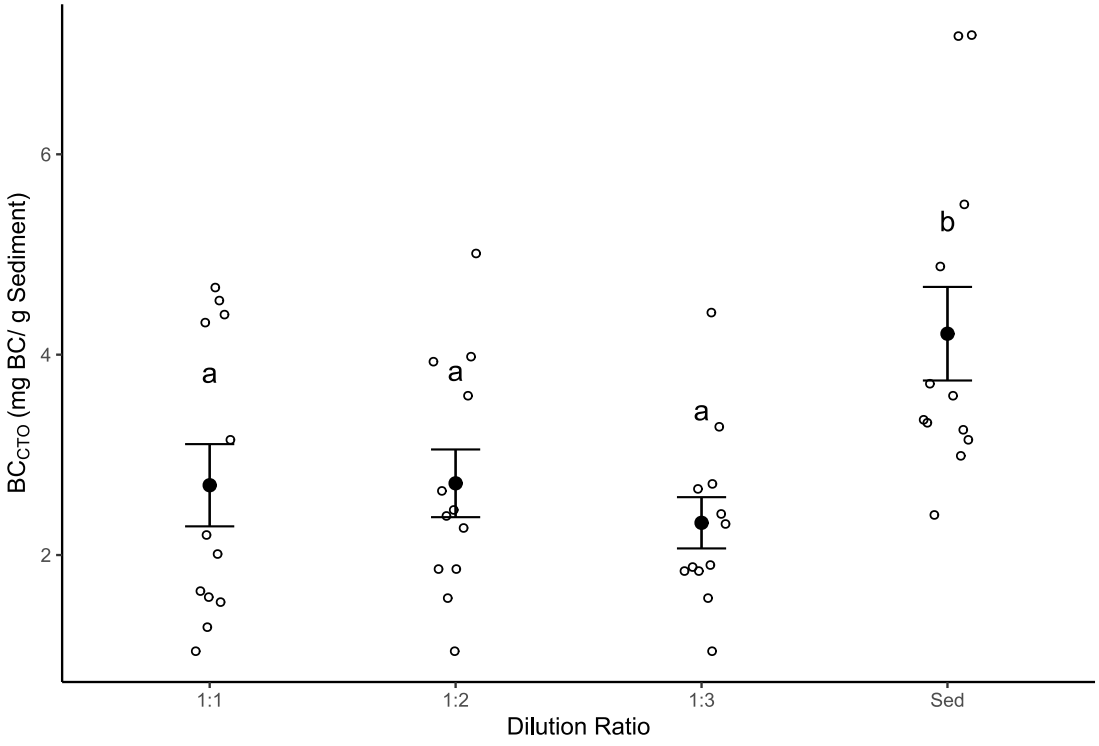

Figure 15: Results of the dilution test across each ratio of dilution. Letters above ratios indicate statistical differences for different letters ($p < 0.05$).

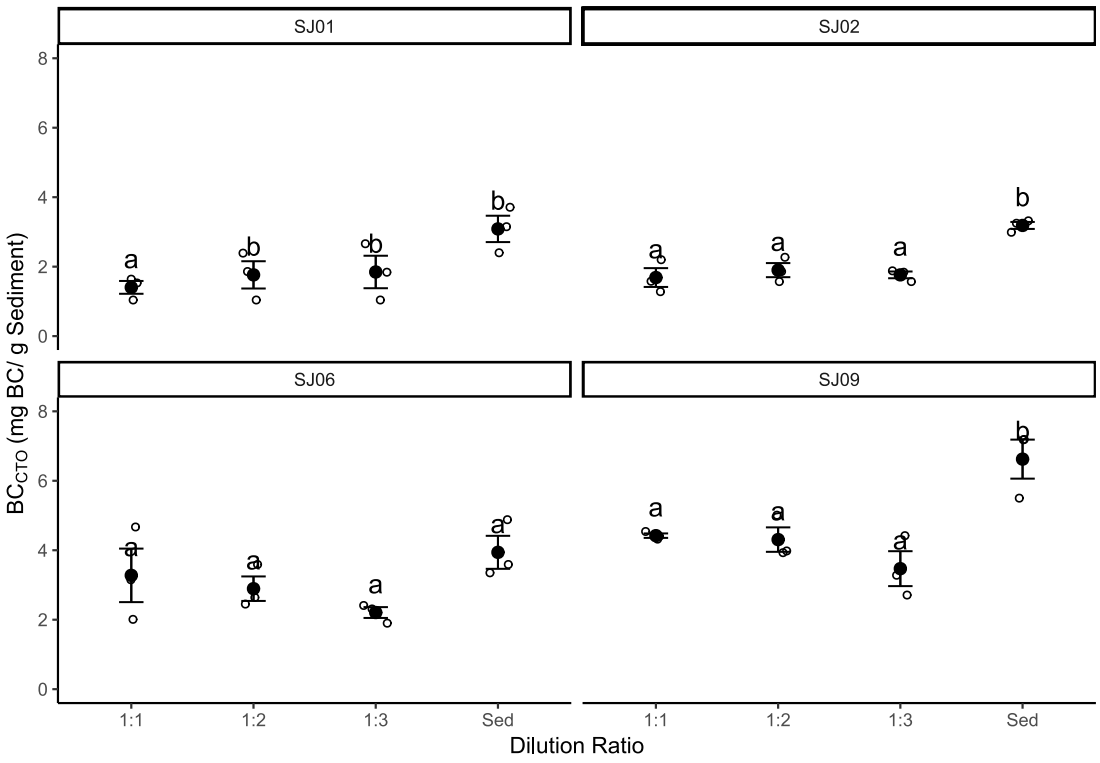

Figure 16: Results of the dilution test across each core. Letters above ratios indicate statistical similarities or differences between groups of samples ($p < 0.05$)

4.1.2 Black Carbon Concentration Comparison for BPCA and CTO-375 Methods

The concentration discrepancy between the two methods is a notable finding. The BPCA method consistently yielded higher BC concentrations compared to the CTO-375 method. Across the entire dataset, BC concentrations were significantly higher for BC_{BPCA} than BC_{CTO} ($p < 0.001$), with an average difference of 3.1 ± 1.7 mg BC g^{-1} soil per sample. This pattern was consistent across all sites, with mean offsets of RF = 4.4 ± 1.0 mg BC g^{-1} soil, RT = 3.5 ± 0.9 mg BC g^{-1} soil, and SJ = 1.5 ± 1.5 mg BC g^{-1} soil, all exhibiting similarly significant differences in concentration ($p < 0.001$) (Fig. 11).

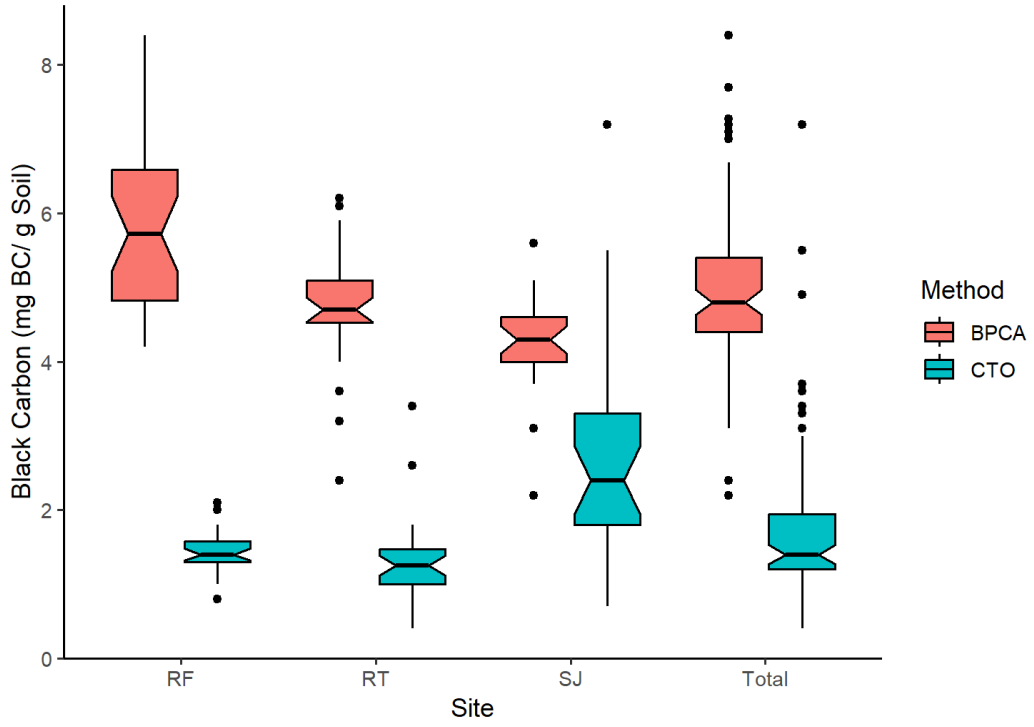

Figure 17: Box plot displaying the difference in BC concentration for the BPCA and CTO methods.

Comparing the difference in concentration between the two methods becomes challenging due to the scarcity of studies providing both CTO-375 and BPCA data for the same tidal wetland samples. However, investigations on terrestrial soils have shown higher BC_{BPCA} concentrations compared to BC_{CTO} (Schmidt et al., 2011; Hammes et al., 2007). Conversely, studies on marine sediments generally indicate higher BC_{CTO} values than BC_{BPCA} (Hammes et al., 2007). This trend aligns with the results obtained using the NIST standard 1941b, analyzed as a standard using both methods. The NIST 1941b standard reported a mean value of 5.35 ± 0.17 mg BC_{CTO} g^{-1} soil, while the BPCA analysis yielded a value of 2.4 mg BC_{BPCA} g^{-1} soil.

The consistent higher concentrations of BC_{BPCA} compared to BC_{CTO} can be attributed to several factors associated with the methods and the combustion continuum. One possible explanation for the higher BC_{BPCA} concentrations is the potential overestimation resulting from the method itself. Previous studies have highlighted overestimates in BC_{BPCA} concentrations due to the inclusion of non-pyrogenic BC (Hammes et al., Bostick et al., 2018). To mitigate this effect, tri- and tetra-carboxylated BPCA components are excluded from the final counts. However, arguments also suggest that the BPCA method may underestimate BC_{BPCA} concentrations because the largest and most condensed aromatic compounds are not fully digested into their constituent BPCAs, leading to incomplete quantitative recovery. Although a conversion factor can address this issue, it still provides a conservative estimate (Glaser et al., 1998).

The CTO-375 method also possesses certain attributes and disadvantages that help explain the results. One reason for potential underestimation using the CTO-375 method is its focus on quantifying highly condensed BC, typically limited to the soot portion of the combustion continuum, while most charcoal is not recognized as BC (Nguyen et al., 2004). Consequently, a larger portion of the combustion continuum is excluded compared to the BPCA method, which encompasses an overlap of charcoals and soot. Another disadvantage of the CTO-375 method is the potential for overestimation caused by sample charring during thermal treatment (Lim and Cachier, 1996; Masiello et al., 2004). This charring issue can be particularly problematic for samples rich in organic carbon (Gustafsson et al., 2002), and previous studies have disregarded BC_{CTO} results due to interference from charring (Lim and Cachier, 1996).

The significance of this charring issue in the present study will be discussed further in subsequent sections.

Overall, the higher BC_{BPCA} concentrations compared to BC_{CTO} can be attributed to the BPCA method's enhanced ability to detect the char component of the combustion continuum. This suggests that tidal wetland soils may report values more similar to terrestrial soils rather than marine sediments in future studies. This finding is particularly relevant for tidal wetland areas that have experienced or are in close proximity to burn sites, where a substantial amount of char could be deposited. Moreover, the BPCA method offers additional valuable information on the relative degree of aromatic compound condensation, making it a more useful tool for investigating BC in tidal wetlands.

4.2 Comparison of Black Carbon Across and within Sites of Varying Burn History

4.2.1 Black Carbon Variation across Sites of Varying Burn History

When revisiting the three selected sites for this project, it is crucial to consider differences in their tidal regimes, mean salinities, and vegetational diversity. The St. Jones marsh, which was included in the study design primarily because it is a Delaware National Estuarine Research Reserve site but also because it has no known burn history, is classified as a mesohaline salt marsh (mean of 10.8 ± 7.0) with a higher tidal regime (site's mean elevation is 0.909 ± 0.108 m, and it has a tidal range mean of 1.5 m with a mean spring tide range of 1.7 m) and lower vegetational

diversity relative to the oligohaline Rocks Tract and Roberts Farm marshes (mean salinity of 2.0 ± 2.1, Rocks Tract mean elevation is 0.820 ± 0.409 m, Roberts Tract mean elevation of 0.658 ± 0.185 m, mean tidal range for the Lower Blackbird Creek is 1.7 m with the average spring tide range being 1.92 m). These variations in physical, chemical, and biological characteristics can contribute to discrepancies in measured values between St. Jones and the other two sites.

These physicochemical differences at St. Jones may be reflected in the total organic carbon (TOC) concentrations. St. Jones reports significantly higher TOC values ($p < 0.001$) compared to the other two sites, with a mean concentration of 62.3 ± 31.1 mg TOC g^{-1} soil, in comparison to 58.4 ± 14.4 mg TOC g^{-1} soil for Roberts Farm and 47.2 ± 12.1 mg TOC g^{-1} soil for Rocks Tract (Fig. 3). While the underlying causes for the higher TOC concentrations at St. Jones are beyond the scope of this research, these findings align with other studies investigating soil carbon in similar areas. St. Laurent et al. (2020) observed significantly higher OC concentrations at St. Jones Reserve (8.38 ± 4.74%) compared to Blackbird Creek Reserve (5.71 ± 2.30%).

Rocks Tract and Roberts Farm, being adjacent to each other along Blackbird Creek, share similar tidal range, salinity, dominant vegetation, and comparable elevations. When examining the data across all sites, both Rocks Tract and Roberts Farm exhibit significantly lower TOC concentrations than St. Jones (ANOVA $p < 0.001$), but a post-hoc test reveals no significant difference between the two sites (Tukey's post hoc test, $p > 0.05$). A student's t-test, however, shows that Roberts Farm has a statistically higher TOC concentration than Rocks Tract, with a mean difference of 11.1 mg TOC g^{-1} soil (student's t-test, $p < 0.01$) (Fig. 18). Thus, while these sites are adjacent and share physicochemical similarities when compared to St. Jones, they

do show differing TOC concentrations. Differences in soil TOC concentrations must be due to differences in TOC production/delivery (e.g., above- or belowground biomass production rates, the delivery of allochthonous TOC from the watershed or the estuary, or burning inputs) and removal (e.g., soil degradation, export to the estuary) processes. While we cannot definitively point to a single factor causing the differences in TOC, we can investigate the potential role of prescribed burns.

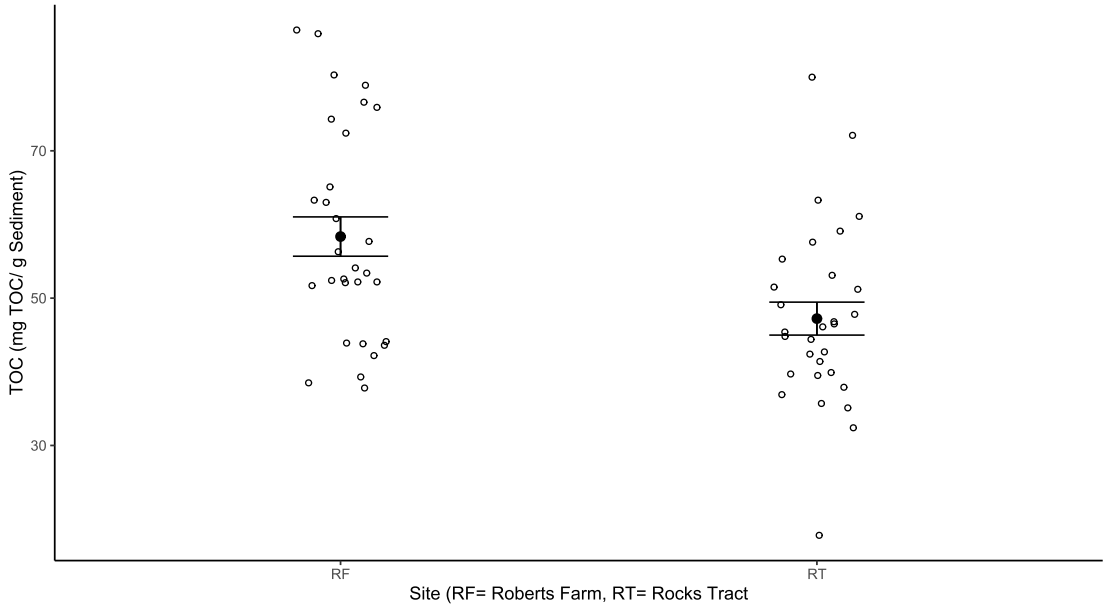

Figure 18: Comparison of Roberts Farm and Rocks Tract TOC with the omission of St. Jones.

Roberts Farm exhibits a slightly higher concentration of BC_{CTO} than Rocks Tract, with means of 1.44 ± 0.26 and 1.31 ± 0.57 BC_{CTO} g^{-1} soil, respectively, but this

difference is not statistically significant when applying ANOVA analysis and a t-test ($p > 0.05$). Assuming charring is not responsible for discrepancies in these samples, it suggests that BC inputs are similar for a burn site and its adjacent areas, at least in terms of the soot fraction of the combustion continuum that gets deposited. The BPCA method yields different patterns in BC abundance with Roberts Farm soils showing the highest (one-way ANOVA with Tukey post-hoc test; $p < 0.001$) BC_{BPCA} concentrations (5.80 ± 1.06 mg BC_{BPCA} g^{-1}). St. Jones soils exhibit the lowest concentration (4.28 ± 0.65 mg BC_{BPCA} g^{-1}; Table 3) with Rocks Tract soils showing a higher (but statistically insignificant, $p>0.05$) mean concentration of 4.76 ± 0.77 mg BC_{BPCA} g^{-1} soil (Fig. 7). Given the presumed combustion derivation for BC, it is reasonable to suggest that the higher BPCA concentrations at the Roberts Farm site are due to the onsite prescribed burns. Combustion processes also yield TOC byproducts not characterized as BC_{soot} or BC_{BPCA} (Wozniak et al., 2020), and it follows that the higher TOC concentrations in Roberts Farm relative to Rocks Tract may also relate to these burning inputs.

If related back to BC_{CTO} concentrations, it appears to indicate that the majority of buried BC originates from char material directly deposited on-site. Soot concentrations may be similar in adjacent sites due to atmospheric and sediment transport, but the primary accumulation of char material seems to be limited to the burn site.

Despite the lack of recent prescribed burns, St. Jones and Rocks Tract show BC_{BPCA} concentrations that are 74% and 82% those of Roberts Farm, respectively. A portion of the BC_{BPCA} observed in the Rocks Tract soils may derive from atmospheric and/or tidal transport of materials produced in the burns at adjacent Roberts Farm. The

high BC_{BPCA} values at St. Jones demonstrate that BC is input from numerous short- and long-range atmospheric, fluvial, and tidal transport processes with the BC ultimately derived from emissions from the combustion of fossil fuels and biomass (Schmidt and Noack, 2000; Mitra et al., 2002). Such inputs have also been observed in the nearby Delaware Bay and River (Mannino and Harvey, 2004) and in aerosols on the eastern seaboard (Wozniak et al., 2011). The three study sites are located in the relatively urbanized east coast of the United States and receive airmasses that are dominantly derived from continental areas (Czarnecki et al., 2023), many of which experience anthropogenic burning and wildfires. Further, the St. Jones site is in close proximity to a highway (Delaware Route 1) and the Dover Air Force Base, and the St. Jones River watershed includes the relatively more urban city of Dover.

Rocks Tract and Roberts Farm, situated further away from major roads and not in close proximity to the Air Force Base, still have the potential for allochthonous BC inputs. While emissions from external sources may still deposit BC, the data suggests that burn history plays an important role in the observed BC concentrations not only at Roberts Farm, in particular, but also Rocks Tract more speculatively. Detailed records dating back to 2017 demonstrate the history of prescribed burns conducted at Roberts Farm (Fig. 2). Much of the BC at Roberts Farm is hypothesized to originate in situ during these burns. Since Rocks Tract is adjacent to Roberts Farm, BC transported hydrologically and atmospherically from the burns is likely to influence those soils as well.

The ratio of B6 and B5 BPCAs can provide information on the source of BC with samples showing higher B6:B5 ratios having higher relative abundances of the most highly condensed aromatic compounds (B6CA) thus logically attributed to

higher temperature combustion sources and representative of the soot end of the combustion continuum (Masiello, 2004; Wagner et al, 2017). Data for prescribed burn temperatures are scarce, and we are unaware of how hot the prescribed burns at Roberts Farm were. Previous work has shown forest fires show maximum temperatures as high as 750°C (Santin et al., 2015), but wetland fires may experience lower temperatures due to the different fuel (grasses vs. woods) and soil inundation. The expectation, therefore, is that the prescribed burns may produce chars with lower B6:B5 ratios than observed from soot materials produced with anthropogenic combustion. St. Jones, due to its proximity to the city of Dover and the Dover Air Force base (and associated jet traffic) as well as its lack of burn history, may be expected to receive the highest proportion of anthropogenic soot inputs (relative to chars). However, that site exhibits the lowest B6:B5 ratio, with a mean of 1.04 ± 0.29, a ratio that is statistically different than observed at Roberts Farm and Rocks Tract ($p < 0.001$) (Table 4) (fig. 8). Roberts Farm has a slightly higher B6:B5 ratio compared to Rocks Tract, but this difference is not statistically significant. The lower B6:B5 ratio at St. Jones suggests either that the prescribed burns in Roberts Farm are conducted at high temperatures or that the inputs to St. Jones may include more wildfire-derived material as opposed to soot.

The lack of significant difference in the B6:B5 ratios for Roberts Farm and Rocks Tract suggests that the two sites experience BC inputs of similar composition. Both sites have relatively consistent quantities of BC_{CTO}, indicating that the more condensed, soot component of the BC pool should be quantitatively similar. The significantly higher BC_{BPCA} concentration at Roberts Farm compared to Rocks Tract might suggest that the less condensed pool of BC (e.g., B5CA) should be higher at

Roberts Farm. It would be expected that the B6:B5 ratio would be lower for Roberts Farm than Rocks Tract. A possible explanation for this discrepancy lies in the quality and quantity of BC at Roberts Farm versus Rocks Tract. By excluding all BPCAs (e.g., B3CA, B4CA) except B6 and B5, the BPCA method still records a relatively condensed end of the combustion continuum, and while this excludes any artifactually-derived BPCAs (e.g., from non-pyrogenic sources), it may hinder source apportionment.

Chapter 5

CONCLUSION AND FUTURE WORK

The overarching goals of this study were to investigate if prescribed burns were increasing the BC in soils at those same environments and to advance our understanding of how BC methodology influences reported concentrations in tidal wetland environments. While BC has been investigated in these environments, few studies have utilized multiple methods to see how they differ qualitatively and quantitively.

Our results show that the Roberts Farm site with the recent history of prescribed burns had higher TOC and BC_{BPCA} concentrations than the adjacent Rocks Tract marsh. While more work should be performed to definitively attribute the increased C concentrations, these increases can preliminarily be attributed to the prescribed burns. Assuming this preliminary mechanism to be true, land managers utilizing prescribed burns for the purpose of *P. australis* removal are receiving an ecosystem service (relative to restoration that does not include burning) through the input of TOC and recalcitrant BC that is buried on site. Whether this C storage ecosystem service matches that in soils of *P. australis* stands remains to be rigorously tested.

From a BC methodological standpoint, the data obtained from the BPCA and CTO-375 methods did indeed yield differing results. The BC_{BPCA} concentrations in the study soils were consistently higher than those measured by BC_{CTO} (BC_{BPCA} mean = 4.97 ± 1.06 mg BC_{BPCA} g^{-1} soil versus BC_{CTO} mean = 1.84 ± 1.19 mg BC_{CTO} g^{-1} soil). The higher BC_{BPCA} concentrations relative to BC_{CTO} mirror results measured in terrestrial soils but conflicts with results from marine sediments. The implication is

that marsh soils being coastally-located and derived from vegetated environments more closely resemble terrestrial soils than marine sediments and receive BC derived from sources lower on the combustion continuum, that travels shorter distances, and is abundant in BPCAs.

Another interesting result from this work is the identification of a potential charring source of error in our most organic-rich samples whilst utilizing the CTO-375 method. The charring was confirmed to have occurred through the use of a dilution test that found a mean decrease in BC_{CTO} of 1.6 ± 0.9 mg BC_{CTO} g^{-1} soil. These results will be useful for researchers aiming to do BC studies in tidal wetlands. Researchers should be aware of the potential for charring in TOC-rich wetland samples and may want to 1) examine their data for TOC-BC_{CTO} correlations, and/or 2) conduct soil dilutions with inert sand as a way to test for positive artifacts due to charring.

No significant difference was found between Roberts Farm and Rocks Tract for the CTO-375 method. This may be an indication that locations adjacent to burns may be receiving similar inputs of the soot-like BC pool. BC_{BPCA} concentrations did differ between the two sites, but the B6:B5 ratios were similar suggesting similar sources and that the Rocks Tract site likely receives BC from the prescribed burns through atmospheric or hydrological transport. This information could be important to land managers wanting to see how burns not only affect the burned site but also nearby locations.

There are many directions for future work that can build on the advances made in this study. The first possible direction of work would be to dilute all of the St. Jones samples (and a subset of the Roberts Farm and Rocks Tract samples) with carbon-free sand to correct for the charring that occurred during the initial CTO-375 analyses.

Such analyses would add reliability to the St. Jones CTO-375 dataset and enable a more rigorous comparison of the soot-like BC pools amongst the 3 sites. Another direction for future work would be to analyze samples that were collected in the second sampling year to understand how stable the soil BC concentrations are in time and to test the stability of the observed trends. Along this same line of thinking, an ideal study of the effects of prescribed burns on soil C storage would be to measure soil C inventories before and for many years after prescribed burns.

A final area that could be investigated through future work is how BC varies within a site depending on burn number. Due to knowing the specific burn locations within Roberts Farm, it can be approximated if certain cores were specifically within burn boundaries. In theory, areas burned multiple times would demonstrate higher concentrations of BC and TOC due to the high char inputs. A study designed specifically to test the effects of multiple burns could provide insight into how the frequency of burns influences C sequestration.

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Appendix A

ROBERTS FARM BURN AREA DATA

There was a limited amount of data analysis conducted for burn number within Roberts Farm, but no significant difference was uncovered for BC_{CTO} or BC_{BPCA} (ANOVA $p > 0.05$). The only areas that display significant difference within the data are TOC and B6:B5 (ANOVA TOC, $p < 0.05$; ANOVA B6:B5, $p < 0.001$) (fig A1 & A2). A Tukey HSD found that only cores that experienced two burns exhibited higher TOC concentrations (ANOVA with Tukey-HSD, $p < 0.05$). Sampling sites with no burns exhibited significantly higher B6:B5 ratios than sites of 1 or 2 burns (Tukey-HSD, $p < 0.01$). Not a lot of information can be drawn from these results yet due to the limited amount of data. It could be possible to draw more conclusions after the second year of data is analyzed or if specific sites were selected within Roberts Farm to account for burn numbers.

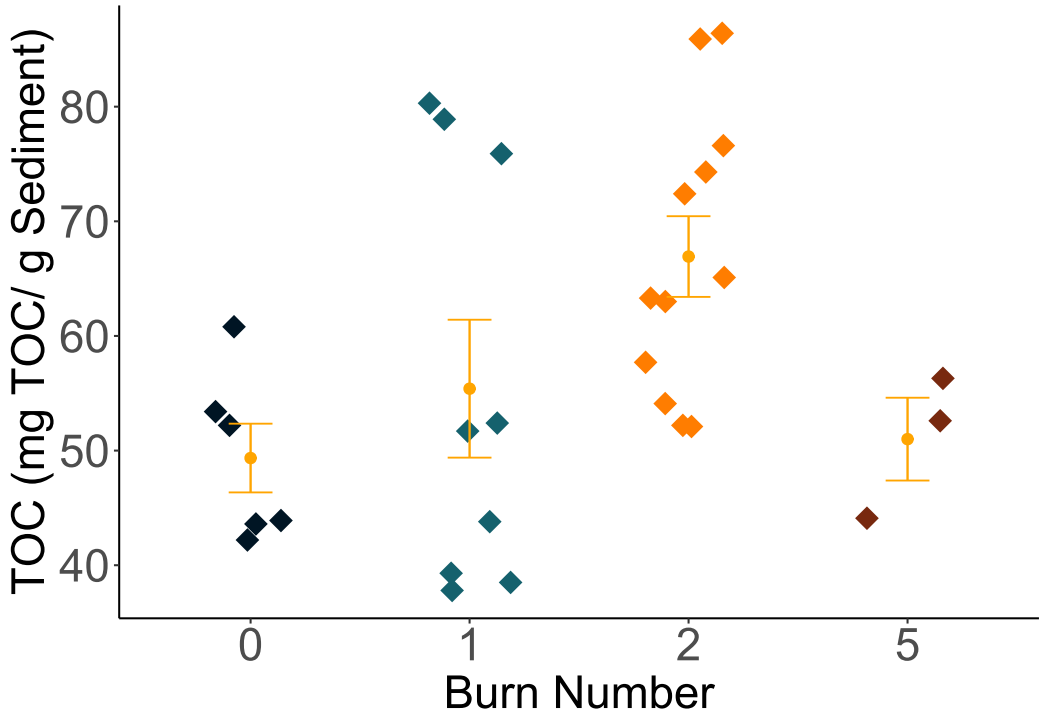

Figure A1: TOC concentrations (mg TOC g^{-1} soil) for the core sections collected at each burn number. Mean concentrations and one standard deviation are denoted by the orange diamond and error bars.

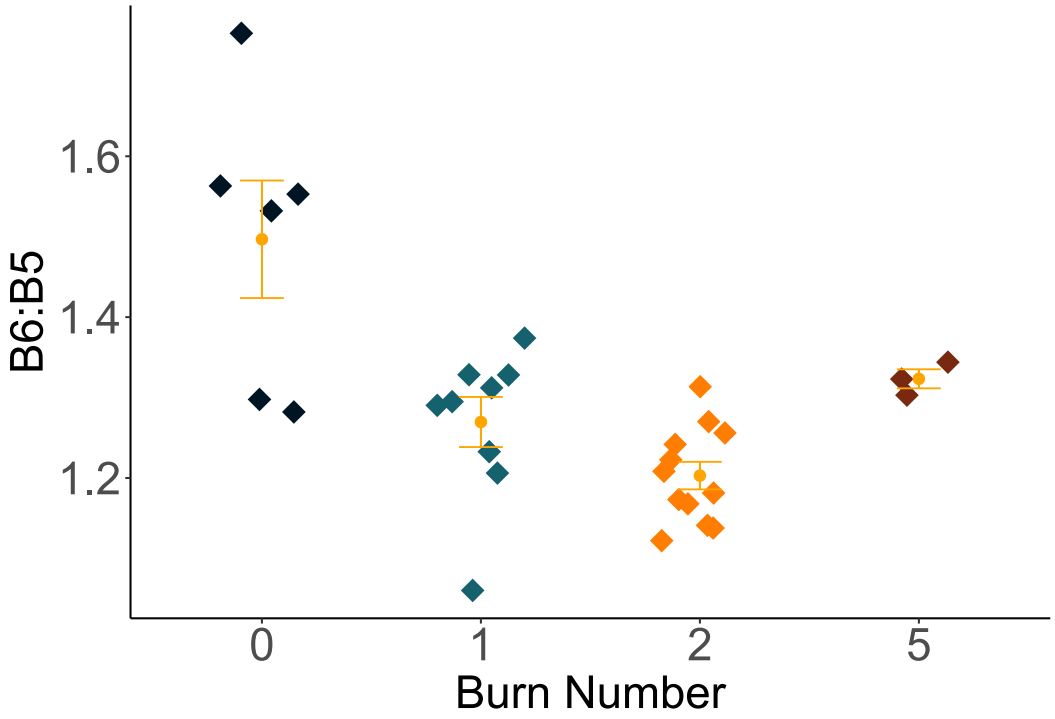

Figure A2: B6:B5 ratios for the core sections collected at each burn number. Mean concentrations and one standard deviation are denoted by the orange diamond and error bars.