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**LOSS-ON-IGNITION AS AN ESTIMATE OF
TOTAL ORGANIC CARBON IN THE SUNDARBAN
MANGROVE SOIL**



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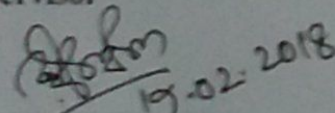


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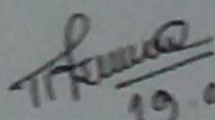
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A Project Thesis has been prepared and submitted in partial fulfillment of the
Requirement for four years professionals B. Sc. (Hons.) in Forestry.

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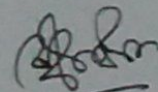

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APPROVAL

This is to certify that the present project entitled "LOSS-OF-IGNITION AS AN ESTIMATE OF TOTAL ORGANIC CARBON IN THE SUNDARBAN MANGROVE SOIL" has been carried out by Tanvir Hossain (Student Id: 130511) under my direct supervision at the Forestry and Wood Technology Discipline of Khulna University, Khulna-9208, Bangladesh.

I recommend that the content of the project report can be accepted in the partial fulfillment of the requirement for the Degree of B.Sc. (Honors) in Forestry.

Supervisor



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S. M. Rubaiot Abdullah

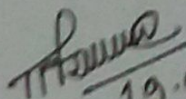
Assistant professor

DECLARATION

I, Tanvir Hossain, declare that this thesis is the result of my own work and it has not been submitted or accepted for any degree to other university or institution.

I, hereby, give consent for my thesis, if accepted, to be available for photocopying and for inter- library loans, for title, summary and outside organizations with my approval.

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19.02.2018

Tanvir Hossain

DEDICATION

To my beloved mother who always inspires me
to be an honest man from
my childhood

Acknowledgement

First of all, I would like to express my deepest gratitude to Almighty Allah for his blessings to grant me the opportunity.

I am grateful to a number of individuals who have contributed in many ways towards the completion of this thesis. Firstly, I would like to express my profound gratitude and acknowledgement to my supervisor, Assistant Professor S. M. Rubaiot Abdullah, Forestry & Wood Technology Discipline, Khulna University for his constant guidance, continual encouragement and patience since the inception of the study. I would like to thank my supervisor for his intensive discussion and constructive comments throughout the thesis with kind co-operation.

Special thanks to those people who actively participated in the successful completion of this study through their time and suggestions.

Finally, I would like to thank my family for their unconditional support and love throughout my life that encourages me to go forward. Special thanks to my friends and colleagues for their inspiration, co-operation and support to those whom I did not mention here.

Last of all, special thanks to Forest Department who conducted BFI 2016-2017 with the collaboration of FAO for providing soil samples during this study and also thanks to Nutrient Dynamics Laboratory, Forestry & Wood Technology Discipline, Khulna University, Khulna.

ABSTRACT

A simple and rapid procedure for the determination of organic matter content in soils of the Sundarbans by Loss-on-ignition and Walkley & Black method were studied. Loss-on-ignition (LOI) technique is easy, cheap and fewer workers needed as compared to chemical methods in which the soil samples were heated at high temperature (450° C) to estimate organic matter and measuring weight losses. The results were compared to those obtained by the dichromate wet oxidation method widely used in soil laboratories for organic matter determination. 10 plots with 3 layer, a total of 30 samples were taken from the Sundarbans for the study. Data transformation followed by normality test and outlier determination by studentized residuals were performed prior to regression analysis. Three linear regression models were compared considering their higher R^2 , adjusted R^2 , RMSE, SSE, Sa, Sb value. Among the three models $\ln OC_{B\&W} = 0.249 OM_{LOI} - 1.0696$ was found best fit with R^2 , adjusted R^2 0.709 and 0.698 respectively. Whereas, the residual plots of the best fit model is random shape indicating reliable fit. So, this equation can be used for getting B&W OC% from LOI OM% for the Sundarbans mangrove soil.

Keyword: Loss-on-Ignition, Wet Oxidation, Organic Carbon, Sundarbans

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List of Acronyms

SOC - Soil Organic Carbon

TOC – Total Organic Carbon

SOM - Soil Organic Matter

OC – Organic Carbon

OM – Organic Matter

LOI – Loss On Ignition

W&B – Walkley & Black

FD – Forest Department

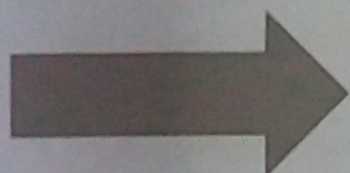
FAO – Food and Agricultural Organization

DC – Dry Combustion

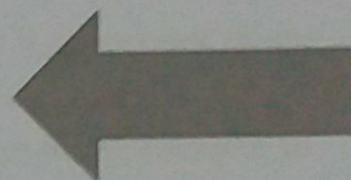
DW – Distilled Water

SRF – Sundarbans Reserved Forest

KU – Khulna University



CHAPTER ONE
INTRODUCTION



CHAPTER 1

INTRODUCTION

1.1 Introduction

Carbon is one of the most common elements in the universe and found virtually everywhere on earth: in the air, the oceans, soil, and rock. Carbon is part of geologic history in rock and especially the ancient deposits that formed coal, oil and other energy sources we use today. Carbon is also an essential building block of life and a component of all plants and animals on the planet (Corning *et al.*, 2016)

Soil has some basic physical, chemical and biological properties, which makes it functionally complete resources and interrelated. The inherent characteristics of soil which are mainly the resultant of parent material (Bezabih *et al.*, 2016).

Soil organic carbon (SOC) – the amount of carbon stored in the soil is a component of soil organic matter – plant and animal materials in the soil that are in various stages of decay. Soil organic carbon is the basis of soil fertility. It releases nutrients for plant growth, promotes the structure, biological and physical health of soil, and is a buffer against harmful substances. The amount of soil organic carbon present in soil can vary hugely according to soil and landscape types, and climate change in the same paddock over time depending on climate. Temperature, rainfall, land management, soil nutrition and soil type all influence soil organic carbon levels. Increasing soil organic carbon has two benefits – as well as helping to mitigate climate change, it improves soil health and fertility. If more carbon is stored in the soil as organic carbon, it will reduce the amount present in the atmosphere, and help reduce global warming. The process of storing carbon in soil is called 'soil carbon sequestration'. Carbon makes up approximately 60% of the Soil organic matter (SOM), with the remaining 40% of SOM containing other important elements such as calcium, hydrogen, oxygen, and nitrogen. SOM is commonly, but incorrectly used interchangeably with SOC. Soil carbon enters the soil as soil organic matter (Kirkgaard, 2007).

Several methods exist for determining soil organic carbon, and each one has its own advantages and limitations. Consequently, a comparison of the experimental results obtained when these methods are employed is hampered, causing problems in the comparison of carbon stocks in

soils. Wet combustion methods, including Walkley-Black, Mebius and Colorimetric determination as well as dry combustion methods, such as Elemental and Gravimetric Analysis were used. Quantitative textural and mineralogical (kaolinite, goethite and gibbsite) analyses were also carried out. The wet digestion methods underestimated the concentration of organic carbon, while the gravimetric method overestimated (Juliana *et al.*, 2014).

Loss on Ignition (LOI) Method carrying some importance in favor of its own. Sequential LOI is a simple method for estimating the content of organic matter. Conversion of the organic-matter content to a C basis may be difficult because the C content of organic matter is not consistent and varies with vegetation type, degree of decomposition, age, and other factors (Bath and Bauer 2002). Easy implementation in the laboratory makes the method widely used (e.g., Dean 1974, 1999; Korsman *et al.*, 1999; Dodson and Ramrath 2001; Heiri *et al.*, 2001; Boyle 2001, 2004; Bendell-Young *et al.*, 2002, and Beaudoin 2003). The method is based on differential thermal analysis.

Walkley-Black (W&B) Titration method has some important criteria. The Walkley-Black (WB) titration method is one of the classical methods for rapid analysis of organic carbon (OC) in soils and sediments. The method is based on the oxidation of organic matter by potassium dichromate ($K_2Cr_2O_7$)-sulfuric acid mixture followed by back titration of the excessive dichromate by ferrous ammonium sulfate ($Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$). This is an expensive and time-consuming process, but it is more accurate than LOI method (Vahel *et al.*, 2017).

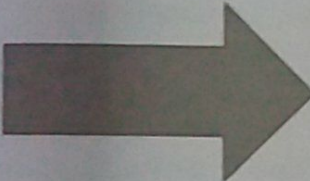
Recently Forest Department (FD) conducting Forest Inventory throughout the Bangladesh and near about 1800 sample plots were studied to determine of organic carbon content using LOI method. LOI method is conducted for estimating organic carbon. But LOI method doesn't give a correct result. But Walkley-Black (W&B) Titration Method is more convenient and accurate method.

Soils of the Sundarbans mangrove forest differ from other inland soils in that they are subjected to the effects of salinity and waterlogging, which naturally affect the vegetation. In places soils are semi-solid and poorly consolidated. pH ranges widely from 5.3 to 8.0. Organic matter content varies between 4% and 10% in dry soil. The area is the result of extensive fluvial-

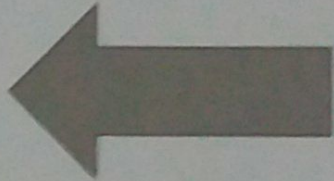
marine deposits of the river Ganges and the Bay of Bengal and the character of the sediment is silty clay. (Rahman *et al.*, 2017)

1.2 Objectives of the study

- ❖ To obtain organic carbon of soil by Walkley-Black Titration method.
- ❖ To obtain organic matter by Loss on Ignition method.
- ❖ To get a regression model to determine organic carbon by Walkley-Black Titration method from organic matter by Loss on Ignition method.



CHAPTER TWO
LITERATURE REVIEW



CHAPTER 2

LITERATURE REVIEW

2.1 Carbon and Organic Carbon

Total organic carbon (TOC) is the carbon (C) stored in soil organic matter (SOM). Organic carbon (OC) enters the soil through the decomposition of plant and animal residues, root exudates, living and dead microorganisms, and soil biota. SOM is the organic fraction of soil exclusive of non-decomposed plant and animal residues. Nevertheless, most analytical methods do not distinguish between decomposed and non-decomposed residues. SOM is a heterogeneous, dynamic substance that varies in particle size, C content, decomposition rate, and turnover time. Soil Organic Carbon (SOC) is the main source of energy for soil microorganisms (Edwards, 1999).

2.2 Carbon in Soil

Soil is the largest pool of terrestrial organic carbon in the biosphere, storing more C than is contained in plants and the atmosphere combined (Schlesinger 1997). The abundance of organic C in the soil affects and is affected by plant production, and its role as a key control of soil fertility and agricultural production has been recognized for more than a century (Dokuchaev 1883, Hilgard 1906, Jenny 1941, Tiessen *et al.*, 1994). The patterns and controls of soil organic carbon (SOC) storage are critical for our understanding of the biosphere, given the importance of SOC for ecosystem processes and the feedback of this pool to atmospheric composition and the rate of climate change (Raich and Potter 1995, Trumbore *et al.*, 1996, Woodwell *et al.*, 1998). SOC storage is controlled by the balance of C inputs from plant production and outputs through decomposition (Jenny 1941, Schlesinger 1977). In humid climates, both production and decomposition increase with temperature, but relative increases in decomposition are greater (Nakane 1975, Schlesinger 1977, Oades 1988).

2.3 Importance of carbon in soil

The carbon cycle is a fundamental part of life on earth. 'Soil organic carbon' (SOC) – the amount of carbon stored in the soil is a component of soil organic matter – plant and animal materials in the soil that are in various stages of decay. Soil organic carbon is the basis of soil fertility. It releases nutrients for plant growth, promotes the structure, biological and physical health of soil, and is a buffer against harmful substances. Soil organic carbon is part of the natural carbon cycle, and the world's soils holds around twice the amount of carbon that is found in the atmosphere and in vegetation. Organic material is manufactured by plants using carbon dioxide from the air and water. Plants (and animals, as part of the food chain), die and return to the soil where they are decomposed and recycled. Minerals are released into the soil and carbon dioxide is released into the atmosphere. (Nakane 1975, Schlesinger 1977, Oades 1988).

It is widely accepted that soils are an effective sink for atmospheric carbon. Soil organic carbon is fundamental to soil fertility.

- Decomposition of soil organic matter facilitates nutrient availability. Plant growth is nourished by the release of nitrogen, phosphorus and other nutrients. Soil organic carbon improves soil structure. (Jenny 1941, Schlesinger 1977).
- The water holding capacity and rainfall infiltration properties of organic carbon soils creates better landscape moisture availability. Root development and rainfall variation tolerance is also significantly enhanced in soils with improved aggregation from carbon. These are strong indicators of the soils biological health. Ecological soil function utilizes carbon as the initial food source. (Dokuchaev 1883, Hilgard 1906, Jenny 1941, Tiessen *et al.*, 1994).
- The type and function of soil microbes is impacted by the availability of organic carbon. A healthy soil system supports pasture nutrient uptake, assisting root growth and crop disease suppression has also been associated. Soil organic carbon is the basis of sustainable agriculture. (Raich and Potter 1995, Trumbore *et al.*, 1996, Woodwell *et al.*, 1998).

- In addition to the atmospheric benefits of carbon capture in soils, the ecology and function of agricultural systems are improved. Resulting physically cohesive soil resists soil losses by wind or water erosion. By maintaining adequate pasture and endemic flora levels the carbon cycle is sustained, promoting more pasture. (Oades 1975, Schlesinger 1977, Oades 1988)

2.4 Methods of carbon assessment in soil

There are three methods for soil C determination available in the literature:

- (i) Wet oxidation (also known as wet combustion), which consists of a partial oxidation associated with a more labile soil C pool, addition of diluted potassium dichromate with concentrated sulfuric acid, and back titration with ferrous ammonium sulfate.
- (ii) Wet oxidation and carbon dioxide (CO₂) collection and determination in a trapping solution and
- (iii) Dry oxidation, known as dry combustion (DC), where a complete C oxidation is achieved by a high-temperature induction furnace and it is the most reliable technique in acidic soils (Chaco'n *et al.*, 2002).

The standard method for the determination of carbon in soil, dry combustion, is slow unless expensive automated apparatus is available (Tabatabai, 1970). Wet combustion although requiring simpler apparatus than dry combustion, is also slow (Enwezor, 1965).

2.5 Relationship between LOI and W&B method

Soil organic carbon occurs in soil and derived from the decomposition of plants and animals. It considers as the main content of soil organic matter and has an important role in the global carbon cycle and influence negatively on the climate change. The term of the soil organic matter describes the constituents of organic matter and the carbon occurring in the soils. Wide forms of organic carbon are present in soils and begin from low decomposition forms of organic matter such as litter to high decomposition such as humus. Organic matter has a major influence on soil chemical and physical properties. Commonly the Soil organic carbon is measured by dry combustion (Loss on ignition method), or a wet acidified dichromate oxidation (Walkley and Black method). The technique of Loss-on-ignition (LOI) supplies an alternative way in

which the soil samples were heated at high temperature to ignite organic matter and measuring weight losses. This technique is easy, cheap and fewer workers needed as compared to the chemical methods. So, the LOI technique is greatly used for determining of SOM or soil organic carbon (SOC) in forest soils and sediments, and to measure carbonate in sediments. However, this technique is less used in soils of low fertility, although some studies have shown that the LOI technique gives an accurate estimate of carbonate for sediments. The standard method of W&B has been recommended for estimating the organic matter of the agricultural soil. However, there are serious issues with the routine use of this procedure. Organic carbon determined by the Walkley-Black method uses Dichromate as one of the reagents. Dichromate is Highly Toxic and Class I Carcinogens which pose a serious risk to health. Depending to Beaudoin a very few differences were observed in the estimation of SOC between LOI and W&B methods. The previous appears good estimates of SOC, but with low accuracy; in soils with low organic carbon content cannot rely on other methods used, including the W&B method. It is necessary to select the easy method for determining of (SOC) in soils of the semi-arid region and also to evaluate methods on the basis of regression relations between LOI and W&B methods. So the aim of this study was to assess two methods of (SOC) determination, as well as their comparability and compatibility, accuracy, the speed of determination and convenience, using reference soil (Dean, 1975).

2.6 Loss on Ignition method

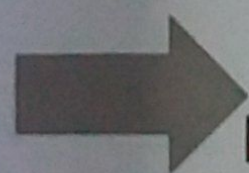
Organic matter content is an important and characteristic property of soils which is widely and routinely determined. The advantages and limitation of various methods available for such determination have been discussed by (Nelson and Sommers, 1982), who pointed out that the "loss-on-ignition" method may be considered as one of the most accurate methods since it does not involve any assumed conversion factor. It is simple, does not need highly skilled personnel and advanced apparatus, and can be conducted routinely on relatively large batches of samples. Sequential loss on ignition (LOI) is a common and widely used method to estimate the organic and carbonate content of sediments (e.g., Dean, 1974; Bengtsson & Enell, 1986). In a first reaction, organic matter is oxidized at 500°C–550°C to carbon dioxide and ash. In a second reaction, carbon dioxide is evolved from carbonate at 900–1000°C, leaving oxide. The weight loss during the reactions is easily measured by weighing the samples before and after heating

and is closely correlated to the organic matter and carbonate content of the sediment (Dean, 1974; Bengtsson & Enell, 1986). Dean (1974) evaluated the method and concluded that LOI provides a fast and inexpensive means of determining carbonate and organic contents of clay-poor calcareous sediments and rocks with precision and accuracy comparable to other, more sophisticated geochemical methods (Bengtsson & Enell, 1986), in their instructions to the technique, mention that the method gives a rough indication of the organic matter and carbonate content of sediments. Depending on the ignition temperature, various losses of volatile salts, structural water and inorganic carbon may occur (Dean, 1974; Bengtsson & Enell, 1986; Sutherland, 1998) and it is important to check the ignition temperature carefully for organic matter determination. Nonetheless, a short survey of recently published palaeolimnological studies shows that there are still substantial differences in the methods used. Exposure times vary from 1 to 4 h (e.g. Spaulding *et al.*, 1997; Henderson & Last, 1998) and ignition temperatures from 500°C to 550°C (Virkanen *et al.*, 1997; Korsman *et al.*, 1999).

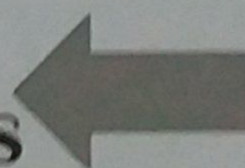
2.7 Walkley & Black Titration method

The choice of which carbon assessment method to use is a critical decision which affects the estimation of soil organic carbon (SOC) stocks and their change over time (Brye & Slaton, 2003). Many historical soil databases report SOC values analyzed by the Walkley-Black (W&B) dichromate method or one of its numerous variants. In some cases these data sets have been recently reconsidered and the locations resampled to assess temporal changes in SOC contents and stocks in the context of greenhouse gas balance assessment (Ronse *et al.*, 1988 and Lettens *et al.*, 2005 in Belgium, Bellamy *et al.*, 2005 in England, Wang *et al.*, 2003 in China). However, as instruments and analysis procedures change, methodological differences are inevitable. The difficulty is that modifications in analytical methodology, operational range limits and laboratory performance are rarely recognized and quantified in carbon accounting efforts. (Skjemstad *et al.*, 2000) determined specific W&B conversion factors for nine Australian laboratories to allow comparison of data generated over a long period. Taking dry combustion total organic carbon (TOC) as the standard, they found absolute recovery differences of up to 25% between laboratories, but also within laboratories as the W&B method was modified with time. They concluded that carbon stock assessment is inaccurate and highly tentative, unless these differences are recognized. Nelson & Sommers (1982) stated that

procedures such as W&B should be considered to give approximate or semi-quantitative estimates of SOC because of the lack of an appropriate are compared with other standardized SOC assessments (Hussain & Olson, 2000; Mikhailova *et al.*, 2003). The majority of W&B recovery studies focus on soils under agricultural management, reporting recovery differences resulting from management system (Hussain & Olson, 2000), vegetation (Diaz-Zorita, 1999), soil type (Allison, 1960) and depth (Drover & Manner, 1975). No references were found of recovery studies exclusively focusing on forest soils. The W&B method is based upon several assumptions. The most important is that carbon is the chief reducing agent present in soils and that carbon, oxidizable hydrogen and reducible nitrogen are present in the soil organic matter (OM) in about the same proportions (Walkley & Black, 1934). Concern has been raised about possible interferences by other soil constituents with dichromate. CaCO_3 was found to be without influence in dichromate titrimetric methods, but chloride, ferrous iron and higher oxides of manganese have been shown to undergo redox reactions in chromic acid mixtures leading to incorrect values for OC (Walkley, 1947). Furthermore, elemental carbon, mostly present in forest soils as charcoal as a result of forest fires or human charcoal production, influences the recovery considerably depending on the W&B method variant (Bremner & Jenkinson, 1960) and the particle size, origin and nature of the charred material (Skjemstad & Taylor, 1999), correction factor for each soil analyzed. Indeed, several studies have demonstrated the necessity to determine soils specific correction factors for W&B data sets when these data all these factors could influence a W&B determination and an attempt should be made to test their relevance for forest soil carbon assessment. The aim of this work was to (i) determine the recovery and conversion factors that are appropriate for the soils under temperate lowland forest in Flanders, Belgium, (ii) assess the lower and upper quantification limits of the original W&B method for these soils, (iii) determine the within- and between-laboratory measurement uncertainty, (iv) examine the factors determining recovery, both in the field and laboratory, and finally (v) estimate the number of samples needed to determine a laboratory- and method specific recovery factor reliably.



CHAPTER THREE
MATERIALS AND METHODS



CHAPTER 3

MATERIALS AND METHODS

3.1 Study area

The Sundarbans Reserved Forest (SRF) is situated in the extreme south-west corner of Bangladesh between the river Baleswar and Harinbhanga adjoining to the Bay of Bengal. The SRF is situated at the southern part of Khulna, Bagerhat and Satkhira civil district lying in between latitude $21^{\circ} 27' 30''$ & $22^{\circ} 30' 00''$ North, and longitude $89^{\circ} 02' 00''$ & $90^{\circ} 00' 00''$ East. (Rahman *et al.*, 2017).

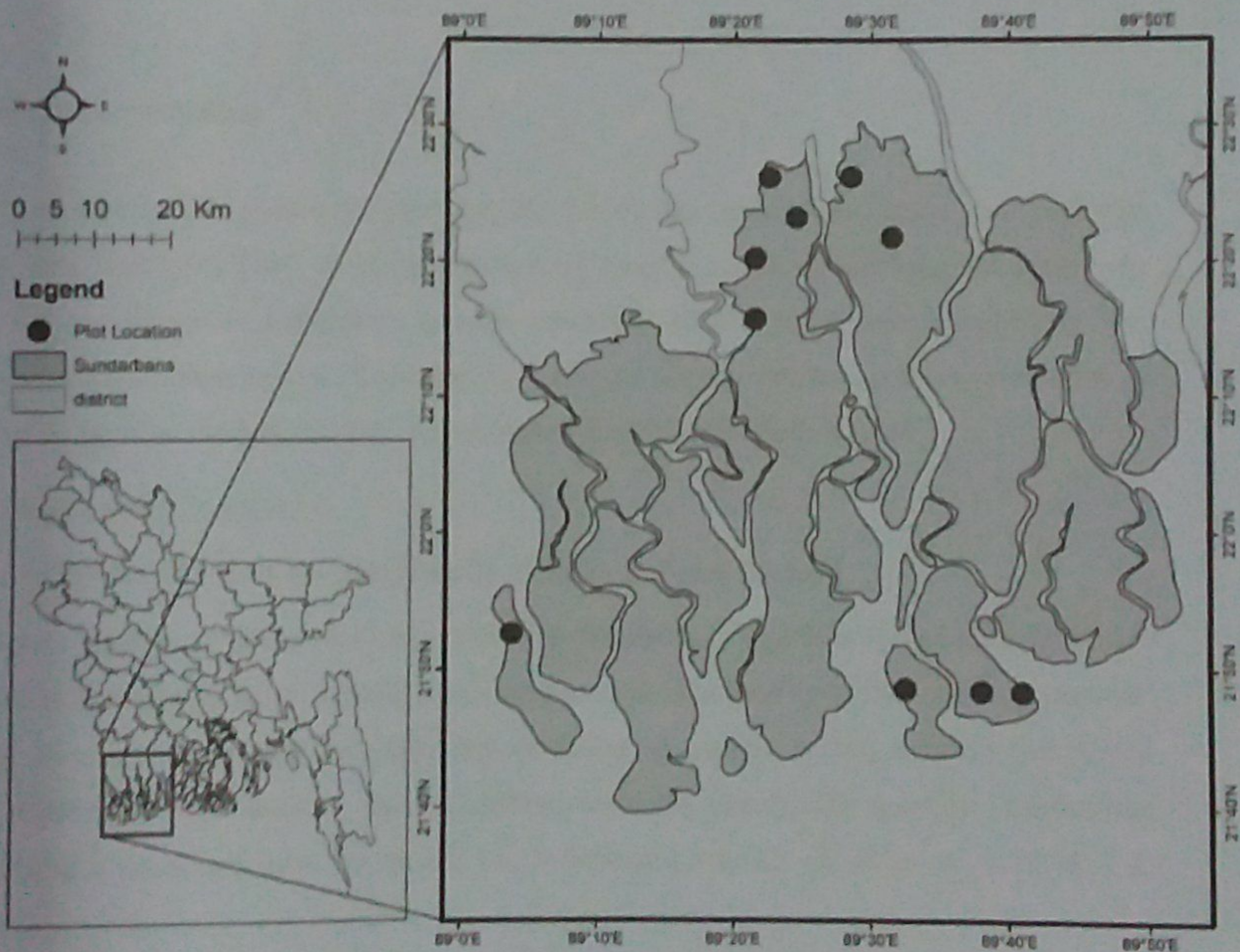


Fig 3.1: Map of the study area

3.2 Climate and Soil profile of study area

In the Sundarbans, April and May are the hottest months, while December and January are the coolest months. The mean annual maximum and minimum temperatures vary between 32°C and 20°C. Mean annual relative humidity varies from 77 to 80 %. The mean annual rainfall ranges between 1900 mm. and 2500 mm. (Rahman *et al.*, 2017).

Soils of the Sundarbans mangrove forest differ from other inland soils in that they are subjected to the effects of salinity and waterlogging, which naturally affect the vegetation. In places soils are semi-solid and poorly consolidated. pH ranges widely from 5.3 to 8.0. Organic matter content varies between 4% and 10% in dry soil. The area is the result of extensive fluvial-marine deposits of the river Ganges and the Bay of Bengal and the character of the sediment is silty clay. (Rahman *et al.*, 2017)

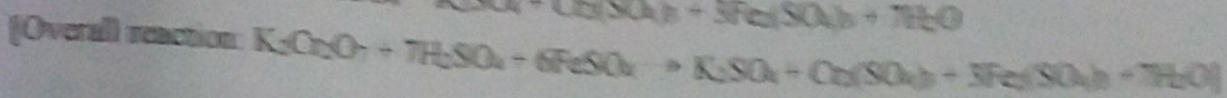
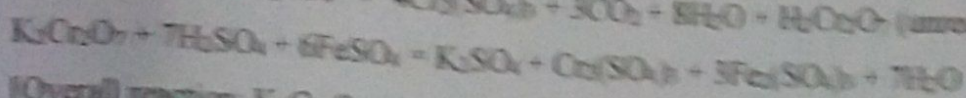
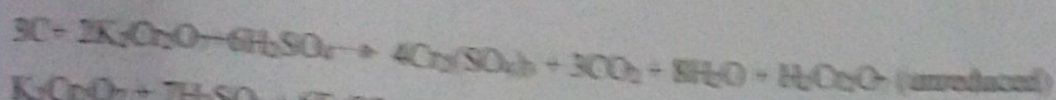
3.3 Sample collection

Approximately, 280 plots were taken by NFI, which was related to my study area. Each plot had three layers (0-15 cm / 15-30 cm / 30-100 cm). Samples including each layer were preserved at Nutrient Dynamics Laboratory, Khulna University, Khulna which were provided by NFI. There were 10 plots having 30 samples in total were taken to measure soil organic matter by Loss on Ignition method and soil organic carbon by Walkley-Black method.

3.4 Laboratory Analysis

3.4.1 Organic Matter Determination by Walkley & Black Method

Organic carbon compounds are highly reducing substances. Chromic acid ($K_2Cr_2O_7 + H_2SO_4$) is a suitable oxidizing agent to oxidize soil organic carbon according to the following reaction. The excess chromic acid left after the oxidation of organic carbon may be determined volumetrically with standard ferrous solution (reducing agent). The quantity of substance oxidized is calculated from the amount of chromic acid reduced. (Walkley, A. & Black, I.A. 1934)



Standard 1N $K_2Cr_2O_7$ was prepared by dissolving 49.033 g of $K_2Cr_2O_7$ in water and diluted to 1000 ml. Ferrous sulphate ($FeSO_4 \cdot 7H_2O$) 1N solution was prepared by dissolving 277.92 g $FeSO_4 \cdot 7H_2O$ in water and diluted to 1000 ml. $FeSO_4$ solution was filtered to remove impurities for appearing light green color. Diphenylamine indicator was made by dissolving 0.25 g indicator into 10 ml dw and 50 ml concentrated H_2SO_4 into it.

Approximately, 2.00 g dried soil was weighed out by spatula and was transferred to a 500 ml conical flask. Then 10 ml 1N $K_2Cr_2O_7$ was added by means of a pipette. After that 10 ml of concentrated H_2SO_4 was added by pipette in a fume hood and was swirled gently to mix. Because H_2SO_4 is hazardous to health. Then the mixture was allowed to cool for half an hour with occasional slight shaking (if the color of the mixture appears green then additional 10 ml of 1N $K_2Cr_2O_7$ was added. As green color indicates that all oxidizing agent added is used up to oxidize organic carbon). Next, the suspension was diluted with about 150 ml water to provide a clearer suspension. After that, 10 ml of 85% H_3PO_4 was added by a pipette, and 0.2 g of NaF (powdered) by spatula. It should be mentioned that H_3PO_4 had added normally in the laboratory. NaF had no reaction in work and it was used for color. Before final titration, a blank titration was executed without soil. And just before it, we had filtered ferrous sulphate solution to remove unused metal or dust in it appeared light green color. At last, 3 ml diphenylamine indicator was added for conducting titration. The mixture appeared deep violet in color. Excess $K_2Cr_2O_7$ solution was titrated with $FeSO_4 \cdot 7H_2O$ solution left in the flask. Finally, we have got bottle green color in mixture after titration and we had expected it.

CALCULATION

Soil organic carbon

$$1000 \text{ ml } 1N \text{ } FeSO_4 \text{ solution} = 1000 \text{ ml } 1N \text{ C} = 3 \text{ g C}$$

$$1 \text{ ml } 1N \text{ } FeSO_4 \text{ solution} = 0.003 \text{ g C}$$

$$(B-T) \text{ ml } S \text{ N } \text{FeSO}_4 = (B-T) \times S \times 0.003 \text{ g C}$$

Therefore, W g soil contains $[(B-T) \times S \times 0.003] \div W \text{ g C}$

100 g soil contains $[(B-T) \times S \times 0.003 \times 100] \div W \text{ g C}$

It has been estimated that only about 77% C of the organic compounds in soil is oxidized by normal $\text{K}_2\text{Cr}_2\text{O}_7$ solution in this method. A factor is used to correct this ($100 \div 77 = 1.3$). The constant (1.3) is called recovery factor.

$$\text{So, organic carbon in soil (\%)} = [(B-T) \times S \times 0.003 \times 1.3 \times 100] \div W$$

Where,

B = Amount of FeSO_4 required in blank titration.

T = Amount of FeSO_4 required in soil titration.

S = Strength of FeSO_4 (from blank titration)

W = Weight of soil.

Soil organic matter

The organic matter of soil is determined by multiplying the content of organic C with a factor 1.724 ($100 \div 58$) on the assumption that, organic matter of average soil contains 58% organic C by weight. This constant is called Van Bemmelen factor. Therefore, organic matter in soil (%) = % organic carbon C \times 1.724

3.4.2 Organic Carbon Determination by Loss of Ignition Method

Organic carbon of soil sample can be determined from Loss of Ignition method. The Loss of Ignition (LOI) can be determined with the following formula:

$$\text{LOI (\%)} = \frac{\text{Loss of weight after burning (g)}}{\text{Ovendry weight (g)}} \times 100$$

A total no. of 30 dry and clean Porcelain Crucible were taken as required. After that crucible cup was measured in high precision balance and records were kept. Approximately 1.00 g of

soil sample was taken approximately in the numbered Porcelain crucible cup. To make the sample free from moisture, it was dried in 105°C with a forced air oven for 1 hour. Then weight of the sample was taken along with the Porcelain crucible cup and records were kept. After that Cups were placed into the desiccator were entered into air oven and temperature was raised slowly up to 450°C. Samples were burned into muffle furnace for 4 hours until the sample was became white ash. After completion of burning, samples were cooled down and placed into a desiccator for further cooling. Then weight of the samples was measured again by balance for remaining soil ash and records were kept. At last Calculate the LOI% and Organic carbon content from the equation. (Alan *et al.*, 2008)

Calculation

$$\text{LOI OM(\%)} = \frac{W(105^\circ\text{C}) - W(450^\circ\text{C})(g)}{W(105^\circ\text{C})(g)} \times 100$$

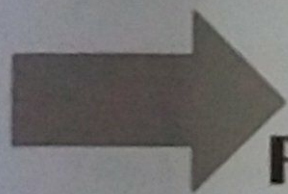
Where,

W (105°C) = Sample weight after drying at 105°C

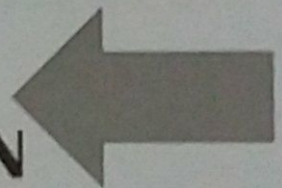
W (450°C) = Sample weight after burning at 450°C

3.5 Statistical Analysis

The normality of distribution of organic matter by Loss of Ignition and organic carbon by Walkley and Black methods for entire data sets were tested by Kolmogorov-Smirnov (K-S test). Both the data of Loss of Ignition and Walkley & Black were transformed to get normal data. Ln transformation and square root transformation were done but arc sign transformations were not conducted as it does not come from count data. Then it was decided by the normal value of P which is called significant level, and the data is normal when $P > 0.05$. Test of studentized residuals were conducted by accepted value for identifying outliers. Outlier indicates those value which is high leverage and influential. The value of studentized residual over 3 was considered as outlier. All statistical analysis was performed using SPSS-v20 and Microsoft Excel 2013.



CHAPTER FOUR
RESULT AND DISCUSSION



CHAPTER 4

RESULT AND DISCUSSION

4.1 Result

4.1.1 Normality Test and outlier determination

Kolmogorov- Smirnov (K-S) test was performed for normality test. This test indicates original data, ln transformation, square root transformation by Loss of Ignition method were significant ($P > 0.05$) and ln transformation of W&B was significant ($P > 0.05$) (Table no. 1). Square root transformation and original data of W&B were not found significant ($P < 0.05$). Regression analysis was performed with the normal data by considering original data, ln transformation, square root transformation of LOI OM and ln transformation of W&B OC as dependent variable. Three linear regression analysis among W&B ln OC vs LOI original OM, W&B ln OC vs LOI ln OM and W&B ln OC vs LOI square root OM were conducted. Prior to analysis outlier were eliminated by studentized residual and only one data was found influential outlier that was deleted from entire 30 data set.

Table 4.1: Normality test

LOI OM (Independent Variable)				W&B OC (Dependent Variable)		
Data	Statistics	df	Sig.	Statistics	df	Sig.
Transformation						
Raw Data	0.124	29	0.200*	0.198	29	0.005
Ln	0.124	29	0.200*	0.124	29	0.200*
Transformations						
Square Root	0.103	29	0.200*	0.164	29	0.044
Transformations						

(*) indicates the significant value by Kolmogorov- Smirnov normality test.

4.1.2 Regression analysis

Analysis were conducted with three model (Table 2) to obtain R^2 , adjusted R^2 , RMSE, SSE, Sa, Sb. Model No. 01 (Table 2) was found best fit because it has highest R^2 , adjusted R^2 value. At the same time it has lowest RMSE, SSE, Sa, Sb value compared to other two method.

Table 4.2: Regression analysis

Model No.	Equation	a	b	R^2	Adjusted R^2	Rmse	SSE	Sa	Sb
01.	$\ln y = a + bx$	-1.70	0.249	0.709	0.698	0.053	0.2307606	0.156	0.031
02.	$\ln y = a + b \ln x$	-1.620	1.145	0.670	0.658	0.060	0.2454573	0.244	0.157
03.	$\ln y = a + b\sqrt{x}$	-2.243	1.093	0.698	0.687	0.055	0.2348469	0.308	0.141

Note: $y = \text{OC\% W\&B}$, $x = \text{OM\% LOI}$

From those three model of regression analysis it can be said that, $\ln y = a + bx$ was accepted. So, $\ln \text{OC\% W\&B} = 0.249 \text{ OM\% LOI} - 1.70$ can be used for converting LOI OM% into W&B OC%.

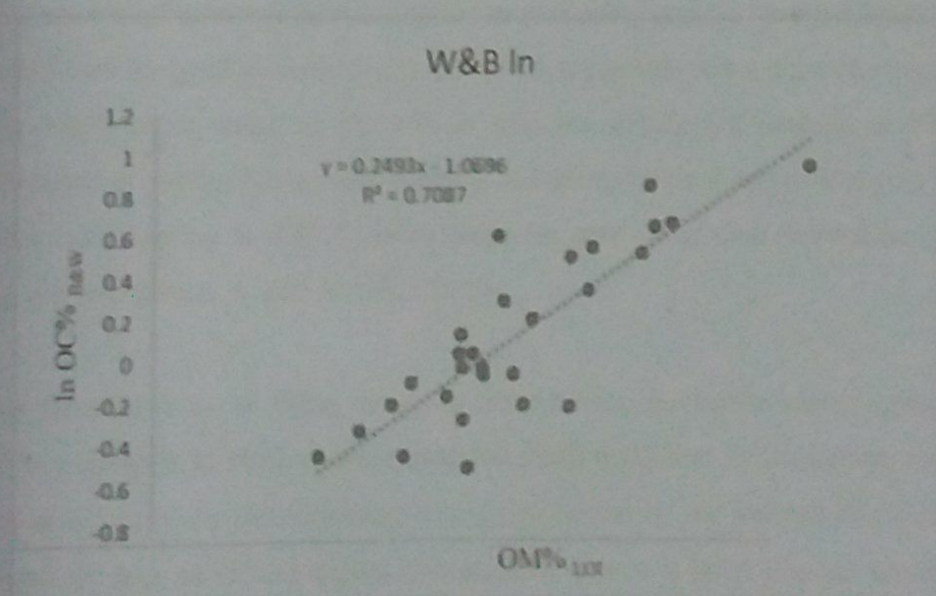


Fig 4.1: Regression model for OM% LOI and wet oxidation ln OC% B&W

4.1.3 Residual analysis

Residual plot was found in random shape. Non-random shape follows certain trend or shape and it is not good for model fit. LOI OM Residual plot did not follow any non-random shape (Fig. 4.2). So, the model may be a good fit for the data.

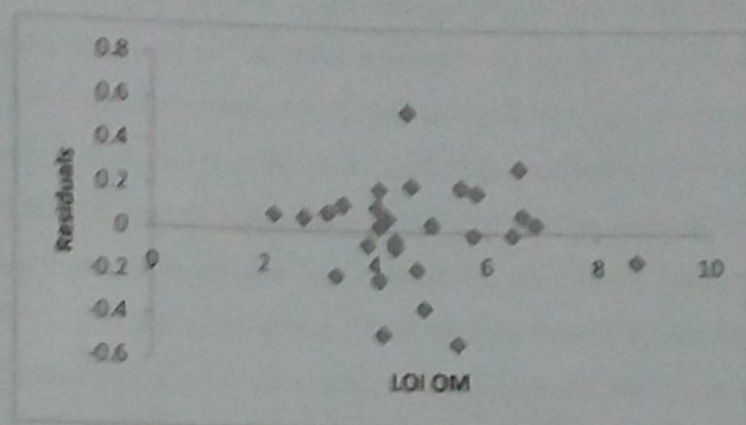


Fig 4.2: Residual plot for LOI OM% and wet oxidation in OC%

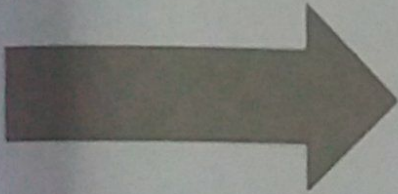
4.2 Discussion

Two major sources of error can be encountered in estimating organic matter content in soils by the proposed loss-on-ignition method. (1) Dehydration reaction is not completed at 105°C, so that some hygroscopic water of the soil is removed during the heating at 450° C. (2) Dihydroxylation of phyllosilicates and, perhaps, decarboxylation of carbonates may take place during prolonged heating at 450° C. Both processes may cause over estimation of the OM content in the soil. (Banin, A. and A. Amiel. 1969)

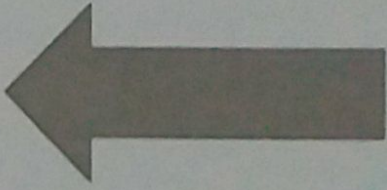
The errors introduced by all these causes in the estimate of organic matter content by the proposed loss-on-ignition Method are considered small compared to the various assumptions and errors involved in the determination of organic-carbon by the Dichromate wet oxidation (W&B) method, such as the assumption that organic carbon is at an average valance of zero and the assumption regarding the degree of completeness of oxidation. An important opinion is that the ease and technical simplicity of the proposed loss-on-ignition method may outweigh

the disadvantage of some uncertainty with regard to different soil samples from different zones (Banin and Amiel, 1969)

Loss on ignition and Walkley & Black explained 0-15 cm, 15-30 cm and 30-100 cm of the variation in soil organic carbon in regression equation in using 30 samples of mangrove soil. The relationship of soil organic carbon in loss on ignition are very weak than Walkley & Black method. The organic matter of soil samples was analyzed by the loss on ignition method that calculated values was some error because of moisture content exists in soil at 105°C but 450°C of the temperature, it does not give accurate result. Loss on ignition shown that quantitative organic determination in coastal soil in Bangladesh that can be obtained by a simple loss on ignition without any pre-treatment, for drying at 105° C for 1 hours followed loss on ignition for 4 hours at 450° C. While the Walkley & Black titration method for the determination of the organic carbon content in soil. It is most applicable method for analyzing of organic carbon. Based on the linear regression said that correlation equation is more suggested to be applied to measure organic carbon. Loss on ignition method shown very small errors that are introduced by the presence of carbonates, kaolinites and low amount of iron-oxide in soil. Walkley & Black titration is a simple technique as only basic equipment is required for the analysis and no carbon removal treatment.



CHAPTER FIVE
CONCLUSION



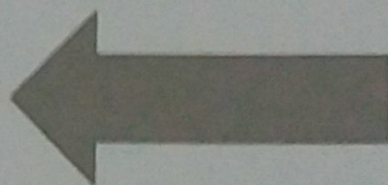
CHAPTER 5

CONCLUSION

The proposed loss on ignition procedure has the advantages of simplicity and offers a rapid alternative for routine determination of soil organic carbon from the mangrove soil. The values of LOI methods were significantly different from the values generated by W&B method. It was observed in this study that the two methods were reliable but W&B method was more suitable and accurate result in assessing of soil organic carbon. So, in $OC\%_{W\&B} = 0.249 OM\%_{LOI} - 1.70$ can be used for converting LOI OM% into W&B OC%.



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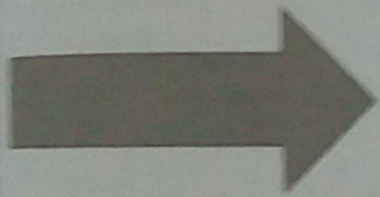
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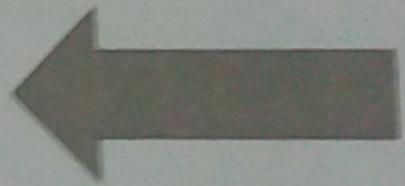
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APPENDICES



APPENDICES

7.1 W&B ln vs LOI original value

<i>Regression Statistics</i>	
Multiple R	0.841854
R Square	0.708718
Adjusted R Square	0.697515
Standard Error	0.230761
Observations	28

ANOVA

	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	3.368655	3.368655	63.26059	1.97E-08
Residual	26	1.384512	0.05325		
Total	27	4.753167			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>	<i>Lower 95.0%</i>	<i>Upper 95.0%</i>
Intercept	-1.069	0.156	-6.870	2.71E-07	-1.390	-0.749	-1.389	-0.749
LOI OM	0.249	0.031	7.953	1.97E-08	0.185	0.314	0.185	0.314

7.2 W&B ln vs LOI ln value

<i>Regression Statistics</i>	
Multiple R	0.8188
R Square	0.670434
Adjusted R Square	0.657758
Standard Error	0.245457
Observations	28

<i>ANOVA</i>					
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	3.186685	3.186685	52.89164	1.01E-07
Residual	26	1.566482	0.060249		
Total	27	4.753167			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>	<i>Lower 95.0%</i>	<i>Upper 95.0%</i>
Intercept	-1.62	0.244	-6.651	4.69E-07	-2.121	1.119	-2.121	-1.119
LOI ln	1.145	0.157	7.273	1.01E-07	0.821	1.468	0.821	1.468

7.3 W&B In vs LOI sqrt value

Regression Statistics	
Multiple R	0.83565
R Square	0.698311
Adjusted R Square	0.686707
Standard Error	0.234847
Observations	28

ANOVA

	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	3.319186	3.319186	60.18133	3.13E-08
Residual	26	1.43398	0.055153		
Total	27	4.753167			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>	<i>Lower 95.0%</i>	<i>Upper 95.0%</i>
Intercept	-2.242	0.308	-7.29	9.66E-08	-2.875	-1.611	-2.875	-1.611
LOI SQRT	1.093	0.141	7.758	3.13E-08	0.803	1.383	0.803	1.383