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Report Title: Carbon Sequestration in Reclaimed Mined Soils of Ohio

Type of Report: Progress Report for 4th Quarter

Reporting Period Start Date: 1 July 2004

Reporting Period End Date: 30 September 2004

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Date Report was issued: Month [October] Year [2004]

DOE Award No: DE-FC26-03NT41903

Name and Address of Submitting Organization: The Ohio State University, Research Foundation, 1960 Kenny Road, Columbus, OH 43210-1063

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ABSTRACT

This research project is aimed at assessing the soil organic carbon (SOC) sequestration potential of reclaimed minesoils (RMS). The experimental sites, owned and maintained by the American Electrical Power, are located in Guernsey, Morgan, Noble, and Muskingum Counties of Ohio. These sites, characterized by age chronosequences, were reclaimed with and without topsoil application and are under continuous grass or forest cover. During this quarter, water infiltration tests were performed on the soil surface in the experimental sites. Soil samples were analyzed for the soil carbon and nitrogen contents, texture, water stable aggregation, and mean weight and geometric mean diameter of aggregates. This report presents the results from two sites reclaimed during 1978 and managed under grass (Wilds) and forest (Cumberland) cover, respectively. The trees were planted in 1982 in the Cumberland site. The analyses of data on soil bulk density (ρ_b), SOC and total nitrogen (TN) concentrations and stocks were presented in the third quarter report. This report presents the data on infiltration rates, volume of transport and storage pores, available water capacity (AWC) of soil, particle size distribution, and soil inorganic carbon (SIC) and coal carbon contents. The SIC content ranged from 0.04 to 1.68% in Cumberland tree site and 0.01 to 0.65% in the Wilds. The coal content assumed to be the carbon content after oven drying the sample at 350^0C varied between 0.04 and 3.18% for Cumberland and 0.06 and 3.49% for Wilds. The sand, silt and clay contents showed moderate to low variability (CV $<$ 0.16) for 0-15 and 15-30 cm depths. The volume of transmission (VTP) and storage pores (VSP) also showed moderate to high variability (CV ranged from 0.22 to 0.39 for Wilds and 0.17 to 0.36 for Cumberland). The CV for SIC was high (0.7) in Cumberland whereas that for coal content was high (0.4) in the Wilds. The steady state infiltration rates (i_c) also showed high variability (CV $>$ 0.6) and ranged from 0.01 to 0.98 cm min^{-1} in Cumberland and 0.1 to 1.68 cm min^{-1} in Wilds. The cumulative

infiltration (I) was highly variable (CV > 0.6) and ranged from 4.2 to 110 cm in Cumberland and 17.4 to 250 cm in Wilds. The AWC for 0-15 cm depth also showed moderate variability (CV = 0.3) for Cumberland but high for Wilds (CV = 0.4).

The sand and silt contents showed strong spatial dependence with nugget-sill ratio of 15 and 23%, respectively with a range of 50 m in Cumberland site. Strong spatial dependence for sand content was also obtained for Wilds. The VSP, AWC, I, clay content, VTP, and i_c, showed moderate to low spatial dependence (nugget-sill ratio varied from 32 to 72% in Cumberland and 37 to 88% in Wilds). These preliminary results along with those reported earlier during the third quarter suggest that the management effects are important and indicative of these sources of variability.

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1.0 **Executive Summary**

This research project is aimed at assessing the soil organic carbon (SOC) sequestration potential of reclaimed minesoils (RMS) and is supported by US Department of Energy- National Energy Technology Laboratory. The proposed research focuses on: (1) assessing the sink capacity of RMS to sequester SOC in selective age chronosequences, (2) determining the rate of SOC sequestration, and its spatial (vertical as well as horizontal) and temporal variations, (3) developing and validating models of SOC sequestration rate, (4) identifying the mechanisms of SOC sequestration in RMS, (5) assessing the potential of different methods of soil reclamation on SOC sequestration rate, soil development, and changes in soil mechanical and water transmission properties, and (6) determining the relation between SOC sequestration rate, and soil quality in relation to soil structure and hydrological properties.

Before 1972, surface mining operations were performed by removing the soil and underlying strata and piling them on a side. After mining operations were complete, the excavated area was planted to trees or grass without grading or reclamation due to the nonexistence of any specific guidelines. After 1972, Ohio Mineland Reclamation Act (also 1977 SMCRA) made it mandatory to grade the area back to its original topography and reclaim it with topsoil application prior to sowing grass or planting trees. In this project, several experimental sites were identified, which were reclaimed both prior to SMCRA regulation (without topsoil under grass or forest) and after (with topsoil under grass or forest). All these sites, characterized by distinct age chronosequences of reclaimed minesoil, are located in Guernsey, Morgan, Noble, and Muskingum Counties of Ohio, and are maintained and owned by the American Electrical Power.

Water infiltration tests were conducted between 1st July and 15th September, 2004. During this period, soil analysis was carried out in the lab to determine the particle size distribution, soil organic, inorganic and coal carbon contents, soil moisture characteristic curves, water stable aggregation, and mean and geometric mean diameter of aggregates. This report presents the results of soil inorganic carbon (SIC) and coal carbon concentrations, soil texture, available water capacity (AWC) of soil, volumes of storage pores and transmission pores, steady state infiltration and cumulative infiltration from two sites reclaimed in 1978 with topsoil application (Wilds and Cumberland). The statistical variability was assessed by descriptive statistics, and variations in soil properties were expressed by ranking the coefficient of variation (CV) into different classes. The geostatistical variability was assessed by the regionalized variable theory and using *Variowin* and ArcGIS geostatistical Analyst, variograms of each soil property were computed. Distinct classes of spatial dependence for soil variables were obtained by the ratio of the nugget to the sill value.

The sand, silt and clay contents showed moderate to low statistical variability (CV < 0.16) for 0-15 and 15-30 cm depths for both Cumberland and Wilds sites. The SIC content was 0.8±0.5 % in Cumberland and 0.1±0.02% in wilds. Both SIC and coal carbon content showed high variability for both sites. The volume of transmission (VTP) and storage pores (VSP) measured on intact soil cores also showed moderate to high variability (CV ranged from 0.22 to 0.39 for Wilds and 0.17 to 0.36 for Cumberland). The steady state infiltration rates (i_c) and cumulative infiltration (I) were 0.52±0.4 and 0.32±0.2 cm min-1 for Wilds and Cumberland, respectively and showed high

variability (CV > 0.6) for both sites. The AWC for 0-15 cm soil also showed moderate statistical variability (CV = 0.3) for Cumberland but high for Wilds (CV = 0.4). The sand and silt contents showed strong spatial dependence with nugget-sill ratio of 15 and 23 %, respectively with a range of 50 m in Cumberland site. Strong spatial dependence for sand content was also obtained for Wilds. The VSP, AWC, I, clay content, VTP, and i_c, showed moderate to low spatial dependence (nugget-sill ratio varied from 32 to 72% in Cumberland and 37 to 88% in Wilds). These preliminary results along with those reported earlier for the third quarter suggest that the management effects are important and indicative of these sources of variability. The determination of SIC and coal carbon contents are important for correct assessment of SOC pools and the knowledge of actual sequestration rates.

2.0 Experimental

2.1 Experimental Sites:

The two experimental sites were reclaimed in 1978 in conformity with the Ohio Mineland Reclamation Act of 1972 with topsoil application and were under grass cover (Wilds) and forest cover (Cumberland) (Fig. 1). Trees were planted at the Cumberland tree site during1982. Both experimental sites are owned by American Electric Power (AEP) Co. and both sites were reclaimed in 1978.

2.2 **Analysis of Soil Samples**

2.2.1 **Particle size distribution**

The air-dried samples were passed through wooden rollers to break up the remaining clods (Gee and Bauder, 1986) and the material coming out of the rollers was passed through a 2- mm sieve. About 51 g of sieved soil sample (< 2mm) was used for the determination of particle size distribution by the hydrometer method using distilled water at room temperature of about 22^{o}C (Gee and Bauder, 1986).

2.2.2 **Soil Organic Carbon Concentrations**

During the fourth quarter, the remaining air-dried soil samples for carbon-nitrogen analysis from each depth and site were ground separately to pass through 0.25 mm sieve. About 1 g of the soil was used for the determination of total carbon (TC) and total nitrogen (TN) concentrations by the dry combustion method (Elementar, GmbH, Hanau, Germany).

2.2.3 **Soil Inorganic Carbon and Coal Carbon Contents**

The SIC content was determined using the procedure of Bundy and Bremner (1972) with some modifications. Briefly, 1.5 to 2 g of soil (< 2 mm) was weighted in a serum bottle that was crimp-sealed. A glass syringe was then used to inject 4- mL HCl (2 M) into the bottle. The carbon dioxide (CO_2) produced was injected into gas chromatograph (Shimadzu, GC 14A). Using a thermal conductivity detector, the concentration of CO_2 was obtained and was converted to SIC content. The coal carbon content was determined by adding 4 ml of 6 N HCl to 2 g of soil (< 2 mm), the suspension was stirred and left for 6 hours. The supernatant was washed with deionized water and after draining the extra water, it was dried in an oven under 350^{o}C for 24 hours. The carbon content of this sample was determined by the dry combustion method (Elementar, GmbH, Hanau, Germany) and was assumed to represent the coal carbon content of soil.

2.4. **Statistical Analysis**

Descriptive statistics including mean, standard deviation, CV, maximum, minimum, skewness, and kurtosis were obtained for each measured soil variable using the Statistical Analysis System (SAS Institute, 1989). All measured soil physical and chemical properties data were tested for normality. Using *Variowin* (Pannatier, 1996) and ArcGIS geostatistical Analyst (ESRI, 2004), variograms of each soil physical property and cross-variograms were obtained. The spherical models were fitted to the variograms (Fig 2):

$$\begin{aligned} \gamma(h) &= C_0 + C_1\left[\frac{3h}{2a} - \frac{h^3}{2a^3}\right] && \text{for } h \le a \\ &= C_0 + C_1 && \text{for } h \ge a \end{aligned} \qquad (1)$$

where C_0 is nugget, h is lag distance and a is range of spatial dependence to reach the sill (C_0 + C_1).

Variations in soil properties were expressed by ranking the CV into different classes: least (<15%), moderate (15 to 35%) and most (>35%) (Wilding, 1985; Shukla et al., 2004). Distinct classes of spatial dependence for soil variables were obtained by the ratio of the nugget to the total sill value (NSR). The variable was considered strongly spatially dependent when the NSR was $\leq$ 25%, moderately spatially dependent for 25% < NSR < 75% and weakly spatially dependent for the NSR of $\geq$ 75% (Cambardella et al., 1994).

3.0 **Results and Discussion**

3.1 **The Variability of Soil Properties**

Tables 1 to 4 list the descriptive statistics of the original data from Cumberland and Wilds including mean, median, coefficient of variation, skewness, kurtosis, maximum and minimum values for 0-15, and 15-30 cm depths, respectively. Despite some skewness in the data for sand, silt and clay contents, the mean and median values for all these parameters were similar and median was either equal to or smaller than the mean for most of the parameters and data were normally distributed (Tables 1 to 3). The standard error of the mean for sand and clay contents was higher for 15-30 than 0-15 cm depth for both sites. The particle size analysis showed that 20.2±3.5 % composed sand, 60.3 ±4.2%silt, and 20±1.4 clay for Cumberland and 22.2±3.2%, 57.2±3.2, and 20.6±1.6%, respectively for Wilds. The variability was moderate in sand content (CV~0.17) and low in silt and clay contents for both sites for 0-15 cm depth (~7%). Minor differences in silt and clay contents were observed among 0-15 and 15-30 cm depths (Table 1).

The SIC and coal carbon content values showed high skewness for both sites and mean and median values indicated that the data were not normally distributed. The SIC content ranged from 0.01 % to 1.62% in Cumberland and 0.01 to 0.65 % in Wilds for 0-15 cm depth. The coal carbon content varied between 0.04 and 1.68 % in Cumberland and 0.06 and 3.49% in Wilds for the 0-15 cm depth. The coefficient of variation was high (CV > 0.35) for both SIC and coal carbon contents for both sites (Table 2).

The mean and median values for VTP, VSP and AWC were fairly similar for both sites and except for the AWC for wilds, the variability was moderate (0.17< CV< 0.36). The VTP was slightly higher for Cumberland (0.11±0.02) than Wilds (0.09±0.02). However, VSP and AWC were lower for Cumberland (0.14±0.05; 1.87 ±0.67 cm) than Wilds (0.20±0.08; 2.65±1.18) (Table 3). The mean and median infiltration rate and cumulative infiltration were more similar for Cumberland than Wilds Site. The coefficient of variation was high (CV > 0.58) for both sites (Table 4). The mean i_c and I were higher for Wilds (0.52±0.38 cm min^{-1}; 88.17±52 cm) than Cumberland (0.32±0.20 cm min^{-1}; 53.15±52 cm.

3.2 **Spatial Variability In Soil Properties**

The measured soil properties showed differences in their spatial pattern in both Cumberland and Wilds sites. The spatial dependence showed an isotropic behavior, which can be due to the low variability in soil management treatments and soil forming factors for the study area. Several different models were fitted to the variogram and the spherical model (Eq. 1) was the best with least sum of squares (Fig. 2). There was no anisotropy evident in the directional semivariograms for any soil property. Therefore, isotropic models were fitted using *Variowin*. All variogram models of sand, silt, and clay contents, AWC, VSP, VTP, i_c, and I showed a positive nugget effect, which may be explained as the sampling error, random and inherent variability, or shorter-range variability of soil properties than the chosen grid size of 20 x 20 m. The relative size of nugget effect among different soil properties is described by expressing the nugget variance as a percentage of total semivariance or total sill (Trangmar et al., 1985).

For Cumberland tree site, the nugget- sill ratio or NSR of 15% for Cumberland and 19% for Wilds showed strong spatial dependence for sand content in the 0-15 cm depth. The silt content with NSR of 23% showed strong spatial dependence for Cumberland site only. The AWC, VTP, VSP, I and i_c showed moderate spatial dependence (32 to 72% for Cumberland site and 37 to 88 for Wilds site) (Table 6).

4.0 **Conclusions**

The preliminary results of descriptive statistics show that most of the data were normally distributed. The statistical variability was moderate to low in most soil properties including particle size. However, water transmission properties and inorganic and coal carbon contents showed high variability for both sites. Sand content showed strong spatial dependence for 0-15 cm depth from both sites. However, silt and clay contents showed moderate to weak spatial dependence for both sites. The spatial variability for the combined data also showed moderate to low spatial variation. These preliminary results suggest that spatial variability exists in the area and that an explicit recognition of these sources of variability is essential for designing any site specific management practice and the correct estimation of SOC sequestration rates.

5.0 **Tasks to be performed in the next Quarter (October- December 2004)**

1. Water stable aggregation analysis
2. Determination of hydrolyzable, recalcitrant and coal carbon content for the remaining sites
3. Developing relationship between SOC and other soil physical and chemical properties
4. Statistical and geostatistical analysis for data on soil physical and chemical properties

6.0 **References**

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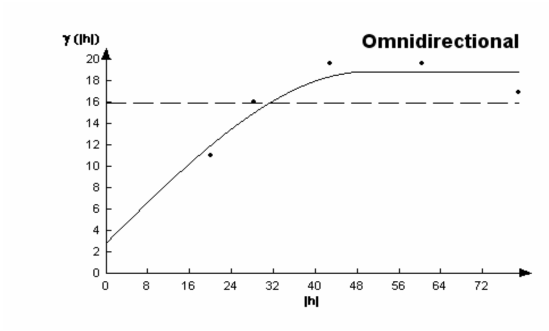
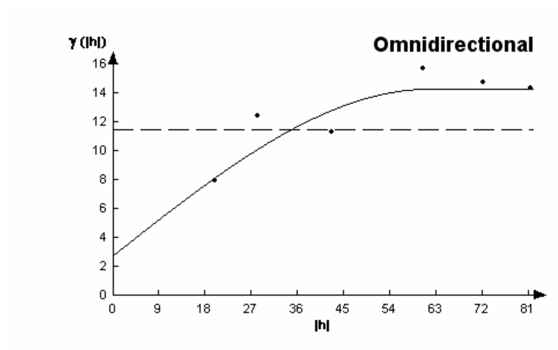
A. Wilds grass site

B. Cumberland tree site

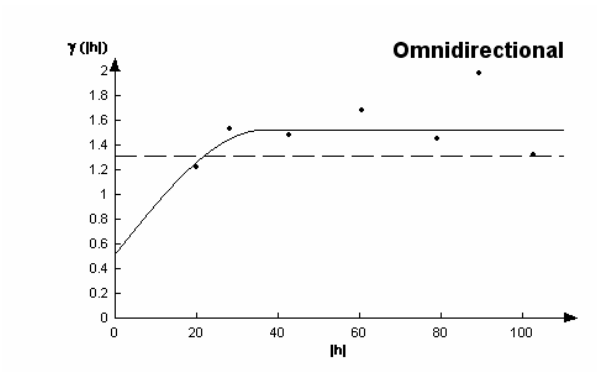
Fig. 1. Experimental sites reclaimed in year 1978: (A) under grass, and (B) under forest cover



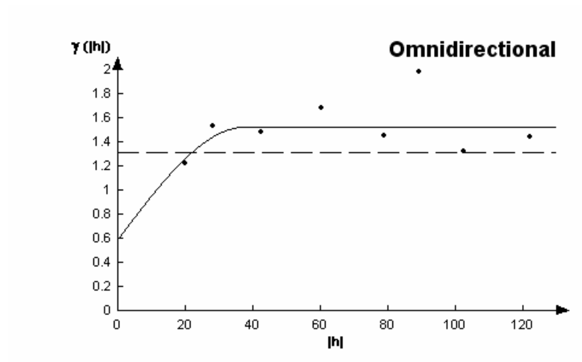
A. Sand content in 0-15 cm depth



B. SOC stock in 15-30 cm depth



C. AWC in 0-15 cm depth



D. AWC in 0- 15 cm depth

Fig. 2. Sample variograms for 0-15 cm depth for sand content (A) for Cumberland and (B) for Wilds, and Available water content (AWC) (C) for Cumberland and (D) for Wilds.

Table 1. Summary statistics for particle size analysis for the Cumberland site under forest and Wilds grass site for 0-15 and 15-30 cm depths. Both sites were reclaimed in 1978. Note: sand, silt and clay are contents expressed as %.

	Cumberland			Wilds		
	Sand	Silt	Clay	Sand	Silt	Clay
			0- 15 cm depth			
Mean	20.20	60.27	19.53	22.23	57.20	20.57
Median	21.84	59.00	19.16	22.56	57.14	20.44
Std Error	0.67	0.76	0.25	0.64	0.58	0.29
Std Dev	3.53	4.17	1.35	3.51	3.16	1.61
CV	**0.17**	**0.07**	**0.07**	**0.16**	**0.06**	**0.08**
Skewness	-0.93	1.09	-0.82	-0.12	0.56	0.24
Kurtosis	0.45	1.47	1.37	-0.26	-0.16	-0.37
Minimum	11.84	52.00	16.16	14.56	52.28	17.44
Maximum	25.84	72.00	22.16	29.28	65.00	24.44
			15-30 cm depth			
Mean	19.27	64.32	16.41	22.23	58.67	19.11
Median	18.70	64.64	16.66	22.56	59.00	19.44
Std Error	0.95	0.47	0.48	1.45	1.45	0.33
Std Dev	1.90	0.94	0.96	2.52	2.52	0.58
CV	**0.10**	**0.01**	**0.06**	**0.11**	**0.04**	**0.03**
Kurtosis	-0.07	1.09	-1.29	-	-	-
Skewness	1.09	-1.33	-0.85	-0.59	-0.59	-1.73
Minimum	17.84	63.00	15.16	19.56	56.00	18.44
Maximum	21.84	65.00	17.16	24.56	61.00	19.44

Table 2. Summary statistics for soil inorganic carbon (SIC) and coal carbon contents in both sites for 0-15 cm depth. Note both contents expressed as %.

	Cumberland		Wilds	
	SIC	Coal	SIC	Coal
Mean	0.99	0.82	0.12	0.47
Median	1.25	0.92	0.03	0.26
Std Error	0.11	0.10	0.03	0.21
Std Dev	0.60	0.56	0.17	0.83
CV	0.61	0.69	1.42	1.76
Skewness	-0.75	0.04	1.88	3.65
Kurtosis	-1.21	-1.39	3.18	13.92
Range	1.61	1.65	0.64	3.43
Minimum	0.01	0.04	0.01	0.06
Maximum	1.62	1.68	0.65	3.49

Table 3. Summary statistics for volume of transport and storage pores (VTP and VSP) properties for the Cumberland site under forest for 30-50 cm depth. The site was reclaimed in 1978. Note: VTP and VSP are expressed as % and AWC as cm.

	Cumberland			*Wilds*		
	VTP	VSP	AWC	VTP	VSP	AWC
Mean	0.11	0.14	1.87	0.09	0.20	2.65
Median	0.11	0.14	1.92	0.09	0.18	2.44
Std Error	0.00	0.01	0.11	0.00	0.01	0.19
Std Dev	0.02	0.05	0.67	0.02	0.08	1.18
CV	0.17	0.32	0.36	0.22	0.39	0.44
Skewness	-0.29	0.11	0.10	-0.17	1.63	1.56
Kurtosis	1.67	-0.06	-0.08	-0.22	3.79	3.50
Minimum	0.05	0.06	0.54	0.04	0.09	0.94
Maximum	0.15	0.26	3.52	0.13	0.48	6.84

Table 4. Summary statistics for steady state infiltration rate (i_c), cumulative infiltration (I) and available water content for both sites. Note: i_c is expressed in cm min^{-1} and I in cm.

	CUMBERLAND		WILDS	
	i_c	I	i_c	I
Mean	0.32	53.15	0.52	88.17
Median	0.34	52.40	0.41	81.20
Std Error	0.04	5.66	0.07	9.56
Std Dev	0.20	30.98	0.38	52.36
CV	0.61	0.58	0.74	0.59
Skewness	3.16	-0.61	1.65	1.21
Kurtosis	1.05	0.37	2.39	2.05
Minimum	0.01	4.20	0.10	17.40
Maximum	0.98	110	1.68	250

Table 5. The spatial variability of soil particle sizes for Cumberland and Wilds

Property	Model	SS	Nugget	Range (m)	Partial Sill	Nugget/Sill
		Cumberland				
Sand	Spherical	0.010	2.88	49.8	16	15
Silt	Spherical	0.015	5.88	50.4	19.32	23
Clay	Spherical	0.037	1.45	19.8	0.902	62
		Wilds				
Sand	Spherical	0.032	2.76	61.5	11.52	19
Silt	Spherical	0.190	3.14	32.5	7.252	30
Clay	Spherical	0.031	1.77	19.8	1.421	55

Where SS is sum of squares, NSR is nugget-sill ratio (%)

Table 6. The spatial variability of water transmission properties of soil for Cumberland and Wilds

Property	Model	SS	Nugget	Range (m)	Partial Sill	Nugget/Sill
		Cumberland				
AWC	Spherical	0.016	0.52	36.3	1.008	34
VSP	Spherical	0.019	0.002	41.8	0.0043	32
VTP	Spherical	0.053	0.0002	22.5	0.0001	67
i5	Spherical	0.018	0.16	55	0.248	39
ic	Spherical	0.049	0.03	26.4	0.0108	72
I	Spherical	0.010	530.1	79.05	641.7	45
		Wilds				
AWC	Spherical	0.018	0.588	36.3	1.01	37
VSP	Spherical	0.019	0.00	41.8	0.004	41
VTP	Spherical	0.068	0.00	22.5	0.001	33
i5	Spherical	0.013	1.225	55	0.248	83
ic	Spherical	0.030	0.078	26.4	0.011	88
I	Spherical	0.045	810	79.05	641.7	56