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RELATIONSHIPS BETWEEN SOIL WATER REPELLENCY, PHYSICAL AND CHEMICAL PROPERTIES IN HYDROPHOBIC TECHNOGENIC SOILS FROM THE REGION OF MARITSA-IZTOK COAL MINE IN BULGARIA

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Abstract

Atanasova, I. M. Banov, T. Shishkov, Z. Petkova, B. Hristov, P. Ivanov, E. Markov, I. Kirilov and M. Harizanova, 2018 relationships between soil water repellency, physical and chemical properties in hydrophobic technogenic soils from the region of Maritsa-Iztok coal mine in *Bulgaria. Bulg. J. Agric. Sci.*, 24 (Suppl. 2): 10-17

Soil water repellency (soil hydrophobicity) is considered a key mechanism for sequestration of organic carbon. Technogenic soils from mine areas containing clays and irregularly distributed lignitic particles are heterogeneous materials exhibiting small-scale spatial variability of water repellency. Non-vegetated and pine-afforested spoils from the area of Maritsa-Iztok lignite coal basin in Bulgaria were studied. The technogenic soils were characterized by severe to extreme hydrophobicity and heavier texture at the pine-vegetated site, as well as extreme acidity (pH 3-4). Principle Component Analysis (PCA) and cluster analysis were carried out, in order to study the simultaneous interaction of soil characteristics and properties with the aim to assess their role in the overall data variability in the process of data reduction to several unrelated components. The PCA was based on twelve factors: WDPT, sand, silt and clay contents, hygroscopic moisture, cation exchange capacity (CEC), organic carbon (total organic carbon TOC, humic organic carbon HOC and fulvic organic carbon, FOC), total nitrogen (N) and mineral nitrogen (MN) and electrical conductivity (EC). Three principle components were identified with eigenvalue > 1, describing 79% of the total variability. There was a significant positive correlation between WDPT and TOC, HOC, FOC, MN and a negative correlation with the % of hygroscopic moisture. The results obtained indicate that TOC comprised mainly of particulate organic carbon (POC) containing coal particles was significantly correlated with the sand fraction, CEC and MN, and was the main driver of soil water repellency in the studied mine soils.

Key words: soil water repellency, technogenic soil, mine, soil properties, principal component analysis

Abbreviations: WDPT – Water Drop Penetration Time, OC – Organic Carbon, CEC – Cation Exchange Capacity
SWR – Soil Water Repellency, SOM – Soil Organic Matter, TOC – Total Organic Carbon, HOC – Humic Organic Carbon, FOC – Fulvic Organic Carbon, EC – Electrical Conductivity, MN – Mineral Nitrogen, PCA – Principle Component Analysis, CA – Cluster Analyses, PC – Principal Components

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Introduction

It has been widely accepted that restoration of disturbed mine lands should become an imperative practice in many countries following mining activities for ecological, social and economic reasons (Frouz, 2013). The reclamation of land disturbed by coal mining activities involves converting the infertile and often toxic spoils into self-sustaining lands. A widely accepted practice in reclamation activities requires the removal and reinstallment of topsoil from the pre-mining environment to ensure return of productivity of the affected land. A widely accepted reclamation method is through forestation (Haigh and Gentcheva-Kostadinova, 2007, Krümmelbein et al. 2012).

In the Maritsa-Iztok coal producing region reclamation into forestry dominated landscapes has been applied as a common practice since 1970s (Treykyashki, 1987) by depositing clay strata in landfills and reinstallment of humus rich soil layer on top of the spoils. Another way of land reclamation has been to mix coal ash, a waste product from the thermal power plant with geological overburden layers containing yellow, green, blue and black clays deposited in depth of the geological profile over the coal seams that have been excavated in the process of coal production. Tree planting (*Pinus nigra* L.) and ash treatment has been shown to increase substrate pH and improve soil chemical and physical properties (Zheleva et al. 2004). The effects of excavated overburden mine wastes can be multiple, e.g. soil erosion and air and water pollution, metal toxicity, loss of biodiversity, and eventually loss of economic wealth (Wong, 2003; Sheoran et al., 2010). Lignitic technogenic soils exhibit spatial variability of water repellency (hydrophobicity) due to partial mixing of different overburden sediments and irregular distribution of lignitic particles, lignite-coated sand, clay particles and influence water and solute movement in reclaimed soils (Gerke et al. 2001). Water repellency in soils is associated with the formation of hydrophobic layers of organic compounds on soil particle surfaces and/or interstitial hydrophobic particulate organic matter (Morley et al., 2005). These compounds are thought to originate from plant leaf waxes, decomposing organic matter, root exudates, and their biodegradation products or from microbial activity (Hallet et al. 2006). The effect of high soil temperature on water repellency has also been investigated in laboratory-based studies and is well established (Atanassova and Doerr, 2011). Organic compound classes thought to be associated with soil water repellency include alkanes, fatty acids, fatty alcohols, aldehydes, ketones and ω-hydroxy fatty acids and α,ω-dicarboxylic acids characteristic of roots (Morley et al., 2005; Atanassova and Doerr, 2010, 2011; Mao et al. 2015). The degree of water repellency varies with moisture content and temperature (Dekker and Ritsema, 1994; De Jonge et al., 1999).

The major aims of the present study are: (i) to assess various chemical and physical soil properties of vegetated and non-vegetated water repellent (hydrophobic) technogenic soils in the vicinity of the biggest coal producing complex in Bulgaria, and (ii) to get information about the interrelationships between the persistence of soil water repellency and various soil characteristics, therefore outline groups of characteristics of similar sources or origin, as well as those specifically related to soil hydrophobicity.

Materials and Methods

The study was carried out on two plots separated by a distance of 30 m from each other at the village of Obruchishte, Maritsa-Iztok coal mines. The experimental plots were located in a ~ 1 ha large area, which was afforested with *P. nigra*. The spoils were created in 1970s and consisted of loam-textured Pliocene overburden sediments from the nearby open-cast lignite mine Troyanovo 1, GPS coordinates (UTM-system): Site 1: N 42.16434, E 25.94285, and Site 2: N 42.16452, E 25.94318. A lack of plant density and water repellency was observed in plots of ~ 200 m^2 amongst a uniformly pine vegetated area. Grids Δ 2 m, ~ 40 m^2 were constructed at a bare non-vegetated and another pine vegetated sites, ~ 40 m apart from each other. Soil cores were taken to a depth of 0-10 (0-5) and 10-20 cm and at two points to a depth of 0-50 cm using a 3 cm wide and 25 cm long core sampler. The layers at the non-vegetated site (0-20 cm) have sandy loam texture of degraded finely dispersed lignite, coal ash particles and black, greyish-green and yellow clays. At the pine-vegetated site the substrate was of heavier clay texture at the 10-20 cm depth. At both sites, the parent mine sediments contained irregularly distributed lignitic particles of sand grain size, and coarser fragments. Layers of black clays of heavy clay texture were located to a lower depth of > 20 cm. The substrate 0-50 cm was found to be water repellent since water drop penetration time (WDPT) which is an indicator of the persistence of water repellency was > than several minutes. Soil samples were classified as strongly (60 s < WDPT< 600 s) and severely > 600 s water repellent according to the scale of De Bano (1981). The soil samples were weighed to determine the field moisture content and subsequently equilibrated at the ambient air humidity before measuring water drop penetration time (WDPT) in the laboratory at controlled humidity and temperature. After sampling, soils were transferred to the laboratory, air-dried at room temperature and milled to < 2 mm fraction.

All chemical analyses were performed on < 2 mm material without further grinding, except for the organic carbon analysis (sample size < 250 µm). For chemical analysis a 100 g sub-sample of the < 2 mm fraction was obtained by the coning and quartering method to reduce sample variability. Organic carbon (OC) in the studied spoils was determined with the modified Tjurin's method (oxidation with $K_2Cr_2O_7/H_2SO_4$) and organic carbon fractionation by the method of Kononova, 1963, (modified by Filcheva & Tsadilas, 2002), cation exchange capacity was determined as sum of titratable acidity (pH 8.2) and extractable Ca, by saturation with K malate at pH 8.2 (Ganev & Arsova 1980). Sample water-repellency (soil hydrophobicity) was measured by the water drop penetration time (WDPT) method (Doerr et al. 2002). Three droplets of distilled water (80 µl) were placed on the soil surface and the time recorded for droplet penetration. Total nitrogen was analysed by the Kjeldahl procedure and mineral NH_4^+-N and NO_3^--N after Bremner (1965). Soil texture analysis was performed according to the method of Kachinski (Kachinskii, 1965) Statistical analysis (principal component PCA and cluster analysis, CA) was performed by SPSS 22 for MS Windows.

Results and Discussion

We suspect that not only water repellency is the cause of lack of vegetation, but also the low pH and metal toxicity (mostly AI, ~ 33% of total CEC) are the other possible factors (Table 1a), Atanassova et al. unpublished data). The reason for the very low pH encountered is due to the oxidation of sulfide to sulfate which solubilizes Fe (II), subsequently oxidized to Fe (III). Soil water repellency was higher in the surface 0-5; 0-10 cm layers, than in the sub-surface layers (10-20 cm) which correlated with the higher organic matter content. We further investigated potential correlations between persistence (WDPT) of SWR and other soil properties such as total and humified organic carbon, and total and mineral nitrogen, pH, hygroscopic moisture, texture, cation exchange capacity and electrical conductivity.

The organic matter of the more water repellent soil samples was generally higher than that of the less water repellent lower layers (Table 1b).

The correlation matrix is presented in Table 2. The degree of SWR, measured through WDPT was positively correlated with the total and fractionated (humic and fulvic) soil organic carbon ($R_{TOC} = 0.699^*$, $R_{HOC} = 0.499^*$ and $R_{FOC} = 0.442^*$), Table 2.

Some authors (Sepehrnia et al., 2016) reported that soil organic matter (SOM) was associated with water repellency. It has been also speculated that SOM enhances soil water repellency, and vice-versa water repellency protects organic matter against microbial decomposition. A positive correlation between SOM and both the degree and persistence of water repellency was found by Leelamanie (2014) and Jeyakumar et al. (2014). However, Doerr et al., (2000) found a lack of correlation. The explanation for this inconsistency may be that that it is the quality of SOM rather than total quantity that causes water repellency, therefore a certain amount of specific hydrophobic compounds might be needed to cause SWR. Values of organic carbon in the technogenic soils, as determined by the Tjurin's method varied with depth and between plots in the range of 1.7%-11%. These values reflect the resistance of the degraded lignitic particles to microbial hydrolysis and oxidation. The mine soil material in the depth of 0-10 cm, contained lignitic particles of sand grain size. The colour of the technogenic soil is predominantly black (upper layer) with a yellow-green-grayish appearance deeper in the soil profile at > 20 cm depth. We suspect that hydrophobic compounds from the lignite which cause the luster or greasiness of the clays are the most probable source of water repellency. In the water repellent spoils under pine vegetation, soil hydrophobicity might have a miscellaneous source, both lignitic particles and waxes from the pine vegetation. Total organic carbon (TOC) containing lignitic particulate organic matter as a source of water repellency is strongly correlated with the sand fraction of the soil ($R_{TOC} = 0.446^*$ and $R_{HOC} = 0.579^*$ and $R_{FOC} = 0.462^*$).

Relationships between soil chemical properties (e.g. increase of repellency with decreasing pH) and soil water repellency are scarce (Krueger et al. 2016). A positive significant relationship was obtained between WDPT and mineral nitrogen ($R = 0.580^*$) and a negative ($R = -492^*$) with hygroscopic moisture (Table 2). Water repellency is a transient property depending ultimately on soil moisture content. SWR is usually highest in dry soils and disappears when the soil water content exceeds a critical threshold (Doerr et al. 2000). The variability of the critical water content depends on the wetting history of the soil or the heterogeneous distribution of water in and around the soil aggregates (Dekker et al., 2001). Soil water repellency is more commonly encountered in dry soils and has serious consequences for carbon and nutrient cycling, therefore is significantly affecting microbial activity, as well.

Table 1a. Physical and physico-chemical soil properties

Site	Depth cm	WDPT (s)	Sand %		Silt %		Clay %		Hygroscopic moisture %		CEC	
		Range median	Mean ±	SD	Mean ±	SD	Mean ±	SD	Mean ±	SD	Mean ±	SD
Pine vegetation	0-5	14-9589 1200	47.5	7.21	22.4	0.07	28.0	7.21	8.2	0.69	41.27	3.89
	10-20	2-128 10	25.5	20.45	22.04	0.03	55.1	21.72	8.4	0.54	44.60	3.52
non-vegetated	0-10	76-14440 1282	59.0	1.89	21.8	0.08	17.8	3.86	8.2	0.63	67.77	6.16
	10-20	202-2470 614	62.8	4.84	21.3	0.10	17.0	2.37	7.8	1.9	69.90	8.70

* Range and median (underlined).

Table 1b. Soil chemical properties

Site	Depth cm	pH		EC		TOC %		HOC %		FOC %		N total %		ΣN-NH_4+ and NO_3^- mg/kg	
		Mean ±	SD	Mean ±	SD	Mean ±	SD	Mean ±	SD	Mean ±	SD	Mean ±	SD	Mean ±	SD
Pine vegetation	0-5	4.58	0.20	0.19	0.14	5.09	0.42	1.87	0.52	0.66	0.31	0.22	0.03	15.17	2.4
	10-20	4.18	0.43	0.63	0.14	3.00	1.67	1.03	0.89	0.29	0.42	0.10	0.04	11.40	1.7
non-vegetated	0-10	3.18	0.11	1.72	0.76	6.43	3.98	2.49	1.23	0.79	0.45	0.11	0.02	40.33	19.5
	10-20	3.13	0.10	1.61	0.69	6.11	0.96	2.96	0.51	0.92	0.56	0.12	0.01	42.87	18.5

WDPT-mineral nitrogen relationship observed is in line with the studies of Elbl et al. (2014) who have found that changes of soil water content have an impact on microbial activity, hydraulic conductivity, soil hydrophobicity and loss of mineral nitrogen from soil.

A positive, although insignificant correlation was also observed between WDPT and CEC (R= 0.408) and significant correlation with TOC, HOC and FOC (R = 0.466*; R = 0.472*; R = 0.638*) which sustains the hypothesis of the intimate association of the hydrophobicity causing compounds with soil colloids of organic, and/or organo-mineral nature. Cation exchange capacity (CEC) as a source of ions in solution is significantly correlated with EC (R = 0.939*) and mineral nitrogen (MN, R = 0.804*), but negatively correlated with clay (R = -0.603*) and positively with the sand fraction R = 0.636*. Such a trend is plausible if organic matter is assumed to be the main source of cation exchange in the studied soils, while a reduction in the CEC of the clay fraction might have occurred through blocking by organic matter of the negative charge of the clay minerals or by inter-lamellar amorphous gibbsite or brucite layers balancing the permanent charge of the clay minerals in the acid pH = 3-4 leading to a decrease or lack of reactive cation exchange sites. A significant correlation was also observed between EC and FOC, containing the most reactive organic carbon, including low-molecular weight organic carbon, as well mineral nitrogen providing NO_3^- and NH_4^+ ions in solution.

We suspect that the small-scale variability of water repellency may be correlated with the spatial distribution of particulate lignitic particles and lignite-coated minerals grains. We are further studying the effect the spatial pattern of water repellency has on water and contaminant movement in the technogenic soils (unpublished data).

To get more information about the inter-relation between the different soil characteristics and properties, principal component (PCA) and cluster analyses (CA) were conducted with the twelve measured parameters at the different sampling sites and layers. The PCA identified three principal components (PCs) (Table 3) explaining 46.7%, 17.8% and 14.4% of the total variance (79%). The components matrix (Table 3) distinguishes between components that are as different from each other as possible, and helps interpretation by allocating each variable primarily on one of the components. The 1st principal component is mainly loaded by WDPT, CEC, EC, % sand, TOC, HOC, FOC, mineral N (MN) and through a strong inverse relationship with the % clay. Total nitrogen (N) and contents of silt are mainly related to component 2, and % hygroscopic moisture to component 3.

Table 2. Correlation matrix between the measured parameters

* Significant at $p< 0.05$

	WDPT	CEC	SAND	SILT	CLAY	TOC	HOC	FOC	MN	EC	M	N
WDPT	1.000	0.408	0.137	0.066	-0.116	0.699*	0.499*	0.442*	0.580*	0.326	-0.492*	-0.017
CEC	0.408	1.000	0.636*	0.100	-0.603*	0.466*	0.472*	0.638*	0.804*	0.939*	-0.207	-0.375
SAND	0.137	0.636*	1.000	0.263	-0.985*	0.446*	0.579*	0.462*	0.528*	0.441*	-0.112	0.236
SILT	0.066	0.100	0.263	1.000	-0.419	0.182	0.117	0.260	0.029	0.072	0.100	0.408
CLAY	-0.116	-0.603*	-0.985*	-0.419	1.000	-0.442*	-0.553*	-0.463*	-0.498*	-0.413	0.097	-0.297
TOC	0.699*	0.466*	0.446*	0.182	-0.442	1.000	0.862*	0.714*	0.666*	0.284	-.0466*	0.338
HOC	0.499*	0.472*	0.579*	0.117	-0.553	0.862*	1.000	0.645*	0.526*	0.262	-0.238	0.285
FOC	0.442*	0.638*	0.462*	0.260	-0.463*	0.714*	0.645*	1.000	0.612*	0.609*	-0.157	0.212
MN	0.580*	0.804*	0.528*	0.029	-0.498*	0.666*	0.526*	0.612*	1.000	0.733*	-0.706*	-0.120
EC	0.326	0.939*	0.441*	0.072	-0.413	0.284	0.262	0.609*	0.733*	1.000	-0.171	-0.518*
M	-0.492*	-0.207	-0.112	0.100	0.097	-0.466*	-0.238	-0.157	-0.706*	-0.171	1.000	-0.076
N	-0.017	-0.375	0.236	0.408	-.0297	0.338	0.285	0.212	-0.120	-0.518	-0.076	1.000

The dendrogram of Fig. 1 supports represents three major branches (groups). The first major group can be sub-divided into two sub-groups. The first sub-group is represented by CEC, EC, MN and % sand content and the second sub-group by TOC, HOC, FOC and WDPT. The second main group is represented by the % silt and total N and the third main group by the % hygroscopic moisture and the % clay content. The organic carbon parameters TOC, FOC and HOC are intermediately placed in the cluster tree, confirming their linkage to components 1 and 2. The cluster analysis supports the relationships obtained from the PCA analysis for grouping of various soil parameters.

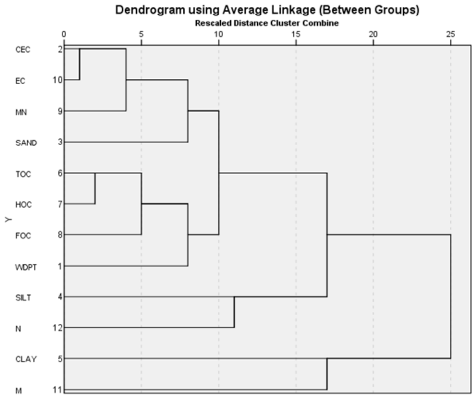

Fig. 1. Cluster analysis of the twelve variables analyzed

Table 3. Components matrix. Three components extracted

	Component		
	1	2	3
WDPT	.611	-.143	-.558
CEC	.840	-.411	.325
SAND	.744	.279	.447
SILT	.248	.541	.288
CLAY	-.732	-.362	-.472
TOC	.820	.239	-.425
HOC	.773	.292	-.189
FOC	.801	.089	.007
MN	.879	-.308	-.164
EC	.702	-.572	.351
M	-.443	.169	.625
N	.087	.922	-.214

Conclusions

Technogenic soils from the area of Maritsa-Iztok lignite coal basin in Bulgaria were studied at two non-vegetated and pine-afforested plots. The soils were characterized by severe to extreme hydrophobicity and heavier texture at the pine-vegetated site, as well as extreme acidity (pH 3-4). Principle Component Analysis (PCA) and cluster analysis were performed with the aim to assess the interrelation between the measured parameters and the persistence of soil water repellency, as revealed by the water drop penetration time (WDPT). Three principle components were identified describing 79% of the total variability. The WDPT was significantly positively correlated with TOC, as well as humic and fulvic organic carbon (HOC and FOC), mineral nitrogen (MN) and negatively correlated with the % hygroscopic moisture. The results obtained indicate that total organic carbon (TOC) containing irregularly distributed coal and ash particles, as well as humic and fulvic organic carbon (HOC and FOC) were significantly correlated with the mineral nitrogen (MN), and were the main drivers of soil water repellency in the studied technogenic soils.

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