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# Reclamation of Drill Cuttings Landfill

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## 1. Introduction

As a result of drilling process conducted during exploration of unconventional natural gas deposits, the soil environment is degraded. Large areas of land are being occupied by drilling rigs and their infrastructure, as well as landfills (Brittingham et al. 2014). Apart from mechanical destruction, which prevent soil from fulfilling its basic functions (Konschnik et al. 2014), chemical pollution may occur as well (Zoback et al. 2010). The process of drilling wells produces drilling wastes, which are heterogeneous in terms of physical and chemical properties (Ball et al., 2012). Waste from drilling industry is predominantly deposited on landfills (Dubiel et al., 2010). Current legal regulations, aimed to protecting the environment from releasing of the pollutants, obligate to insulate the drilling waste landfills and their reclamation (Maloney, Yoxtheimer 2012).

Increase of the biological activity of soil or initiation of soil-based ecosystem development in the case of devastated lands are the basic objectives of the reclamation processes (Bradshaw, 1983). The most important factor influencing the soil forming processes is accumulation of organic matter and its transformations (Fettweis et al. 2005). The organic matter content is considered a key element in determining the quality of soils (Reeves 1997; Diacono, Montemurro 2010). It significantly influences the physical, chemical, and biological properties of soils. It is also the basic indicator of the their productivity (Lal 2001).

Reconciling the environmental and economic goals that should be achieved during the land reclamation processes is possible when the effect of restoration will be sustainable and long-lasting. Moreover the time required to meet the objective should be the shortest, while the cost should be as low as possible (Baran et al. 2014).

In the case of soil on devastated lands restoration, it is necessary to supply organic substances in order to initiate the processes of organic matter accumulation. Introducing exogenous organic matter improves the properties of soil and delivers energy which is required to sustain the metabolic processes of ecosystems (Woś et al. 2014). Simultaneously, the exogenous organic matter constitutes an important element in the organic carbon balance of the reclaimed soils (Vanduskova, Frouz 2013). Organic waste can be used as a source of such a matter in the reclamation of degraded soils or lands.

The aim of the paper was to assess the potential usefulness of composites produced from various types of wastes in restoration on drilling waste landfill.

## 2. Methodology

The pot experiment, which was conducted in an experimental greenhouse of the University of Life Sciences in Lublin, examined composites in regard to their usefulness for the construction of bio-cover on drill cuttings landfill. The composites were made of anthropogenic soil, drill cuttings derived from the processes of shale gas exploration in Poland, sewage sludge (anaerobically digested mixture of primary and excess sludges) from a municipal wastewater treatment plant in Lublin, sewage sludge from industrial wastewater treatment plant in Zakłady Azotowe Puławy, and coal gangue from "Bogdanka" Coal Mine. The selected properties of materials used in the experiment are presented in table 1.

The components were mixed with different configuration and amount. Moreover, the reference mixtures were prepared, which comprised: unfertilized soil, soil amended with NPK in the amount of: N – 50 $kg \cdot ha^{-1}$ (0.327g/pot of ammonium nitrate), $P_2O_5$ – 50 $kg \cdot ha^{-1}$ (0.694 g/pot of polifoska 8); $K_2O$ – 80 $kg \cdot ha^{-1}$ (0.167 g/pot of potassium salt), and soil amended with 30 $Mg \cdot ha^{-1}$ of manure. The examined materials are presented in table 2. The percentage of individual components used in the mixtures and their mass introduced to experimental pots is presented in table 3.

**Table 1.** Selected properties of material used in experiment
**Tabela 1.** Wybrane właściwości materiałów użytych w eksperymencie

| Property | Unit | Anthro-pogenic soil | Drill cuttings | Industrial sewage sludge | Municipal sewage sludge | Coal gangue |
|---|---|---|---|---|---|---|
| pH | KCl | 4.4 | 8.8 | 6.0 | 6.1 | 6.7 |
| Hh | $cmol(+)\cdot kg^{-1}$ | 3.60 | 0.30 | 1.58 | 4.73 | 0.60 |
| S | $cmol(+)\cdot kg^{-1}$ | 3.20 | 50.10 | 25.17 | 29.14 | 10.58 |
| C total | $g\cdot kg^{-1}$ | 7.20 | 8.10 | 58.80 | 207.00 | 4.86 |
| N total | $g\cdot kg^{-1}$ | 0.92 | 0.70 | 15.50 | 37.90 | 0.23 |
| C:N | | 7.8 | 11.6 | 3.8 | 5.5 | 20.8 |
| Cu | $mg\cdot kg^{-1}$ | 19.0 | 28.80 | 13.30 | 301.0 | 54.4 |
| Zn | $mg\cdot kg^{-1}$ | 23.2 | 62.10 | 55.40 | 874.0 | 56.8 |
| Pb | $mg\cdot kg^{-1}$ | 28.2 | 34.90 | 21.80 | 39.50 | 62.3 |
| Cr | $mg\cdot kg^{-1}$ | 25.0 | 16.90 | 12.50 | 89.20 | 16.9 |
| Ni | $mg\cdot kg^{-1}$ | 4.5 | 20.50 | 7.40 | 37.50 | 44.7 |
| Cd | $mg\cdot kg^{-1}$ | 0.44 | 0.66 | 0.51 | 3.52 | 1.02 |
| Hg | $mg\cdot kg^{-1}$ | 0.015 | 0.063 | 0.015 | 0.982 | 0.112 |
| Sr | $mg\cdot kg^{-1}$ | 34.1 | 1340.0 | 1890.0 | 1180.0 | 224.0 |
| Ba | $mg\cdot kg^{-1}$ | 38.1 | 7272.0 | 51.30 | 110.0 | 307.0 |
| Total PAHs | $mg\cdot kg^{-1}$ | 0.010 | 0.0461 | 2.257 | 6.727 | 0.224 |
| Sand (2.0-0.05 mm) | % | 58 | 36 | Not determined | Not determined | 57 |
| Silt (0.05-0.002 mm) | % | 39 | 40 | Not determined | Not determined | 25 |
| Clay (<0.002 mm) | % | 3 | 24 | Not determined | Not determined | 18 |

The experiment started on 10$^{th}$ July 2014. Pots with the volume of 12 $dm^3$ were filled with relevant amount of anthropogenic soil, to which different amount of mineral and organic wastes were added (according the data given in table 3). Substrates prepared in such way were used for the cultivation of white mustard (*Sinapis alba*) cv. Borowska C/1 (growing season – 55 days). In the following year the common buckwheat (*Fagopyrum esulentum Moench*) cv. Kora was growing (growing season

– 55 days). Each variant of soil material was tested in three repetitions. During the growing season, plants were watered with distilled water (0,5 $dm^{-3}$/pot every 3 days).

**Table 2.** Reclamation variants used in the experiment
**Tabela 2.** Warianty podłoży rekultywacyjnych badane w eksperymencie

| Sym bol | Reclamation variant |
|---|---|
| K1 | Soil – control 1 |
| K2 | Soil + NPK – control 2 |
| K3 | Soil + 30 $Mg \cdot ha^{-1}$ of manure – control 3 |
| R1 | Soil + 50 $Mg \cdot ha^{-1}$ of drilling waste + 10 Mg d.m.$\cdot ha^{-1}$ of sludge (L) |
| R2 | Soil + 50 $Mg \cdot ha^{-1}$ of drilling waste + 10 Mg d.m.$\cdot ha^{-1}$ of sludge (L) + 100 $Mg \cdot ha^{-1}$ of coal gangue |
| R3 | Soil + 100 $Mg \cdot ha^{-1}$ of drilling waste + 100 Mg d.m.$\cdot ha^{-1}$ of sludge (L) |
| R4 | Soil + 100 $Mg \cdot ha^{-1}$ of drilling waste + 100 Mg d.m.$\cdot ha^{-1}$ of sludge (L) + 200 $Mg \cdot ha^{-1}$ of coal gangue |
| R5 | Soil + 50 $Mg \cdot ha^{-1}$ of drilling waste + 10 Mg d.m.$\cdot ha^{-1}$ of sludge (L+P, ratio 1:1 d.m/d.m) |
| R6 | Soil + 50 $Mg \cdot ha^{-1}$ of drilling waste [1] + 50 Mg d.m.$\cdot ha^{-1}$ of sludge (L+P, ratio 1:1 d.m/d.m) + 100 $Mg \cdot ha^{-1}$ of coal gangue |
| R7 | Soil + 100 $Mg \cdot ha^{-1}$ of drilling waste + 200 Mg d.m.$\cdot ha^{-1}$ of sludge (L+P, ratio 1:1 d.m/d.m) + 200 $Mg \cdot ha^{-1}$ of coal gangue |
| R8 | Soil (25%) + drilling waste (25%) + sludge (L+P, ratio 1:1 d.m/d.m; 25% d.m.) + coal gangue (25%) |
| R9 | Soil (50%) + drilling waste (10%) + sludge (L+P, ratio 1:1 d.m/d.m; 10% d.m.) + coal gangue (30%) |
| R10 | Drilling waste (50%) + sludge (L+P, ratio 1:1 d.m/d.m; 50% d.m.) |

*P – sludge from ZA Puławy;*
*L – sludge from a municipal treatment plant in Lublin; d.m. dry mass*

The soil samples for laboratory examinations were collected three times: directly after the preparation of mixtures (I period), and following the harvesting of plants (II period – following the harvest of mustard; and III period – following the harvest of buckwheat periods).

**Table 3.** Quantitative composition of the particular materials examined in the pot experiment
**Tabela 3.** Skład ilościowy podłoży badanych w eksperymencie wazonowym

| Symbol | Soil | | Manure | | Drilling waste | | Sludge L | | Coal gangue | | Sludges P+L | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | % in the composite | kg d.m./pot | % in the composite | kg d.m./pot | % in the composite | kg d.m./pot | % in the composite | kg d.m./pot | % in the composite | kg d.m./pot | % in the composite | kg d.m./pot |
| K1 | 100 | 10 | - | - | - | - | - | - | - | - | - | - |
| K2 | 100 | 10 | - | - | - | - | - | - | - | - | - | - |
| K3 | 97 | 9.7 | 3 | 0.34 | - | - | - | - | - | - | - | - |
| R1 | 98 | 9.80 | - | - | 1.7 | 0.17 | 0.3 | 0.034 | - | - | - | - |
| R2 | 94.6 | 9.46 | - | - | 1.7 | 0.17 | 0.3 | 0.034 | 3.4 | 0.34 | - | - |
| R3 | 93.2 | 9.32 | - | - | 3.4 | 0.34 | 3.4 | 0.34 | - | - | - | - |
| R4 | 86.4 | 8.64 | - | - | 3.4 | 0.34 | 3.4 | 0.34 | 6.8 | 0.68 | - | - |
| R5 | 98 | 9.80 | - | - | 1.7 | 0.17 | - | - | - | - | 0.3 | 0.033 |
| R6 | 93.2 | 9.32 | - | - | 1.7 | 0.17 | - | - | 3.4 | 0.34 | 1.7 | 0.17 |
| R7 | 63 | 6.30 | - | - | 3.4 | 0.34 | - | - | 6,8 | 0.68 | 6.8 | 0.68 |
| R8 | 25 | 2.50 | - | - | 25 | 2.50 | - | - | 25 | 2.50 | 25 | 2.50 |
| R9 | 50 | 5.00 | - | - | 10 | 1.00 | - | - | 30 | 3.00 | 10 | 1.00 |
| R10 | - | - | - | - | 50 | 5.00 | - | - | - | - | 50 | 5.00 |

*P – sludge from ZA Puławy; L – sludge from a municipal wastewater treatment plant in Lublin*

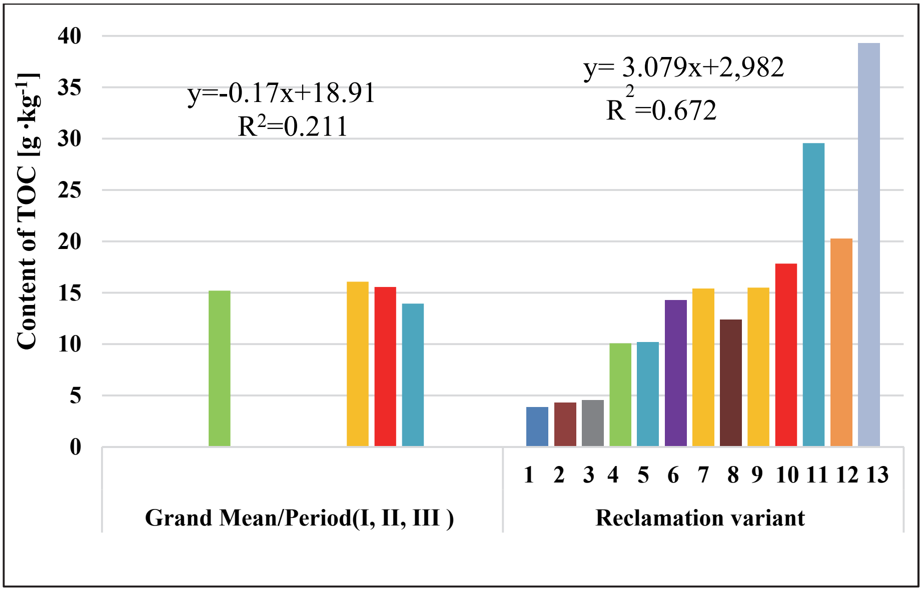
Following parameters were determined in the samples: Total organic carbon (TOC), by means of TOC-$V_{CSH}$, SSM-5000A Shimadzu Analyzer (PN-ISO 14335); Total Kjeldahl Nitrogen, in line with the PN-EN 13342:2002 standard; Content of organic labile fractions with Andrzejewski method, modified by Myśków (Myśków, Zięba, 1981); Fractional composition of humus with Schnitzer method (Schnitzer, Shuppli, 1989). The results were statistically prepared by calculating the analysis of variance (ANOVA). The significance of differences was determined with Tukey's test p = 0.05. Statistical analyses were performed using software STATISTICA 9.0.

## 3. Results and discussion

Initiating ecosystem development, with soil as the main element, is an extremely important task in land reclamation process (Bradshaw, 1983). The content of organic fraction enables to distinguish soils from soilless formations (Lal, 2001). Moreover, it is commonly assumed that the content of organic substance may be an important indicator of soil fertility and its capability to fulfilling environmental functions.

The total organic content in anthropogenic unfertilized soil (K1) amounted to 7.20 $g \cdot kg^{-1}$ and it was maintained on a similar level throughout the whole research period (Tab. 4). An insignificant increase of total organic content was observed in soil fertilized with NPK (K2) in consecutive research periods. Mineral fertilization indirectly raises TOC by increasing biomass production, and therefore, the post-harvest residue, which are a source of humic substances in soil (Kucharik et al. 2001). An insignificant increase of organic carbon content in relation to unfertilized and NPK-fertilized soil was observed in the case of soil fertilized with manure (K3). In the majority of evaluated mixtures, significantly higher organic carbon content was found in relation to the control soils K1-K3 (Tab. 4, Fig. 1).

In the I period, the highest TOC, amounting to 66 $g \cdot kg^{-1}$, was found in the mixtures containing drilling waste (50%) and a mixture of sludges from Lublin and Puławy (50%), i.e. R10 composite. Among the considered reclamation variants with anthropogenic soil, a significant increase in TOC (over twofold) was observed for the variants with 100 $Mg \cdot ha^{-}1$ of drilling waste and 100 $Mg \cdot ha^{-1}$ of sewage sludge from Lublin (composite R3). A major increase in TOC in soil of the evaluated reclamation variants should be related to amount of exogenous organic matter introduced to the substrate (Anderson et al. 2008).The total organic carbon content in the considered period has fluctuated slightly. In the mixtures in which the content of carbon in the I period were significantly higher than the in the next one, a decrease in organic carbon was observed, indicating intensive mineralization of organic substance. In the remaining mixtures, the organic carbon content remained at a similar level or increased slightly. This can be attributed to an increased yield of plants, which simultaneously increases the amount of fresh organic matter that constitutes the main factor in organic compounds production in arable soils (Gunina et al. 2015). The obtained results confirm the theory found in the literature stating that an efficient reclamation which ensures restoration of humic substances in the degraded soil depends on the introduction of exogeneous organic matter and the cultivation of plants with high humus-forming capacity (Liu et al. 2006).




**I Period** – Beginning of the experiment (10.07.2014); **II Period** – following the harvest of mustard (04.09.2014); **III Period** – following the harvest of buckwheat (23.06.2015); ***1*** *K1,* ***2*** *– K2,* ***3*** *– K3,* ***4*** *– R1,* ***5*** *– R2 ,* ***6*** *– R3,* ***7*** *– R4 ,* ***8*** *– R5 ,* ***9*** *– R6 ,* ***10*** *– R7 ,* ***11 –*** *R8,* ***12*** *– R9,* ***13*** *– R10.*

**Fig. 1.** Total organic carbon in the reclamation variants Mean values
**Rys. 1.** Zawartość $C_{org}$ w podłożach rekultywacyjnych. Wartości średnie

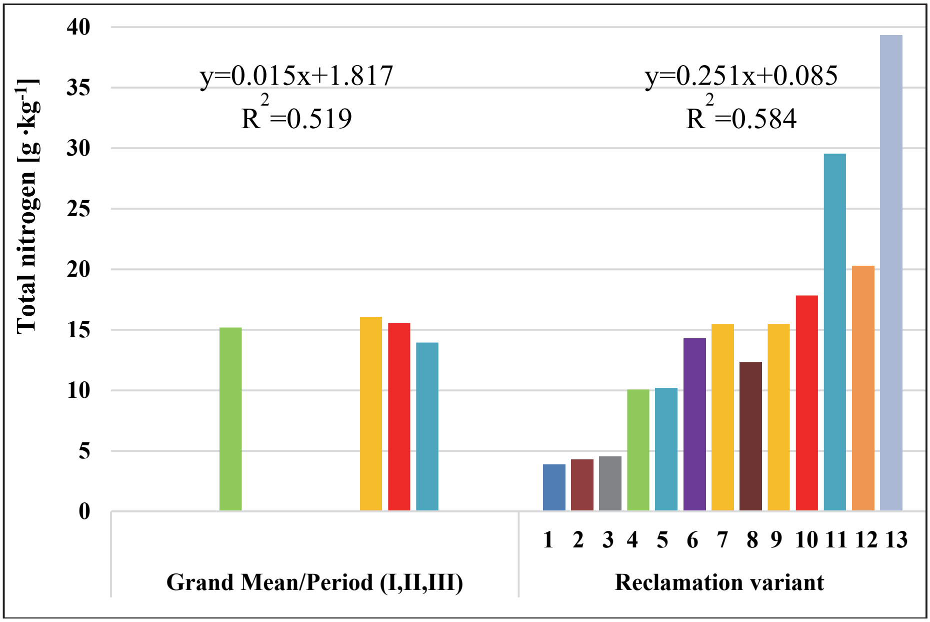



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**Fig. 2.** Content of total nitrogen in reclamation variants. Mean values
**Rys. 2.** Zawartość $N_{og}$ w podłożach rekultywacyjnych. Wartości średnie

Table 4 presents changes in the total nitrogen content in the examined materials. The value of $N_{tot}$ content in the anthropogenic soil has increased significantly after the waste addition (Fig. 2). The content of total nitrogen in the deposited mineral waste is generally considered insufficient to meet the nutritional needs of plants; therefore, application of fertilizers is necessary in order to create a fertile substrate. Organic matter is thought to be the main source of nitrogen in reclaimed soils (Sheoran et al. 2010). The range of $N_{tot}$ values increase in the considered soil mixture was dependent on the type of applied waste and their percentage in a composite.

An increase in the total nitrogen content was observed already with the smallest share of the considered wastes; however, the statistically significant higher $N_{tot}$ content was found in the soil mixture R3, in the variants with 100 $Mg \cdot ha^{-1}$ of drilling wastes and 100 $Mg \cdot ha^{-1}$ of sewage sludge from Lublin. Similarly as in the case of organic carbon content, the $N_{tot}$ was highest in R10 composite, which comprises drilling waste (50%) and a mixture of sludges from Lublin and Puławy (1:1 d.m/d.m ratio), amounting to 5.45 $g \cdot kg^{-1}$ within the measurement period (on average).

**Table 4.** Organic carbon and total nitrogen contents in the examined materials
**Tabela 4.** Zawartość węgla organicznego i azotu ogólnego w badanych podłożach

| Symbol | TOC | | | | $N_{tot}$ | | | |
|---|---|---|---|---|---|---|---|---|
| | $g \cdot kg^{-1}$ | | | | | | | |
| | I | II | III | Mean | I | II | III | Mean |
| K1 | 7.20 | 7.10 | 7.26 | 7.19 | 0.92 | 0.92 | 0.95 | 0.93 |
| K2 | 7.26 | 7.46 | 7.58 | 7.43 | 0.96 | 1.05 | 1.00 | 1.00 |
| K3 | 8.64 | 8.96 | 9.04 | 8.88 | 1.04 | 1.08 | 1.13 | 1.08 |
| R1 | 9.46 | 9.68 | 9.80 | 9.65 | 1.12 | 1.20 | 1.24 | 1.19 |
| R2 | 9.92 | 9.84 | 10.02 | 9.93 | 1.20 | 1.20 | 1.24 | 1.21 |
| R3 | 15.44 | 15.82 | 15.96 | 15.74 | 1.48 | 1.52 | 1.60 | 1.53 |
| R4 | 15.80 | 15.68 | 16.04 | 15.84 | 1.46 | 1.54 | 1.58 | 1.53 |
| R5 | 8.46 | 8.98 | 9.02 | 8.82 | 0.85 | 0.95 | 0.90 | 0.90 |
| R6 | 13.60 | 13.86 | 14.00 | 13.82 | 1.30 | 1.36 | 1.37 | 1.34 |
| R7 | 20.20 | 20.36 | 20.30 | 20.29 | 1.96 | 2.08 | 2.10 | 2.05 |
| R8 | 36.50 | 35.16 | 36.34 | 36.00 | 3.13 | 3.18 | 3.11 | 3.14 |
| R9 | 27.65 | 27.20 | 28.35 | 27.73 | 2.60 | 2.58 | 2.75 | 2.64 |
| R10 | 66.00 | 56.40 | 58.00 | 60.13 | 5.91 | 5.13 | 5.30 | 5.45 |
| Mean | 18.93 | 18.19 | 18.59 | 18.57 | 1.84 | 1.83 | 1.87 | 1.85 |
| NIR for the reclamation variant | 4.39** | | | | 0.39** | | | |
| NIR for the period | 1.44 | | | | 0.13 | | | |
| NIR variant x period | 8.21 | | | | 0.72 | | | |

***I period*** *– beginning of the experiment (10.07.2013r.);* ***II period*** *– following the harvest of mustard (03.09.2014r.);* ***III period*** *– following the harvest of buckwheat (23.06.2015r.). Sludge from Lublin (L); Sludge from Puławy (P).*

In the anthropogenic soil which was unfertilized and fertilized with NPK, the ratio of TOC/Ntot assumed the value of about 7.5. Addition of the evaluated wastes increased the considered index (Fig. 3). In the case of soil with manure and the lowest amount of considered waste (R1), the TOC/$N_{tot}$ ratio increased significantly, reaching the value greater than 8. Increasing the share of waste in the composites caused a further increase in the value of the TOC/$N_{tot}$ ratio. Its highest value, i.e. over 11, was observed in the soil in which the percentage of sludge mixture and drilling waste amounted to 25 and 50% (R9 and R10). It should be emphasized that despite a significant increase of TOC/$N_{tot}$ ratio in the soil of the considered variants, its values were not high enough to cause disturbances in the transformations of organic matter occurring in soil. The humus which is created in the process of soil restoration significantly influences the soil properties; therefore, the efficiency of reclamation depends on the quality of newly created humic compounds (Chaudhuri et al. 2015).

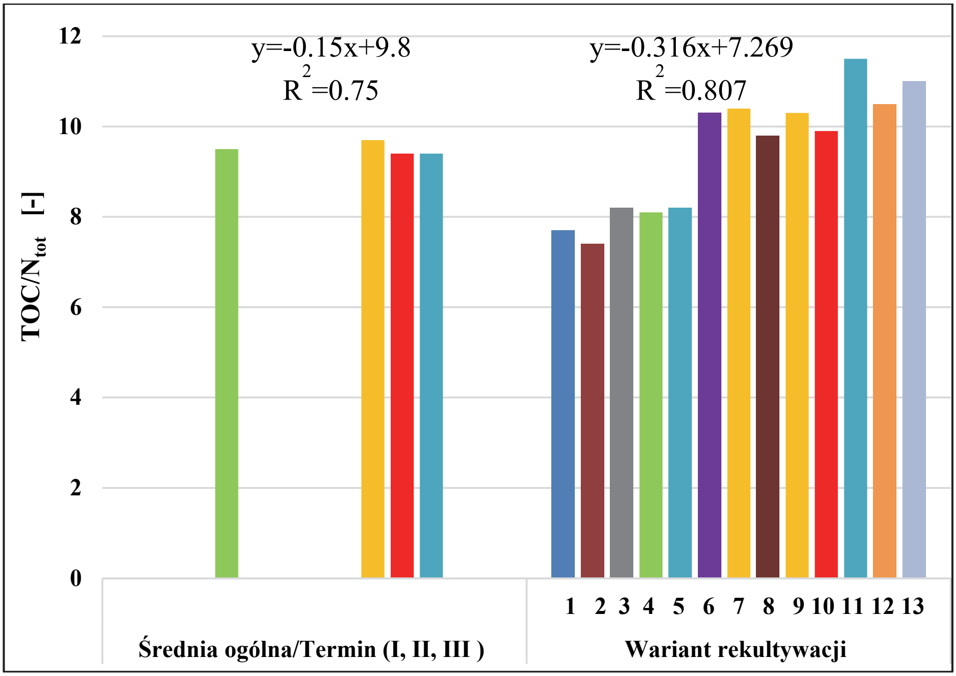


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**Fig. 3.** TOC/$N_{tot}$ ratio in the reclamation variants. Mean values
**Rys. 3.** Stosunek TOC/$N_{tot}$ w podłożach rekultywacyjnych. Wartości średnie

The organic matter of soils comprises heterogeneous mixture of substances which are characterized by a broad range of susceptibility to mineralization (Haynes, 2005). Out of the total amount of organic matter supplied to the soil, only about 30% of carbon compounds is stabilized by humification processes (Janzen et al. 1997). The products of decomposition of plants and microorganisms are a source of labile fraction of organic matter. They constitute a more active fraction and exhibit faster reaction to anthropological factors (Janzen et al. 1997) and climatic changes (Xu et al. 2012) than the stable fraction. Cheng et al. (Cheng et al. 2007) showed that the assessment pertaining to the content of labile fraction of organic matter in soil and their reaction to changing conditions is necessary for modelling carbon budget in soils.

The content of labile fractions of organic matter was largely determined by the type and amount of waste introduced to the soil (Tab. 5). In an unfertilized soil, the content of labile fractions equalled 3.68 $g \cdot kg^{-1}$, which constituted over 51% of TOC. Such high percentage of labile fractions of organic matter indicates its poor quality and superiority of mineralization process over humification (Myśków, Zięba, 1981). A decrease of labile fraction percentage to 47.3% of TOC was observed in the soil enriched with manure (K3). The amount of sewage sludge had a significant impact on this decrease. In the case of the composite containing 50% of drilling waste and 50% of sludges from Lublin and Puławy (R10), only 11.57% of TOC corresponded to the considered fractions. On the other hand, in the composites with sewage sludge applied in the amount of 10 and 50 $Mg \cdot ha^{-1}$ (R1, R2 and R5, R6), the share of labile carbon compounds in TOC reached approximately 44%.

An optimal share of labile carbon compounds, which covers the energy needs of microorganisms and limits the losses of organic carbons in the soil, can be found in the composites with the addition of sludge amounting to 100-200 $Mg \cdot ha^{-1}$ (R3, R4 and R7 composites). In these mixtures, 21-27% of TOC correlated with the carbon of labile compounds, which corresponds to their content in chernozems, and black rendzinas which are considered soils with balanced dynamics of humic compounds transformation processes (Żukowska et al. 1999).

In the considered period, a significant increase in the content of labile fraction carbon was observed between the beginning of the experiment and its period III. The properties of humus depend on the soil-forming process and constitute a characteristic feature of various types of soils (Stevenson, 1994). Humic acids, which are the main component of humus, determine its properties, and thus their role in the natural environment (Ouni et al. 2014). It is commonly known that in arable soils the amount of humic acids and their properties are modified with the type of applied fertilization and the selection of plants in rotation (Diacono, Montemurro, 2010). According to the research, the fractional composition of organic matter was also determined by the type and amount of waste applied in the composites (Tab. 6 and 7). The percentage of humic acids (released carbon) in the unfertilized and NPK-fertilized soil amounted to approximately 55% of TOC. Drill cuttings in the amount of 50 Mg·ha$^{-1}$ along with sewage sludge from Lublin in the amount of 10 and 50 Mg·ha$^{-1}$ (R1-R4 composites) decreased the share of considered fractions of organic matter to about 49% of TOC, which is a typical percentage for soils characterized by medium-quality of humic compounds.

A significant decrease in the percentage of humic acids was observed in the composites with 100 Mg·ha$^{-1}$ of sewage sludge added. The share of the considered connections in these composites ranged from about 26 to 29% of TOC. Taking into account the significant content of organic carbon in these composites, the content of humic compounds which are readily available to microorganisms, is sufficient and does not threaten the transformation of organic matter.

Introducing the considered wastes to composites improved the quality of humic compounds, which was reflected in an increased share of mineralization-resistant humic compounds. The addition of wastes increased the C-HA:C-FA ratio, which is commonly considered an indicator of the humic compounds quality (Tab. 7). The value of C-HA/C-FA in the control soil amounted to roughly 0.55 which is a typical value for soils with poor quality of humic compounds. Significantly higher value of the ratio (0.78) was displayed by R10 composite, i.e. 50% of drilling waste + 50% of mixture of sludges form Lublin and Puławy (1:1 d.m/d.m ratio). The percentage of compounds which are not subject to extraction (humins) in unfertilized soil amounted to approximately 45% of TOC (Tab. 6). On the other hand, in the soil fertilized with manure, it increased to about 48% of TOC. The sludge applied in the amount of 10 and 50 Mg·ha$^{-1}$ decreased the considered fractions of organic compounds to about 51% of TOC. The smallest percentage of humins characterized the substrate comprising drilling waste and the mixture of sewage sludges. Their share approximated 74% of TOC. This may indicate the presence of incompletely humified organic matter.

**Table 5.** Content of C labile fractions in the examined materials
**Tabela 5.** Zawartość labilnych frakcji związków węgla w badanych materiałach

| Symbols | C labile fractions | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | $g \cdot kg^{-1}$ | | | | $g \cdot kg^{-1}$ | | | |
| | I period | II period | III period | Mean | I period | II period | III period | Mean |
| K1 | 3.68 | 3.67 | 3,72 | 3.69 | 51.18 | 51.72 | 51.72 | 51.54 |
| K2 | 3.72 | 3.77 | 3.87 | 3.79 | 50.07 | 50.48 | 50.48 | 50.34 |
| K3 | 4.20 | 4.16 | 4.22 | 4.19 | 47.30 | 46.47 | 46.47 | 46.75 |
| R1 | 4.28 | 4.30 | 4.40 | 4.33 | 44.35 | 44.45 | 44.45 | 44.42 |
| R2 | 4.38 | 4.33 | 4.43 | 4.38 | 44.11 | 44.00 | 44.00 | 44.04 |
| R3 | 4.08 | 4.18 | 4.29 | 4.18 | 25.92 | 26.44 | 26.44 | 26.27 |
| R4 | 3.96 | 4.05 | 4.22 | 4.08 | 25.00 | 25.80 | 25.80 | 25.53 |
| R5 | 3.88 | 3.97 | 4.02 | 3.96 | 43.99 | 44.20 | 44.20 | 44.13 |
| R6 | 3.78 | 3.86 | 4.01 | 3.88 | 27.35 | 27.86 | 27.86 | 27.69 |
| R7 | 4.26 | 4.36 | 4.46 | 4.36 | 21.00 | 21.40 | 21.40 | 21.27 |
| R8 | 5.04 | 4.98 | 5.38 | 5.13 | 14,00 | 14.16 | 14.16 | 14.11 |
| R9 | 4.44 | 4.42 | 4.78 | 4.55 | 16.01 | 16.24 | 16.24 | 16.16 |
| R10 | 6,96 | 7.25 | 7.64 | 7.28 | 11.57 | 12.86 | 12.86 | 12.43 |
| Mean | 4.36 | 4.41 | 4.57 | 4.45 | 32.45 | 32.78 | 32.78 | 32.67 |
| NIR for the reclamation variant | 0.29** | | | | 1.03** | | | |
| NIR for the period | 0.09** | | | | 0.34** | | | |
| NIR variant x period | 0.54 | | | | 1.92 | | | |

***I period*** *– beginning of the experiment (10.07.2013r.);* ***II period*** *– following the harvest of mustard (03.09.2014r.);* ***III period*** *– following the harvest of buckwheat (23.06.2015r.). Sludge from Lublin (L); Sludge from Puławy (P).*

**Table 6.** Content of fractions of humic compounds in the examined materials
**Tabela 6.** Zawartość frakcji związków próchnicznych w badanych podłożach

| Symbols | C released | | | | | | | | Humins | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $g \cdot kg^{-1}$ | | | | %TOC | | | | %TOC | | | |
| | I period | II period | III period | Mean | I period | II period | III period | Mean | I period | II period | III period | Mean |
| K1 | 3.98 | 3.93 | 4.03 | 3.98 | 55.28 | 55.42 | 55.50 | 55.40 | 44.72 | 44.58 | 44.50 | 44.60 |
| K2 | 4.08 | 4.11 | 4.18 | 4.12 | 56.20 | 55.12 | 55.16 | 55.49 | 43.80 | 44.88 | 44.84 | 44.51 |
| K3 | 4.58 | 4.56 | 4.70 | 4.61 | 53.01 | 50.86 | 52.04 | 51.97 | 46.99 | 49.14 | 47.96 | 48.03 |
| R1 | 4.72 | 4.75 | 4.85 | 4.77 | 49.89 | 49.12 | 49.46 | 49.49 | 50.11 | 50.88 | 50.54 | 50.51 |
| R2 | 4.83 | 5.07 | 5.29 | 5.06 | 48.69 | 51.54 | 52.80 | 51.01 | 51.31 | 48.46 | 47.20 | 48.99 |
| R3 | 4.64 | 4.69 | 4.78 | 4.70 | 30.05 | 29.64 | 29.96 | 29.88 | 69.95 | 70.36 | 70.04 | 70.11 |
| R4 | 4.72 | 4.87 | 5.02 | 4.87 | 29.87 | 31.06 | 31.30 | 30.74 | 70.13 | 68.94 | 68.70 | 69.26 |
| R5 | 4.36 | 4.47 | 4.53 | 4.45 | 51.54 | 49.73 | 50.20 | 50.49 | 48.46 | 50.27 | 49.80 | 49.51 |
| R6 | 4.38 | 4.43 | 4.51 | 4.44 | 32.21 | 31.97 | 32.18 | 32.12 | 67.79 | 68.03 | 67.82 | 67.88 |
| R7 | 7.20 | 7.27 | 7.30 | 7.26 | 35.64 | 35.70 | 35.98 | 35.77 | 64.36 | 64.30 | 64.02 | 64.23 |
| R8 | 12.12 | 11.91 | 12.40 | 12.14 | 33.21 | 33.88 | 34.12 | 33.74 | 66.79 | 66.12 | 65.88 | 66.26 |
| R9 | 7.80 | 7.71 | 8.11 | 7.87 | 28.21 | 28.36 | 28.60 | 28.39 | 71.79 | 71.64 | 71.40 | 71.61 |
| R10 | 15.64 | 14.90 | 15.60 | 15.38 | 23.70 | 26.42 | 26.90 | 25.67 | 76.30 | 73.58 | 73.10 | 74.33 |
| Mean | 6.39 | 6.36 | 6.56 | 6.44 | 40.58 | 40.67 | 41.09 | 40.78 | 59.42 | 59.32 | 58.91 | 59.22 |
| NIR for the reclamation variant | 0.42** | | | | | 2.73** | | | 2.73** | | | |
| NIR for the period | 0.13** | | | | | 0.89 | | | 0.89 | | | |
| NIR variant x period | 0.77 | | | | | 5.09 | | | 5.09 | | | |

***I period*** *– beginning of the experiment (10.07.2013r.);* ***II period*** *– following the harvest of mustard (03.09.2014r.);* ***III period*** *– following the harvest of buckwheat (23.06.2015r.). Sludge from Lublin (L); Sludge from Puławy (P).*

**Table 7.** Content of humin and fulvic acids in examined materials
**Tabela 7.** Zawartość kwasów huminowych i fulwowych w badanych materiałach

| Symbols | Humin acids (C-HA) $g \cdot kg^{-1}$ | | | | Fulvic acids (C-FA) $g \cdot kg^{-1}$ | | | | C-HA/C-FA | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | I period | II period | III period | Mean | I period | II period | III period | Mean | I period | II period | III period | Mean |
| K1 | 1.45 | 1.46 | 1.49 | 1.47 | 2.53 | 2.47 | 2.54 | 2.51 | 0.57 | 0.59 | 0.59 | 0.58 |
| K2 | 1.42 | 1.48 | 1.52 | 1.47 | 2.66 | 2.63 | 2.66 | 2.65 | 0.53 | 0.56 | 0.57 | 0.55 |
| K3 | 1.72 | 1.72 | 1.78 | 1.74 | 2.86 | 2.84 | 2.92 | 2.87 | 0.60 | 0.61 | 0.61 | 0.61 |
| R1 | 1.8 | 1.86 | 1.92 | 1.86 | 2.92 | 2.89 | 2.93 | 2.91 | 0.62 | 0.64 | 0.66 | 0.64 |
| R2 | 1.84 | 1.96 | 2.06 | 1.95 | 2.99 | 3.11 | 3.23 | 3.11 | 0.62 | 0.63 | 0.64 | 0.63 |
| R3 | 1.86 | 1.90 | 1.96 | 1.91 | 2.78 | 2.79 | 2.82 | 2.80 | 0.67 | 0.68 | 0.69 | 0.68 |
| R4 | 1.86 | 1.94 | 2.00 | 1.93 | 2.86 | 2.93 | 3.02 | 2.94 | 0.65 | 0.66 | 0.66 | 0.66 |
| R5 | 1.64 | 1.70 | 1.75 | 1.70 | 2.72 | 2.77 | 2.78 | 2.76 | 0.60 | 0.61 | 0.63 | 0.61 |
| R6 | 1.8 | 1.86 | 1.90 | 1.85 | 2.58 | 2.57 | 2.61 | 2.59 | 0.70 | 0.72 | 0.73 | 0.72 |
| R7 | 2.86 | 2.90 | 2.96 | 2.91 | 4.34 | 4.37 | 4.34 | 4.35 | 0.66 | 0.66 | 0.68 | 0.67 |
| R8 | 5.12 | 5.15 | 5.42 | 5.23 | 7.00 | 6.76 | 6.98 | 6.91 | 0.73 | 0.76 | 0.78 | 0.76 |
| R9 | 3.16 | 3.26 | 3.46 | 3.29 | 4.64 | 4.45 | 4.65 | 4.58 | 0.68 | 0.73 | 0.74 | 0.72 |
| R10 | 6.84 | 6.56 | 6.98 | 6.79 | 8.80 | 8.34 | 8.62 | 8.59 | 0.78 | 0.79 | 0.81 | 0.79 |
| Mean | 2.57 | 2.60 | 2.71 | 2.62 | 3.82 | 3.76 | 3.85 | 3.79 | 0.65 | 0.66 | 0.68 | 0.66 |
| NIR for the reclamation variant | 2.11** | | | | 0.48** | | | | 0.03** | | | |
| NIR for the period | 0.07** | | | | 0.16 | | | | 0.01** | | | |
| NIR variant x period | 0.39 | | | | 0.90 | | | | 0.05 | | | |

***I period*** *– beginning of the experiment (10.07.2013r.);* ***II period*** *– following the harvest of mustard (03.09.2014r.);* ***III period*** *– following the harvest of buckwheat (23.06.2015r.). Sludge from Lublin (L); Sludge from Puławy (P).*

## 4. Conclusions

The results of the pot tests carried out on the mixtures of anthropogenic soil, mining waste (drilling waste and coal gangue) and sewage sludge lead to the following conclusions:

- the assessed mixtures were characterized by different organic matter quality and concentration of nutrients,
- the increase of the organic carbon content depended on the amount of exogenous organic matter, supplied by sewage sludge,
- the highest organic carbon content was observed in the substrate contained the same amount of drilling waste and a mixture of sludges from Lublin and Puławy (mixed with the ratio of 1:1 d.m/d.m),
- the optimal share of labile carbon compounds, which satisfied the energy needs of microorganisms and limited the losses of organic carbon, was found in the mixtures in which 100-200 $Mg \cdot ha^{-1}$ of sludge was added,
- the assessed wastes improved the quality of humic substances, which was reflected in a decreased percentage of extracted carbon compounds and increased share of the humic acids to fulvic acids carbon ratio,
- the best quality of humic compounds was observed in the substrate containing 50% of drilling waste and 50% of a mixture of sludges from Lublin and Puławy (mixed with the ratio of 1:1d.m/d.m).

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## Rekultywacja składowisk odpadów wiertniczych

### Streszczenie

W pracy przestawiono wyniki badań właściwości fizykochemicznych materiałów glebopodobnych utworzonych przez zmieszanie w różnych konfiguracjach i proporcjach gleby piaszczystej, odpadów wydobywczych (zwierciny wydzielone ze zużytej płuczki wiertniczej i przywęglowa skała płonna) oraz osadów ściekowych z mechaniczno-biologicznej oczyszczalni ścieków komunalnych w Lublinie i oczyszczalni ścieków przemysłowych w Zakładach Azotowych Puławy. Celem badań było opracowanie sposobu tworzenia biologicznie czynnej pokrywy rekultywacyjnej na składowiskach odpadów wiertniczych. Badania prowadzono w testach wazonowych, z wykorzystaniem gryki (*Fagopyrum esulentum Moench)* i gorczycy (*Sinapis alba*). Jako mieszanki referencyjne zastosowano samą glebę, glebę z dodatkiem nawozów NPK i glebę zmieszaną z obornikiem. Analizowano zmiany zawartości $C_{org}$ i $N_{og.,}$ zawartości frakcji labilnych związków organicznych oraz składu frakcyjnego próchnicy w mieszaninach w trakcie eksperymentu. Stwierdzono, że wprowadzenie osadów ściekowych wpłynęło istotnie na zawartość $C_{org}$ i $N_{og}$ w mieszaninach. Największą zawartością tych pierwiastków charakteryzowało się podłoże złożone w równych proporcjach wagowych z komponentu mineralnego, który stanowiły odpady wiertnicze oraz komponentu organicznego, który stanowiła mieszanina osadów ściekowych z Lublina i Puław, utworzona w stosunku wagowym 1:1 w odniesieniu do suchej masy. Optymalny udział labilnych związków węgla, pokrywający zapotrzebowanie mikroorganizmów na energię i ograniczający straty węgla organicznego stwierdzono w materiale glebopodobnym utworzonym w wariancie z dodatkiem osadu w dawkach 100 i 200 $Mg \cdot ha^{-1}$. Dodatek komponentu organicznego wpłynął na skład frakcyjny próchnicy glebowej, co przejawiło się zmniejszeniem udziału związków węgla ulegających ekstrakcji oraz zwiększeniem udziału humin w związkach próchnicznych. Najlepszą jakością próchnicy charakteryzowało się podłoże złożone z odpady wiertniczych oraz komponentu organicznego, zmieszanych w proporcji wagowej 1:1.

**Słowa kluczowe:**
odpady wiertnicze, składowiska, materiał glebopodobny, rekultywacja

**Keywords:**
drill cuttings, landfill, soil-like materials, reclamation