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# Chemical Properties Transformations of Copper Waste Heaps from Moldova Noua as a Result of their Afforestation

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***Abstract:*** *This paper analyses the chemical transformations of soils from certain copper waste heaps from Moldova Nouă in the context of their afforestation. The analysis is based on a comparison between chemical properties of soils from different areas of the waste heaps, such as the planted and unplanted plateaus and slopes. The soil samples were gathered from soil profiles for each variants considered, and then analysis carried out in the "Marin Drăcea" National Institute for Research and Development in Forestry laboratories. The results were compared and discussed in the context of the waste heaps' morphologic differences, such as land slope, aspect and the presence or absence of forest plantations. As such, the following elements were analyzed and compared: granulometric composition, soil chemical properties, soil reaction, humus content, total nitrogen, potassium content and heavy metals content. Forest vegetation had an important influence on the soil's granulometric composition, on the humus and total nitrogen content, as well as on the soil's reaction. In addition, granulometric composition differences between slopes that are exposed to the main wind and those that are not, suggest the importance of afforestation works for the former.*

***Keywords****: anthropic protosol, soil horizon, soil reaction, nitrogen content*

## 1. Introduction

Mining an important industrial activity, generates multiple and varied negative environmental effects, damaging the atmosphere, lithosphere hydrosphere and biosphere [1]. The degradation of environmental conditions through mining activities leads to an ecological imbalance that affects the life of all living organisms [2]. Field degradation through vertical and horizontal shifting or waste heap landslides are other negative effects that can result from irresponsible mining activities. Through their chemical pollution they can have a long term effect on the soil's fertile properties [1]. Research from coal waste heaps from Anina and Doman show that overlaying vegetation on a waste heap is a complex process especially because herbaceous and/or wood vegetation must develop on a surface with a reduced natural fertility more or less appropriate for the plant's growth and development [3]. The installation of vegetation on waste heaps depends on the presence of bacteria activity which participates in the circuit of elements (nitrogen, carbon sulphur) [4]. In this regard some researchers have studied the evolution of microbiological diversity in Pinu, Puturosu, Dumitrelu and Ilva waste heaps located in Călimani Mountains [5]. Research on the ecological reconstruction of waste heaps from coal exploitations located in the western part of the Getic Plateau for waste heaps afforested with acacia 10-15 years ago, show the start of bioaccumulation and humification processes, a growth of nitrogen content caused by the fast litter decomposition as well as *p*H decrese caused by litter humification (the leaking of carbonates and the presence of fluvic acid) [6]. As for the cultivation of waste heaps and their agrochemical characteristics other authors note that humus formation, mineralization and accumulation processes are slow and stabilize when the maximum potential is reached [7].

Investigations carried out in Lusatia (East Germany) on coal waste heaps show that the forest growing on these mining fields is not weakly developed at least not in the first stages of development. The organic matter cycle is influenced by organic carbon fractions that are unique for these sites [8].

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Investigations carried out decades from the restauration of coal stripping and waste heaps from Zwickau and Lugau/Oelsnitz Saxony (Germany), have shown that a simple revegetation of the waste heaps helps against erosion. However, it does not impede acid drainages as water and oxygen continues to infiltrate waste heaps [9]. A study concerning the concentration of heavy metals on afforested waste heaps was carried out in the mining areas from the Chinese city of Chenzhou and on an uncontaminated control surface from Hong Kong. Significant differences were observed between the control surface and the contaminated soils regarding the content of heavy metals (Pb, Zn, Cu, Cd) and a metalloid (Ca) [10].

Flotation waste heaps (also known as decantation ponds), a result of storing material derived from the flotation process represent anthropic field surfaces in which vegetation development is hard due to sterile instability. This instability is accentuated in the case of Moldova Nouă by winds characteristic to this area [11]. Dust pollution in Moldova Nouă is a result of these surfaces and of the strong wind, and has negative effects even on the other side of the Danube. As a result Romania was forced by international sentencing in 1988 to put a stop to this pollution. In following period the waste heap's plateau was grassed while the slope and part of the plateau were afforested with fast growing species that have an energetic potential [12]. Due to these stabilization works, waste heaps from Moldova Nouă now have a phito-geographic complex with perspectives for the reintegration of these surfaces in the natural landscape Furthermore, the results recorded here are similar in structure with forest areas [13].

## 2. Materials and methods

The research was carried out in two main waste heaps from Moldova Nouă namely "Boșneag" and "Lunca Dunării" (Figure 1). The aim was to evaluate the changes and improvements of the soil's chemical properties after afforestation.

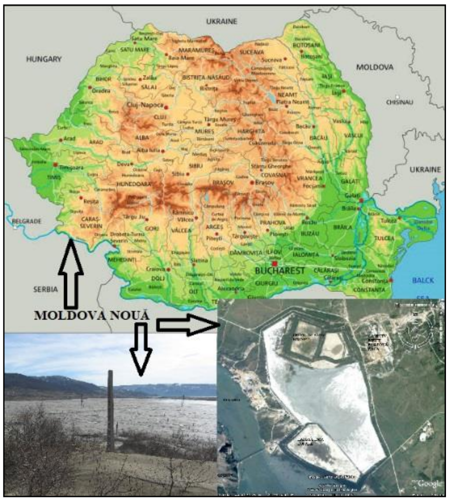


**Figure 1.** The waste heap complex from Moldova Nouă

Specific instruments for gathering and analyzing soil samples were used as research materials. As such, digging tools, such as spades, were used for creating soil profiles, while a measuring tape was used for measuring horizon profiles. In their turn, the samples were analyzed with the help of specific reactive and laboratory utensils.

Observation (regarding the current state of forest vegetation planted on waste heaps, of the horizon, and the soil's characteristics) and experiment (soil analysis) were used as research methods.

The research methods were represented by observation (regarding the current state of forest vegetation planted on waste heaps, as well as the characteristics of horizons and soils) and experiment (soil analysis).

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As such, the research method involved gathering samples from soil profiles located on the waste heap's plateau and slopes, which were constituted in two control plots and nine experimental variants, set out as in figure number two. The samples were chosen from different biotypes from the two waste heaps as follows:

- „Lunca Dunării" waste heap:
  - V1 - Control plot – horizontal field with no vegetation;
  - V2 – Slope with south aspect – the slope's base is covered with soil and planted;
  - V3 - Slope with south aspect – the slope's middle third is covered with soil and planted;
  - V4 – Slope with north aspect – the slope's upper third is covered with soil and planted;
  - V5 - Slope with north aspect - the slope's base is covered with soil and planted;
  - V6 - Slope with north aspect - the slope's middle third is covered with soil and planted;
  - V7 - Slope with north aspect - the slope's upper third is covered with soil and planted.

- „Boșneag" waste heap:
  - V1 - Control plot - horizontal field with no vegetation;
  - V2 – Horizontal field – covered with soil and planted;
  - V3 - Slope with south aspect – the slope's middle third with no vegetation, the A horizon partially smashed;
  - V4 - Slope with south aspect – the slope's middle third is covered with soil and planted, the A horizon partially eroded by wind.


**Figure 2.** The location of experimental and control plots on waste heaps from Moldova Nouă

Soil samples were gathered according to present soil methodology. The samples were then analyzed at "Marin Drăcea" Soil Laboratory with the latest modern instruments and methods: potentiometric method (for determining soil *p*H), Thermo Orion 3 pH-meter (for readings), humid oxidation method (for determining humus), Walkley-Black-Gogoasa and Kjeldahl methods (for determining the titrimetric dosage), humid mineralization (for establishing nitrogen), spectrophotometer in atomic absorption (for determining heavy metals and other elements).

The evaluation of changes and improvements of the soil's chemical properties from Moldova Nouă waste dumps after afforestation, was achieved by comparing the results of soil's analysis for samples gathered from the variants that present forest vegetation with the results of soil analysis for samples gathered from control plots and areas with no afforestation works.

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## 3. Results and discussions

The soil of waste heaps from Moldova Nouă is an anthropic protocol resulted from the processing of local granodiorite (“banatite”). The soil’s sandy-dusty-clay texture is why large quantities of fine particles can be carried by winds. Gravel represents approximately 1/4 of the fine sand and is shows more stability for wind; approximately 1/3 of the total is represented by dust particles (which are easily carried by wind), while clay occupies approximately 1/4 up to 1/3. A part of clay, namely 10-15% is represented by fine, small particles (under 0.002 mm). This fine texture favors the rising of dust from unstable areas during periods with strong winds.

The granulometric composition analysis done for the first 10 cm of soil (Trable 1, Figure 3 and 4) has revealed that fine granulometric fractions (dust and sand) from the planted area of Boșneag waste heap are larger in percentage than in the unplanted area (Figurre 3). This fact proves the positive influence of vegetation in fixing fine soil particles, keeping them on ground and reducing deflation.

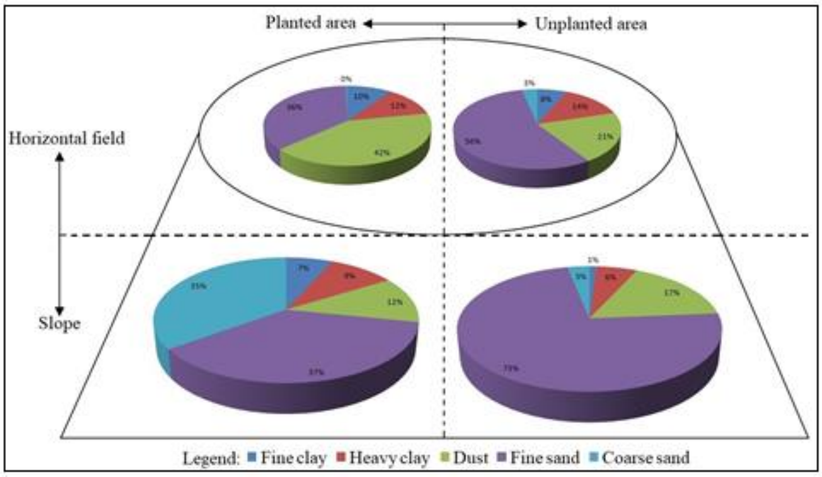


**Figure 3.** The variation of the soil’s granulometric composition from Boșneag waste heap, Moldova Nouă (schematic profile of waste heap)

Furthermore a difference of textures between samples was observed in the Lunca Dunării waste heap namely a higher percentage of coarse particles (sand) on the waste heap’s south slope which can be explained by looking at the wind’s main direction (Figure 4).

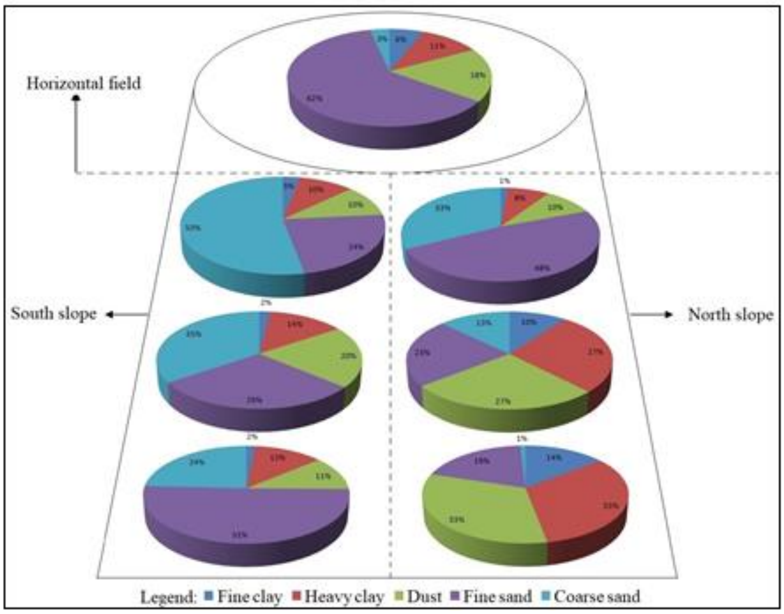


**Figure 4.** The variation of the soil’s granulometric composition from Lunca Dunării waste heap, Moldova Nouă (schematic profile of waste heap)

**Table 1.** The soil's granulometric composition from Moldova Nouă waste heaps

| Nr. crt. | Biotope | Depth (cm) | Granulometric fractions (%) | | | | |
|---|---|---|---|---|---|---|---|
| | | | Heavy clay | Fine clay | Dust | Coarse sand | Fine sand |
| A. ″Lunca Dunării″ waste heap | | | | | | | |
| 1 | Control plot<br>Horizontal field lacking vegetation. Platform | 0-25 | 11.0 | 6.1 | 17.8 | 3.5 | 61.6 |
| | | 25-40 | 17.3 | 2.6 | 14.3 | 19.0 | 46.8 |
| | | 40-72 | 6.8 | 0.3 | 8.9 | 3.5 | 80.5 |
| 2 | South slope<br>The inferior third (base) covered (allochthon soil) and forest vegetation introduced | 0-5 | 12.3 | 1.6 | 11.3 | 24.2 | 50.6 |
| | | 5-35 | 6.9 | 0.4 | 6.4 | 36.6 | 49.7 |
| | | 35-50 | 8.7 | 2.0 | 8.9 | 30.4 | 50.0 |
| 3 | Idem<br>The slope's medium third | 0-20 | 13.6 | 2.0 | 20.5 | 34.5 | 29.4 |
| | | 20-63 | 5.7 | 0.5 | 7.6 | 44.0 | 42.2 |
| 4 | Idem<br>The slope's superior third | 0-10 | 9.9 | 3.1 | 10.5 | 53.0 | 23.5 |
| | | 10-50 | 9.6 | 2.4 | 15.4 | 58.1 | 14.5 |
| | | 50-90 | 5.1 | 0.3 | 5.1 | 47.9 | 41.6 |
| 5 | North slope<br>The inferior third with vegetation and covered (allochthon soil) | 0-20 | 32.6 | 14.4 | 32.8 | 0.9 | 19.3 |
| | | 20-90 | 1.5 | 0.3 | 13.9 | 28.1 | 56.2 |
| 6 | Idem<br>The medium third | 0-8 | 27.5 | 10.2 | 26.7 | 12.8 | 22.8 |
| | | 8-50 | 17.8 | 8.9 | 18.4 | 24.8 | 30.5 |
| 7 | Idem<br>The superior third | 0-33 | 8.0 | 1.4 | 9.8 | 32.6 | 48.2 |
| | | 33-41 | 18.0 | 13.5 | 18.8 | 29.1 | 20.6 |
| B. ″Boşneag″ waste heap | | | | | | | |
| 1 | Control plot<br>Horizontal field lacking vegetation (uncovered)<br>Platform | 0-22 | 12.2 | 9.7 | 41.4 | 0.3 | 36.4 |
| | | 22-36 | 6.8 | 1.2 | 10.1 | 3.2 | 78.7 |
| | | 36-43 | 11.2 | 2.4 | 23.2 | 0.5 | 62.7 |
| | | 43-75 | 4.1 | 0.4 | 7.2 | 11.7 | 76.6 |
| 2 | Horizontal field (platform)<br>covered and with plantations<br>(oleaster, locust, sea buckthorn) | 0-3 | 13.7 | 6.5 | 20.5 | 3.3 | 56.0 |
| | | 3-26 | 12.5 | 4.6 | 14.6 | 3.7 | 64.6 |
| | | 26-39 | 15.6 | 7.2 | 13.9 | 34.4 | 28.9 |
| | | 39-67 | 6.5 | 0.9 | 7.5 | 2.0 | 83.1 |
| | | 67-95 | 5.9 | 0.9 | 10.2 | 4.9 | 78.1 |
| 3 | South slope, medium third,<br>lacking vegetation (uncovered), A horizon partially shattered | 0-29 | 5.9 | 0.5 | 16.9 | 3.2 | 73.5 |
| | | 29-52 | 2.2 | 0.2 | 23.4 | 4.0 | 70.4 |
| | | 52-90 | 8.0 | 1.5 | 10.3 | 2.7 | 77.5 |
| 4 | Idem<br>with vegetation and covered (allochthon soil),<br>A horizon partially eroded by wind | 0-5 | 9.5 | 6.7 | 12.0 | 35.0 | 36.8 |
| | | 5-35 | 3.8 | 0.4 | 7.2 | 36.8 | 51.8 |
| | | 35-47 | 7.1 | 0.8 | 5.5 | 46.9 | 39.7 |
| | | 47-87 | 17.5 | 19.0 | 23.4 | 21.2 | 18.9 |

In this regard, wind has an important role in disseminating fine soil particles and should be taken into account when planting the slopes of waste heaps exposed to this phenomenon. Fine sands predominate on plain surfaces (platforms), while coarse sands can be found on slopes. As such, the main processes are represented by sedimentation (on plateaus) and erosion and deflation (on slopes). Other authors have also observed that dune ridges are less evolved soils, underdeveloped profiles and poorly supplied with nutrients compared to dune slopes and interdunes, whose physical and chemical properties are more favorable to plant growth [14].

The waste heap's soil transform steadily in a forest soil, under the influence of vegetation. A distinct soil horizon occure especially on plateaus and at the base of afforested slopes. The humus has a good quality, being mull calcic, black-brown in color and well structured (glomerular structure). The litter's decomposition is very fast (3-5 months).

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In regard to the soil's chemical properties, it can be observed that the soil has a weak alkaline reaction.

The soil's acidity increases (*p*H decreases) in the soil's first 25 cm on plateaus and south planted slopes. This fact emphasizes once-again the importance and influence of vegetation in improving pedo-genetic conditions under very unfavorable conditions present on waste heaps (Figure 5).

The soil's acidity strongly decreases between 10 and 20 cm (*p*H increases). A significant drop in acidity can be observed for all soil horizons of unplanted plateaus and slopes. Testing the significance of *p*H increase, between planted and unplanted areas from tailing dumps (Table 2), reveals a significant difference between the *p*H values of planted and unplanted areas (***$p < 0.001$, t-Test).

**Table 2.** Statistical t-Test results for *p*H increases between planted and unplanted areas (Data are ex-pressed as mean ± S.D.)

| t-Test: Two-Sample Assuming Unequal Variances | | |
|---|---|---|
| | pH increases | |
| | *Planted* | *Unplanted* |
| Mean | 7.618148 | 7.953636 |
| Variance | 0.032062 | 0.041725 |
| Observations | 27 | 11 |
| Hypothesized Mean Difference | 0 | |
| df | 17 | |
| t Stat | -4.7537 | |
| P(T<=t) one-tail | 9.2E-05 | |
| t Critical one-tail | 1.739607 | |
| P(T<=t) two-tail | 0.000184 | |
| t Critical two-tail | 2.109816 | |

The soil lacks humus in the unplanted areas and becomes weakly humiferous in the ones where forest vegetation was installed (Table 4). As it can be seen in table 3, t-Test results indicate significant differences among soil samples from planted and unplanted areas (***$p < 0.001$, t-Test).

**Table 3.** Statistical t-Test results for humus content variation between planted and unplanted areas (Data are expressed as mean ± S.D.)

| t-Test: Two-Sample Assuming Unequal Variances for Humus content | | |
|---|---|---|
| | Humus content | |
| | *Unplanted* | *Planted* |
| Mean | 0.02 | 0.901111 |
| Variance | 0.0001 | 0.786964 |
| Observations | 3 | 27 |
| Hypothesized Mean Difference | 0 | |
| df | 26 | |
| t Stat | -5.15807 | |
| P(T<=t) one-tail | 1.11E-05 | |
| t Critical one-tail | 1.705618 | |
| P(T<=t) two-tail | 2.22E-05 | |
| t Critical two-tail | 2.055529 | |

The acidity also decreases on the northern planted slope that is exposed to the main wind. However the decrease is much slower and constant up to higher depths. The acidity increases constantly up to higher depths on the south planted slope (Figure 5).

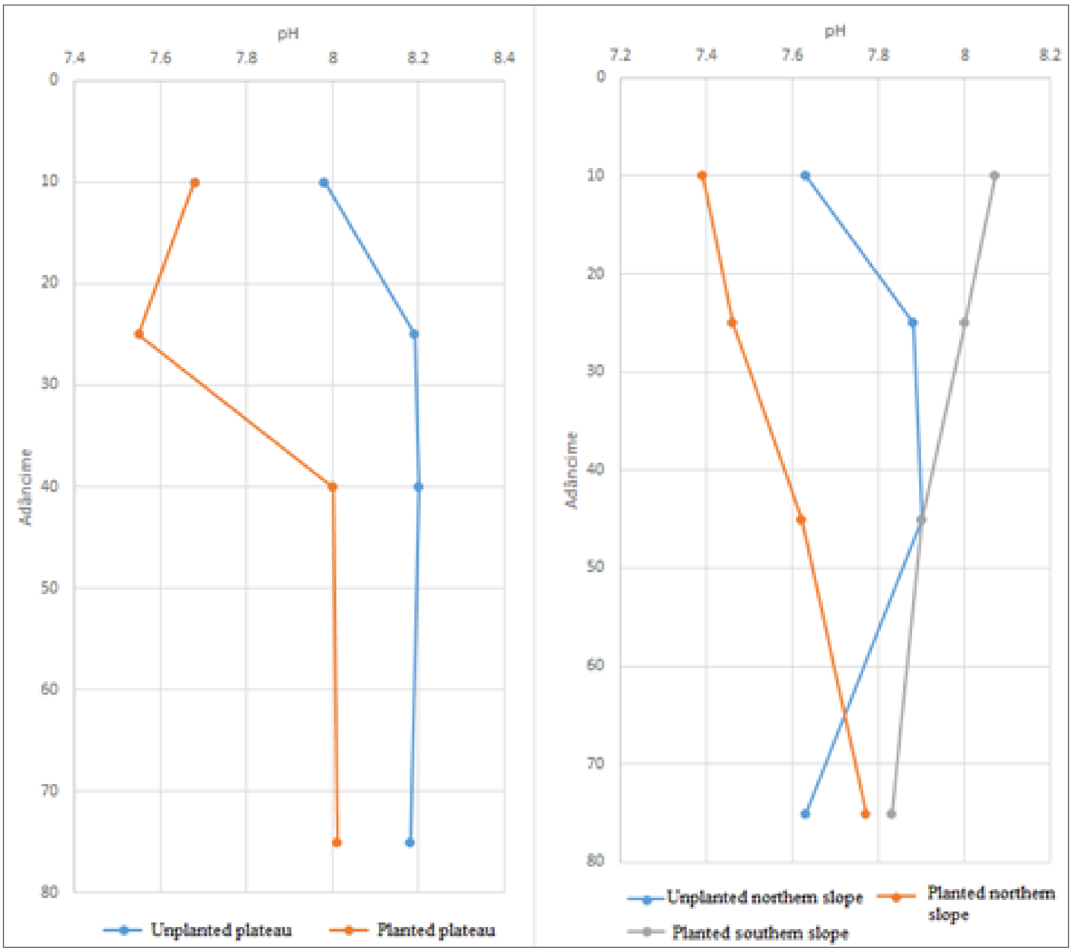


**Figure 5.** The soil acidity from Moldova Nouă waste heaps
(plateau – left and slope – right)

Looking at nitrogen fertility the soil is average. However nitrogen does not originate from the waste heap material but is of secondary origin and brought in with the covering soil (Table 4).

It is important to mention that nitrogen-fixing species (locust, oleaster, sea buckthorn. clover) were introduced on waste heaps together with improvement works.

On the other hand the soil is sufficiently supplied with potassium originating from the waste heap's sterile (Table 4).

Amongst heavy metals (Mn, Zn, Cu, Cr, Ni) copper records quantities that much exceeds the admissible limits. As such the soil is excessively loaded with this element a fact that can be explained by the qualities of rocks that are used even for extracting this mineral. As for the other metals the soil is weak up to moderately polluted (Table 4). In an abandoned mining area from Hunedoara County the results obtained for soil samples show an overrun of the alert and / or intervention threshold for the following metals: arsenic cadmium cobalt copper manganese nickel lead and zinc [15].

**Table 4.** The chemical properties of soils from Moldova Nouă waste heaps

| Biotope | Depth (cm) | pH | Humus (%) | Nt (%) | K (%) | $CO_3Ca$ (%) | Mn ppm | Zn ppm | Cu ppm | Cr ppm | Ni ppm |
|---|---|---|---|---|---|---|---|---|---|---|---|
| A. ″Lunca Dunării″ waste heap | | | | | | | | | | | |
| Control plot I N slope, lacking vegetation 1/3 medium | 0-22 | 7.63 | 0.02 | - | 10.50 | 8.2 | 244 | 206 | 509 | 19.76 | 17.98 |
| | 22-42 | 7.88 | - | - | 10.65 | 7.4 | 231 | 179 | 676 | 29.56 | 16.21 |
| | 42-57 | 7.90 | - | - | 9.14 | 5.6 | 231 | 85 | 277 | 10.83 | 7.56 |
| | 57-80 | 7.63 | - | - | 1.73 | 5.2 | 131 | 104 | 564 | 18.76 | 12.88 |
| N basis slope, with vegetation and covered | 0-18 | 7.73 | 3.18 | 0.146 | 15.84 | 2.2 | 711 | 153 | 291 | 40.52 | 41.74 |
| | 18-90 | 7.74 | 0.03 | - | 4.29 | 24.0 | 610 | 410 | 674 | 11.14 | 6.85 |
| Idem 1/3 medium | 0-8 | 7.39 | 1.11 | 0.061 | 17.23 | 16.5 | 617 | 126 | 383 | 40.45 | 44.85 |
| | 8-19 | 7.46 | 0.90 | 0.97 | 3.17 | 24.8 | 543 | 542 | 1724 | 7.31 | 9.11 |
| | 19-73 | 7.62 | 0.36 | 0.05 | 4.71 | 6.4 | 289 | 146 | 1001 | 12.58 | 9.53 |
| | 73-90 | 7.77 | 0.45 | - | 5.27 | 3.0 | 98 | 94 | 1163 | 10.34 | 7.54 |
| Idem 1/3 superior | 0-33 | 7.52 | 1.80 | 0.049 | 18.41 | 3.8 | 197 | 106 | 582 | 16.57 | 12.16 |
| | 33-41 | 7.26 | 2.24 | 0.097 | 15.22 | 1.8 | 449 | 153 | 258 | 37.78 | 36.80 |
| | 41-70 | 7.24 | 0.03 | - | 7.66 | 3.2 | 167 | 118 | 692 | 17.45 | 16.11 |
| S slope with vegetation and covered | 0-3(5) | 7.56 | 1.23 | 0.085 | 15.21 | 12.4 | 394 | 785 | 1581 | 15.25 | 13.02 |
| | 5-35 | 7.51 | 0.03 | - | 5.04 | 19.4 | 392 | 948 | 1758 | 13.03 | 7.55 |
| | 35-88 | 7.57 | 0.18 | 0.023 | 3.53 | 15.2 | 372 | 885 | 2110 | 10.69 | 9.44 |
| Idem 1/3 medium | 0-20 | 7.51 | 1.65 | 0.073 | 6.00 | 12.2 | 770 | 107 | 62 | 23.59 | 20.56 |
| | 20-63 | 7.70 | 0.36 | - | 5.00 | 16.8 | 387 | 522 | 1459 | 13.80 | 7.63 |
| | 63-90 | 7.51 | 0.48 | - | 4.50 | 16.2 | 401 | 559 | 2102 | 11.37 | 8.99 |
| Idem 1/3 superior | 0-10 | 7.68 | 1.44 | 0.122 | 10.24 | 7.8 | 734 | 94 | 98 | 16.53 | 17.04 |
| | 10-50 | 7.67 | 1.32 | 0.090 | 2.38 | 10.6 | 608 | 85 | 26 | 14.06 | 17.59 |
| | 50-90 | 7.51 | 0.39 | - | 4.71 | 10.6 | 309 | 302 | 1005 | 11.26 | 6.61 |
| B. Boşneag waste heap | | | | | | | | | | | |
| Control plot II horizontal field, platform | 0-22 | 7.98 | 0.01 | - | 19.15 | 28.6 | 708 | 235 | 432 | 20.94 | 7.75 |
| | 22-36 | 8.19 | - | - | 6.16 | 29.2 | 534 | 423 | 691 | 13.51 | 3.67 |
| | 36-43 | 8.20 | - | - | 9.72 | 28.6 | 705 | 268 | 426 | 18.14 | 5.48 |
| | 43-75 | 8.18 | - | - | 5.84 | 24.0 | 524 | 480 | 640 | 11.05 | 3.21 |
| Plateau with vegetation and covered | 0-3 | 7.70 | 1.63 | 0.050 | 10.77 | 29.6 | 681 | 435 | 799 | 15.07 | 6.49 |
| | 3-26 | 7.68 | 0.36 | 0.016 | 9.26 | 29.4 | 622 | 443 | 838 | 12.90 | 7.16 |
| | 26-39 | 7.55 | 0.72 | 0.094 | 10.35 | 13.6 | 484 | 268 | 409 | 19.04 | 14.49 |
| | 39-67 | 8.00 | 0.03 | - | 3.97 | 28.8 | 674 | 433 | 591 | 12.53 | 3.18 |
| | 67-95 | 8.01 | 0.32 | - | 4.57 | 30.0 | 683 | 451 | 522 | 14.18 | 3.83 |
| Control plot III S slope. lacking vegetation | 0-29 | 8.07 | 0.03 | - | 5.27 | 31.0 | 639 | 549 | 596 | 12.80 | 3.97 |
| | 29-52 | 8.00 | - | - | 7.80 | 31.0 | 651 | 476 | 679 | 12.41 | 6.47 |
| | 52-90 | 7.83 | - | - | 8.14 | 29.6 | 720 | 556 | 519 | 13.60 | 8.18 |
| S slope 1/3 medium with vegetation and covered | 0-5 | 7.77 | 0.63 | 0.049 | 3.44 | 25.6 | 642 | 1026 | 1834 | 9.67 | 5.96 |
| | 5-35 | 7.76 | 3.02 | 0.134 | 3.85 | 7.2 | 314 | 323 | 925 | 16.44 | 7.93 |
| | 35-47 | 7.66 | 0.12 | - | 3.69 | 23.2 | 575 | 1245 | 2275 | 8.59 | 6.01 |
| | 47-87 | 7.61 | 0.32 | - | 15.09 | 0.6 | 719 | 160 | 141 | 36.41 | 31.85 |

## 4. Conclusions

Through their growth and development, forest plantations from Moldova Nouă waste heaps generate considerable quantities of wood, foliar biomass, fruits and seeds. A part is continuously stored on the soil's surface as litter, contributing significantly to the soil's improvement. This is proven through the investigations and results that show that the soil is weakly humiferous in areas in which forest vegetation was installed, compared with unplanted areas where the humus is missing.

Apart from improving the soil, the litter produced by the rich foliage of wood or grass species also stabilizes the sandy sterile, reducing deflation and changing and stabilizing the soil's granulometric structure.

The creation of specific forest microclimates also contributes to the reduction of soil acidity. As such, *p*H increases were recorded in areas covered by forest vegetation compared with the ones that lack vegetation.

Nitrogen fixing species successfully fulfill their role as this element is found in the soil of afforested waste heaps. The nitrogen has a local origin, besides its allochthone one originating from the soils used for covering.

The soil's sufficient potassium supply is ensured by the waste heaps' sterile which contains significant quantities of potassium.

From the point of view of heavy metal pollution, the soil is weak up to moderately polluted, with the exception of copper, which registers the highest transgressions of admissible limits. This element remains in large quantities in the sterile, even after processing and extraction.

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