EFFECTS OF SOIL TREATMENT BY COAL MINING CARBONIFEROUS WASTE SLUDGE IN MAIZE GROWING

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ABSTRACT

The multifuncional role and importance of organic matter in soil is widely known. It is also known that the organic matter in soil is subjected to microbiological-biochemical processes of transformation, which includes synthesis of humus as well as it's decomposition -mineralization. Mineralization means transformation-decomposition of organic matter by microbiological processes to mineral products; plant nutrients and water + CO_2 as starting and ending component of photosyntesis. Nutrients are partly plant available with fertilizing effect, partly lost from the soil - leaching in ground water, causing it's eutrophication, but CO_2 in atmosphere participates in greenhouse effect. Practically, mineralization means decreasing of organic matter content in soil and soil degradation [1,4].

In natural ecosystems (phytocenoses natural forests and meadows), it is almost a balanced between inflow and consumption of organic matter, while the cultural and anthropogenic soils agrobiocenosis in general, this relationship is disturbed that there is a disproportion between the inflow and loss [1,4]. Therefore, various materials that contains organic material (waste, various flotation, sludge, etc.) are often used with more or less success. One of such materials, as well as the potential fertilizer, is carboniferous lake sludge like waste of coal mining sedimented at the bottom of the lake in huge quantities, which is the subject of our reasearch. The research were conducted to determine its fertilizing efects and value for repairing of physical and chemical properties of soil.

The research refered to:
- Laboratory analysis of physical and chemical characteristics of the carboniferous sludge samples,
- Analysis of soil of the experimental field
- Research on heavy metals concentration in soil of the experimental farm and in carboniferous sludge, and

Research of fertilizing effects of sludge, comparative mineral fertilizer and farmyard manure treatment by field trials with 4 variants and 4 repetitions.

Soil and other laboratory research were conducted using standard methodologies using in soil science, while in the field experiments conducted using the exact field experiments [2]. Investigations were carried out on the culture of maize (Zea Mays) and lasted for one year (2009.).

The results are encouraging because there had been a series of positive changes in the soil, yields of above ground mass mass and grain of maize were more than 50% (50,2%) higher compared to the control. The treatment applied lake sludge there was a series of positive changes in physical and chemical properties of soil. In particular, it significantly increased the content of organic matter or humus, from 3 to over 4% in the soil, humus content increased on average about 20%.

The presence of heavy metals and other pollutants in carboniferous sludge do not exceed the maximal allowed concentration (M.P.S.), on the basis of instructions for defining the allowed quantities of harmful substances in soil and methodology of its analysis (the Gazzete of FBiH No 11/99). By this Instruction are identified the substances considered to be harmful for agricultural soil, the allowed quantities of harmful substances in soil, measures for preventing soil pollution and contamination control.

KEYWORDS: Carboniferous waste coal mining sludge, humus, heavy metals in sludge, fertilization

INTRODUCTION

Waste coal mining carboniferous sludge, which is the subject of our research, represents a dislocated carboniferous suspended material from the separation of mines Banovići and Đurđevik, sedimented at the bottom of the lake “Modrac“. This lake, or reservoir, was made in 1964. on the river Spreča, upstream from Lukavac and covers an area of 1.700 ha. This water accumulation, functioning as water supply for technology industrial plants in Tuzla and Lukavac, is formed by Turija River and its tributaries Oskava and Gostelja. These water streams hydrodynamically dislocate carboniferous material sedimenting as sludge at the bottom of the reservoir. Estimates indicate that the daily sedimentation is over 700 m^3, so that the current amount is estimated at 12 -14 million m^3 [5].

According to criteria of modern *homo oeconomicus* in our country, this material is classified in the industrial waste of coal mining. The term “waste”, as a natural category is unacceptable. Nature knows no waste, because everything that leaves a natural cycle enters the second, which establishes the natural chain balance.

Great world scienist, creator of the natural system of elements, D. I. Mendeleev more than a century ago defined this problem as follows: “there is no waste, there is only ignorance of how to use the waste” [10].

Therefore this sludge may be defined as a secondary raw material with the relevant use-values. It is indisputable that this is a material of carboniferous composition, and it is known that the usefull in land reclamation values that farmers and the owners of coastal land were convinced in. They are using this sludge for a long time as organic fertilizer. No presence of heavy metals that might endanger the usability of food or plant was determined. We have decided to eliminate that failure [11].

Based on the findings of previous information and experiences of local farmers, it is only a hypothetical state that this sludge can have a variety of practical value such as:
- organic fertilizer,
- fill the production of organic-mineral fertilizers,
- products for energy needs,
- to obtain products for stimulating plant growth and metabolic feature products,

However, our interest is related to the evaluation of the waste sludge as an organic fertilizer to improve the physical and agrochemical properties of soil and application value with the expected content of heavy metals. During 2009. year, some analyses and research investigation have been conducted, which are elaborated in this paper. The research comprised the following activities:
- analyse of basic physical and chemical properties of sludge to establish applicability for fertilization,
- analysis of soil of the experimental field, initially and finally - at the end of the exact field trials,
- heavy metals concentration in the soil and sludge to establish the acceptability of sludge.

Farmers were the first who realized the importance of coal for plants. On their own land, nearby the mines, they used waste of coal as organic fertilizer. According to the quotation of A. Tavčar [16], the first research workers who attributed fertilizing effects to its organic substance were Kisel (1928. and 1931.), Kisel and Liese (1930, 1931, 1932, 1935.). All efforts of use of waste of coal mining sludge as fertilizer is necessary at first to investigate using the exact field trials with main crops as maize.

Till the end of the Second World War stayed within the frames of laboratory research and did not give answers to a large number of questions. After the Second World War, many countries of American and Euro-Asian continent (SAD, Russia, Japan, Austria, Poland, Germany etc.) are intensifying the studies of organic substance in coal as fertilizer. In the former Yugoslavia, the significant results on this issue were made by a group of experts from Croatia and Slovenia. In Bosnia and Hecegovina, the studies covering this topic are rare. Resulović research on coal of Đurđevik mine and Omerćehajić et al Field experiments on salt with coal components [11, 12, 13,].

MATERIAL AND METHOD OF INVESTIGATION

In accordance with the set objective, we analyzed the investigation in four characteristic and specific groups.

Soil of the experimental field

These investigations were conducted on two occasions, before and after establishing the exact field experiment with corn, with the aim of comparative analysis of the impact of application of carboniferous sludge and other usual fertilizers onto the possible positive changes in soil plot. In both cases, studies were carried out from average soil samples of two ground depths, from 0 -10 cm and from 10 -20 cm, for each treatment separately. Average sample is made from 5 respective “pricks” on each replication ie a total of 20 “pricks” from mixed soil sample was obtained 1 average sample. In thi way, 8 samples were prepared at the start of studies (2008.) as initially, and 8 samples at the end of the experiment after 19 months (2009.) as finally one. The soil sample analysis found some important characteristics according to standard methodologies. All analysis in this paper are made in Laboratory of Departrment of Soil Science of Faculty of Agriculture University of Sarajevo.

Out of physical characteristics there was only soil texture examined by pipet method B, sedimentation in water with a previous dispersion with Natrium pyrophosphate. This method determines the soil particles as follows: sand, 2 to 0,02 mm in diameter, silt from 0,02 to 0,002 mm, and clay, <0,002 mm.

Following chemical characteristics were analysed:
- soil reaction - active and substitution (pH in H_2O and KCl) on pH meter with combined electrode in the suspension of 1:2.5,
- humus content in soil, colorometrical with prior oxidation with $K_2Cr_2O_7$,
- content of organic components, heating on temperature of 550^0C,
- $CaCO_3$ content using Scheibler kalcimetre,
- plant available P_2O_5 content in mg/100 g soil, by colorometrical method,
- plant available K_2O content in mg/100 g soil, by flame photometry.

Field and laboratory investigations of sludge

Carboniferous sludge samples were prepared by taking this material in the coastal part of the lake “Modrac” at the site between Kiseljak, Suhe and Šerića. On this site there were three characteristic places selected at a distance of several hundred meters, from which the screw probe was used to take samples from the 0-400 cm profile of the mightiest black carboniferous sludge. For average sample preparation the sludge was taken throughout the length of the profile of the probe and thus have obtained three average samples for analysis.

On such prepared samples, some laboratory analysis according to standard pedological methods were carried out.

Study of heavy metals content in soil and sludge

Research on heavy metal content in soil were carried out after field trials (19 months), ie, after application of sludge and other fertilizers, in certain variants. The content of the following elements was analysed: As, Zn, Pb, Cd and Ni. The obtained results are expressed in mg/kg soil, as total content and were analysed by atomic absorbing spektometry after aqua regia extraction (HNO_3/HCl), and according to regulation HRN ISO 11 047: 2004.

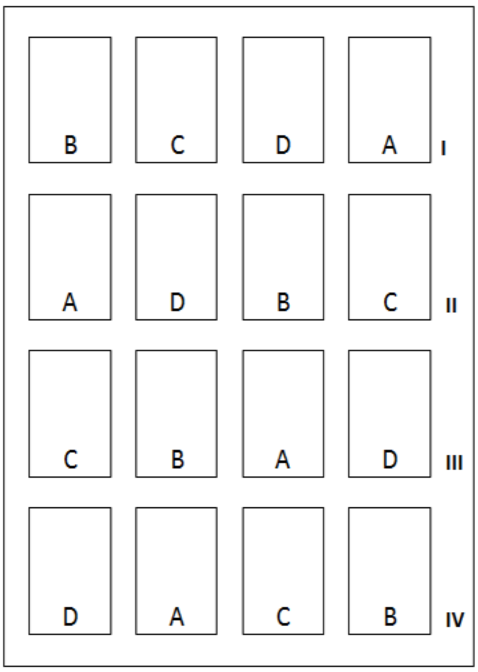
Analyses on the heavy metals concentration were carried out on samples of carboniferous waste coal sludge, using the same methodology as for soil samples.

Methodology of the exact field trial

In the experiment scheme (Figure 1.) are vividly displayed variants (treatments), the shape and size of the experimental plots, replications, and other materials used for this experiment. The method of random distribution of variants within one replication - Latin square was used with 4 variants in 4 replications.
Experimental field area: 268m^2,
Plot area: 12m^2

Variants:
A – CONTROL VARIANT – without any treatment,
B – FERTILIZATION - NPK and 700 kg / ha,
C – FARMYARD MANURE - 50 t / ha,
D – CARBONIFEROUS SLUDGE - 100 t / ha

EXPERIMENT SCHEME
SCHEDULE OF VARIANTS LATIN SQUARE 1: 200



Replications: I, II, III, IV

Establishment and crop growing practices of maize (Zea mays) in experiment, during the vegetation period 2009. year, were carried out by following the dynamics of work:

The primary purpose of preparing the soil for planting maize in the experiment was carried out by plowing to a depth of 28 cm, in autumn 2008. year. Just before plowing the soil samples were taken for soil analysis – initial state.

In spring of 2009. according to the scheme of the experiment 16 experimental plots were edged and variants and replications were marked on the ground.
- There were 4 variants, each made with 4 replications, as follows:
- CONTROL - without application of fertilizers or substrates (control),
- NPK FERTILIZATION - NPK mineral fertilizer (15:15:15) 0,84 kg/experimental plot,
- Fy. MANURE - farmyard manure 60 liters/experimental plot,
- -SLUDGE - carboniferous sludge, 120 liters/experimental plot.

Sowing soil preparation and fertilizer input was carried out manually to a depth of 25 cm. Sowing of maize was also carried out manually in rows 70x20 cm. At each experimental plot were formed 5 rows with 18 plants in the row, or a total of 90 plants, which corresponds to the crop density of 75.000 plants/ha.

Crop growing practices on corn during the growing season is carried out according to normal practice in this crop and there have been done two irrigation in dry period.

Harvesting of corn was performed on 6th of October in the crop stage of physiological maturity.

Data processing methods

In the mathematical and statistical analysis we have taken two indicators, namely: total above-ground plant green mass of maize and dry grain yield, expressed in kilograms per hectare (calculated on hectare).

Data analysis for the assessment of the significance of differences in the properties of the examined variants was carried out by “p” test over the “t” test in which the “t” empirical. The calculation was performed for 7 degrees of freedom and the probability 0,05 = 2,365; 0,01 = 3,449, and 0,001 = 5,405. In this way, identified three levels of statistical significance were identified as follows: *statistically significant difference, statistically highly significant difference**, statistically very highly significant difference***, no statistically significant difference^{0}.

RESULTS AND DISCUSSION

Physical and chemical properties of sludge

The focus of complex research of this paper is carboniferous waste coal sludge in order to valorize it's influence – effects on the soil and its fertility. Investigation includes texture (mechanical composition) and some relevant agrochemical properties of sludge.

a) The texture of carboniferous sludge

The average relative content ratio of particles of different diameters in the sludge is:
- 75.6% of sand (particles of 2 - 0,02 mm) with the interval 75,2 - 75,8%, and almost no variation between samples, (Vc = 0,4%),
- 8,9% of silt (particles with 0,02 -0,002 mm), with variation in the range of 8,5- 9,3% and in very small variation (Vc = 4%),
- 15,5% of clay as colloidal fraction (particles <0,002 mm, with the interval 15,3 - 15,7%, almost no variation (VC = 1%).

Following criteria using for soil, the texture of the sludge would belong to the sandy loam (SL). According to Gračanin [6] regarding the contents of colloids the sludge would be as moderate colloidal (10 - 20% of particles <0,002 mm).

b) Agrochemical properties of sludge

The reaction of sludge is as follows:

pH in H_2O = 7,3 - a slight variation from 7,1 to 7,4 (Vc = 2%).
pH in nKCL = 6,6 - very little fluctuation from 6,1 to 6,8 (VC = 5%).

Therefore, this sludge is of neutral to slightly alkaline reaction, although it is known that coal is predominantly acid reaction [9]. This is because along with the accumulated sludge, hydrodynamic transport and other substances are passeed primarily marl substances with higher content of carbonates, which are directly influenced the reaction of the sludge.

The humus content in the sludge was 14,13% with variation in the interval from 13,7 - 14,5% - a slight fluctuation (Vc = 2%).

However, studies that were conducted at the Mining University in Tuzla, in earlier years, gave slightly different results, or the content of humic substances, was on average 21,4% [11]. In addition, further extraction and separation of different humic substances shows the following results:

Total humic acids 10% of which are:
- free 0,87%
  bound 9,13%

Various salts (Humate), 11,4% of which are:
- easily soluble 0,88%
  hardly soluble 8,26%

In the course of further research it would be very useful to investigate in details the contents of humic substances present in the sludge, because it is practically its most important value.

The contents of plant available nutrients is very low and amounts:
- P_2O_5 average of 4,6 mg/100gr of mass, in the range 3,7 - 5,5 (Vc = 16%).
- K_2O average 18,5 mg/100gr of mass, in the interval 17,0 - 20,8 (Vc = 9%).

Accordingly, the sludge belongs to materials with low content of available nutrients, which means that its value should be sought in influence on other physical and chemical properties of soil, primarily in the content of organic matter and humus in which they are safe and stimulating nutritive substances for plant growth and development.

Explored values of the adsorption complexes are as follows:
- "S "= 96,91 cmol, "T" = 98,4 cmol, "H" = 1,49 cmol "V" = 98,44%.

Therefore, this sludge is classified as a rich substrate adsorbed basic cations, and the character of humus is saturated with bases [6].

In this study, the most important results were achieved by the Institute in Saint Petersburg, which has developed a new method of extraction of humic acid from coal. By a further elaboration they get of a wide range of concentrated products humic (Leonardit) in the form of K-humat, NH_4 humat, Mg humat. When humic acid is taken to one liter of the concentrate that corresponds to value 7 - 8 tons/ha of manure.

Properties of soil of the experimental field

The soil on which field experiment with corn was realised, belongs to the class of humus-accumulative soils, soil type is Leptosol with clear typical A - C horizons in profiles. The previous crop was vegetable [12;13].

By waste sludge application (treatment "Sludge") for only 19 months (2008. and 2009.) there were relevant positive changes in the soil that can be seen from Table 1.

Table 1. Changes of properties of soil of experimental field (18 mounths after treatmnet)

INDICATORS	Control		Fertilization		Fy. Manure		Sludge	
	initial	final	initial	final	initial	final	initial	final
Texture								
Sand (2,0-0,02 mm)	14,80%	18,80%	14,80%	20,20%	14,80%	21,30%	14,80%	49,80%
Silt (0,02-0,002 mm)	27,30%	26,80%	27,30%	24,70%	27,30%	24,90%	27,30%	14,80%
Clay (<0,002 mm)	58,00%	54,50%	58,00%	55,20%	58,00%	55,20%	58,00%	35,50%
Textural class	G	G	G	G	G	G	G	PGI
Chemical properties								
pH in H_2O	7,49	7,74	7,49	7,65	7,49	7,70	7,49	7,70
PH in KCl	6,29	6,87	6,29	6,89	6,29	6,87	6,29	6,81
$CaCO_3$ (%)	1,74	0,82	1,74	1,44	1,74	2,26	1,74	0,82
Humus (%)	5,33	4,22	5,33	4,44	5,33	4,12	5,33	9,73
P_2O_5 (mg/100 g soil)	10,44	3,00	10,44	10,71	10,44	25,35	10,44	4,29
K_2O (mg/100 g soil)	62,91	53,66	62,91	66,66	62,91	77,13	62,91	50,02

Comparing initial data with ending - final one after the application variant (19 months) there was a positive change in the soil only in the variant with sludge application, because the soil on this variant of the heavy clays crossed the sandy clay. In other variants there has been no change in texture of soil, even in the variant with manure applications, and these changes are minor and of no importance.

In terms of agrochemical changes of soil properties there were no relevant features except for a significant increase of humus in the variant of application of waste sludge. Humus content of this variant has increased from 5,33% to 9,73% and humus content of this variant has increased by 4,40%. It is interesting to note that in the variant with farmyard manure aplication, there has been no increase in humus. This means that carboniferous component of sludge strongly affects the increase of humus in the soil, due to the specific properties of coal as organic matter. It is well known property of the coal that is prone to oxidation, or better said, autoxidation, which occurs due sorption oxygen from the air. This process is in fact the oxidation-hydrolitic decomposition of coal. Under the influence of oxygen there are different chemical processes, especially oxidation of lignin as one of the basic ingredients of organic substances in coal. Further chemical process leads to the formation of humic acids in this way, and accumulation of humus in the soil [7].

Thus in the content of humus should be sought the basic value of carboniferous sludge.

Content of heavy metals and other pollutants

These studies were done in order to determine the environmental acceptability of carboniferous sludge in the case of its wider application and use on agricultural soils. If this substance, among other things, contained toxic elements like heavy metals entering the food chain, in this case it would be useless, regardless of his best agrochemical properties in agriculture. Therefore, the analysis selected five most important elements, relevant in food chain and with significant impact on the environment (As, Zn, Pb, Cd and Ni).

Results of analyzes of content of these elements, both in soil and sludge from the ecological aspect very encouraging. Almost all the analytical values obtained are not high to be able to talk about potential contamination with these elements, especially if they are in the form of carbonates, which was virtually unsoluble in water. The analytical content of these elements is shown in Table 2.

Table 2. Analysis of hevay metals

ANALYSED MEDIA	As mg/kg	Zn mg/kg	Pb mg/kg	Cd mg/kg	Ni mg/kg
Coal Sludge	21	53	8	<0,1	66,0
The soil in the experiment	30	92	27	<0,1	67,0
M.P.S.	30	300	150	2	60
Salt (Coal sludge / Soil)	70/100	18/31	5/18	<5/<5	110/112

The degree of pollution or contamination (So) is the ratio between the determined and the maximum acceptable concentration multiplied by 100 Thus (So) in our case is as follows: [1].

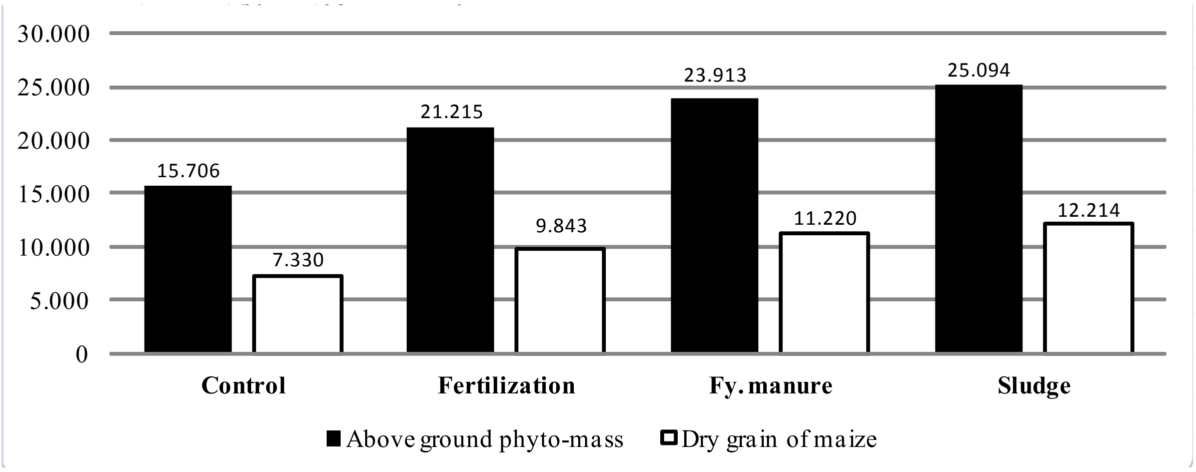
Chemical symbol	Sludge (%)	Soil (%)
As	70	100
Zn	18	31
Pb	5	18
Cd	<5	<5
Ni	110	112

If So <100, then the contamination is small and getting smaller if the salt is less than 100.

The results of the exact field trials

Research using the exact field experiment tested the performance evaluation of individual treatment on the positive physical and chemical properties of soil, obtained by laboratory analysis.

The most important and relevant indicator of effects of different treatments on test crop, were obtained in two indicators: above ground phyto-mass, particularly yield of dry grain. In our experiment, we take maize as test crop and investigated the effect of treatments on the yield of dry grain of maize (Zea mays). Yields of this crop have been translated into two basic quantification; total above ground mass and yield of dry grain, whose numerical values can be compared between different variants of the experiment. The yields of these two features are shown in Graph. 1.



Graph 1. Average yield of above ground phyto-mass and yield of dry grain of maize (kg/ha)

Yields for both features have the following descending order: Sludge > Fy. manure > Fertilization > Control, respectively. This means that the option of treatment with carboniferous sludge gave the best results of above ground phyto mass and yield of clean dry grain of maize.

Biometric evaluation of results - yields of maize

Assessment of the significance of differences for the two variants of compared quantities: dry grain and total above ground phyto-mass was tested using “t” test. The results are shown in Table 3.

Table 3. Differences between grain and above ground phyto – mass of maize

Compared variants	For pure grain				For above ground phyto-mass			
	d	Sd	t	p%	d	Sd	t	p%
Control - Fertilization	-2.513	904	2.778	2,60*	-4.509	1.293	3,487	1,13*
Control - F.Y. manure	-3.890	799	4.869	0,19**	-7.207	1.263	5,756	0,10***
Control - Sludge	-4.884	818	5.971	<0,10***	-8.388	1.531	5,478	0,12***
Fertilization - Fy. Manure	-1.377	899	1.532	$17,7^{O}$	-2.698	837	3,223	1,50*
Fertilization - Sludge	-2.372	915	2.591	3,5*	-3.094	1.204	2,569	4,10*
F.Y. Manure - Sludge	-994	812	1.224	$27,0^{O}$	-1.181	1.172	1,008	$35,0^{O}$

EXPLANATION OF SYMBOLS:

d = difference between the average absolute values of the compared variants

Sd - average error difference, which is $\sqrt{S\overline{x}_1^{\ 2} - S\overline{x}_2^{\ 2}}$

Sx = average error of mean values $S_X = \frac{\sigma}{\sqrt{n}}$

„T" = $\frac{d}{Sd}$

p% = test from the appropriate table for the calculated "t"

t_{tab} – to 0,05=2,365; za 0,01=3,449; to 0,001=5,405 (for 7 degrees of freedom)

*Statistically significant difference

**statistically highly significant difference

***very highly statistically significant difference

ºno statistical significant difference

CONCLUSIONS

Conducted research of maize (Zea mays) as test crop, using exact field trial experiment, indisputably established that this crop had best responded to treatment with application of waste sludge, compared to the other two trial variants, where the conventional mineral and organic fertilizers (NPK and farmyard manure) were applied. This was reflected in the yields of both; above ground phyto-mass, and dry grain of maize.

On the other hand, the analytical data of laboratory research on the content of physiologically active (plant available) nutrients (P_2O_5 and K_2O), are indicating that carboniferous waste sludge) is very poor in these nutrients as fertilizer according to this chemical feature it has no value.

Practically, this means that all the value of waste sludge as fertilizers was quite high content of various humic substances in soil and stimulating metabolic effect as well as physical properties of soil – hydrotermical at the first place.

By visual observation, their creation, and the research, waste coal sludge has the properties of coal, which is rich in humic substances (humic acid, lignin and humin). Many researches confirm stimulated cumulative effect of these substances on plant growth and development [7, 8, 15, 16]. According to this, carboniferous mass, should be evaluated as a provider of continuing humus and plant growth stimulators.

Humic substances affecting the adoption of increased CO_2, ATP synthesis increased respiratory mitochondrial function and thus generally in photosynthesis. Studies indicate a positive effect humic acids which are closely linked and fulvic acids [15].

Given the positive physical and especially chemical characteristics of the particular waste carboniferous sludge from the lake Modrac and using here highlighted achievements in the world, this substrate could represent “the black gold of agriculture.“

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