

# Optimisation of potassium nitro humate synthesis from lignite

Benjamin Catovic<sup>1</sup>, Almir Sestan<sup>1</sup>, Aldina Kesic<sup>1</sup>, Majda Srabovic<sup>1</sup>, Melita Huremovic<sup>1</sup>

<sup>1</sup>(Department of Chemistry, University of Tuzla, Bosnia and Herzegovina)

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**ABSTRACT :** It is known that all types of coal can be oxidized, with the fact that dark coals are the easiest to oxidize, especially lignite. The oxidation process enriches the coal with humic acids. If oxidized lignite is then treated with a base solution (KOH, NaOH), humates are formed, products similar to humus, which can be used in production of organo-mineral fertilizers. In the case of intensive agricultural production, it is necessary to add such organic fertilizers to the land in addition to mineral fertilizers. Lignite, which is used in the experiment was excavated from the roof mine layers and fragmented to the granulation size of 0-15 mm. Lignite is further sieved on the granulation size of 0-5 mm and pre-dried in an oven at 105°C. Using the process of neutralization of oxidized lignite using KOH, under different neutralization conditions, potassium nitro humate was synthesized, which was confirmed by the FTIR method. In all samples of potassium nitro humate the content of humic acids, K<sub>2</sub>O and total nitrogen were determined. Based on the obtained results the optimal synthesis conditions were determined depending on the concentration of KOH, the volume of the KOH solution used, as well as the duration of oxidized lignite neutralization.

**KEYWORDS** – lignite, humic acid, humate, biogenic elements, mineral fertilizers, organic fertilizers

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## I. INTRODUCTION

Previous research has shown that both biogenic elements, on the one hand, and humic substances, on the other hand, are necessary for the normal growth and development of plants. In addition, the large reserves of lignite in Bosnia and Herzegovina represent a significant raw material base for the production of humic fertilizers [1]. These facts raise the question of the possibility of producing organo-mineral fertilizers, i.e. incorporating biogenic elements into humic substances isolated from lignite coal. Research on the lignite of the Tuzla basin, which was carried out with the aim of examining the possibility of its application in agriculture, showed that this lignite, thanks to its favorable physical and chemical properties, can be considered a potential raw material for the production of humic fertilizers.

The microstructure of lignite coal and its chemical properties, the ability to bind other organic and various inorganic compounds in ionic and molecular form, with physical and chemical forces of varying strength, make lignite coal a very favorable raw material for the production of organo-mineral fertilizer [2]. The main plant nutrients (N, P and K) must be bound in a form that will enable their uptake by plants, but also prevent easy leaching or harmful changes on plants and in the soil. In this way, it is possible to obtain marketable products, i.e. complex fertilizers, which meet the current requirements in the production of the so-called healthy food, preservation and protection of the quality of all environmental components, but the most important thing is that with the use of such fertilizers there is a constant improvement of the qualitative and productive properties of the land [3].

Humic acids are associated with the mineral fraction of the soil, forming colloidal humus-clay and humus-silt complexes. These aggregates increase the cohesive forces that cause the attraction of clay components and very fine soil particles [4]. In this way, the physiological function of humic acids is manifested in their influence on the oxidative exchange and enhancement of the energy potential of the plant.

Lignite cannot be considered as fertilizer in the true sense of the word, but it can serve as a source of organic carbon and have a positive impact not only for the transport of nutrients but also, as already stated, the transport of risky elements by immobilizing them in the humic (or organic) complex.

Given that the intake of the main plant nutrients is mediated by humic substances, it is the growth of plants directly and indirectly under the influence of humic substances. It was shown that there is a positive correlation between the content of humus in the soil and the yield of plants. The increased intake of the main plant nutrients: N, P and K acts as a stimulating effect of humic substances on plant growth. In addition, when adequate humic substances are present in the soil, the requirement for the application of mineral fertilizers is reduced [5,6].

It is necessary to reduce the fertilizer requirements and keep the fertilizer ingredients in the plant root zone by applying humate-based fertilizers. The use of either dry or liquid humic substances in soils greatly increases the efficiency of fertilizers. The uptake of Ca and Mg increases when plants are irrigated with liquid suspensions of humic and fulvic acids [7].

According to Hristeva, humic acids, regardless of their origin, are physiologically active in the ion-dispersed state and are included in the general metabolism. Penetrating the plant cell with their polyphenolic and quinoid groups, they have a direct stimulating effect on accelerating the regeneration of nucleoside-di-phosphate into nucleoside-tri-phosphate, thus influencing the speed of synthesis of the entire DNA-RNA system. In this way, the physiological function of humic acids is manifested in their influence on the oxidative exchange and enhancement of the energy potential of the plant organism [8].

Natural humic substances from low-calorie lignite are superior ingredients of fertilizers. The best sources of humic acids for fertilizer application are lignite - highly oxidized, low-calorie, which contain a relatively high concentration of small molecular units (fulvic acid). The smaller the molecules of humic and fulvic acids, the greater the fertilizing value and the plants take up trace minerals more easily [9].

The results indicate that lignite really contributes to the controlled transport of plant nutrients, most likely by the formation of organo-mineral complexes with a slow release of nutrients compared to their form in the soil solution. The positive influence of lignite on plant yield is statistically evident even in quality soils, so reaching the proportion of NPK fertilizers can be replaced by the use of cheaper lignite. [10]

The structure of humic acid, shown in Figure 1., contains three phenolic OH groups, quinone structure, nitrogen and oxygen as bridge units and COOH groups differently distributed on the aromatic rings [11].

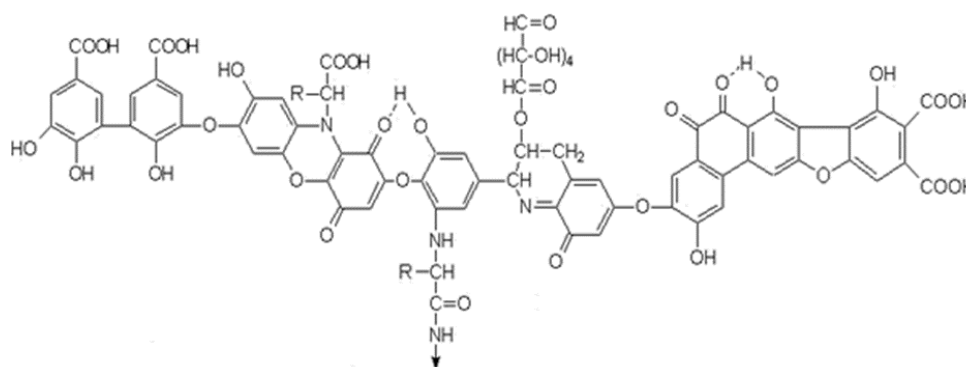


Figure 1. Model of humic acid [12]

It is important to point out that the oxidation of coal produces humic acids, i.e. the coal is enriched with humic acids. Coal is most often oxidized with a nitric acid solution, whereby oxidized lignite enriched with humic acids is obtained. However, if oxidized lignite is treated with a base solution (e.g. NaOH, KOH, NH<sub>4</sub>OH), i.e. neutralized, humates, products similar to humus, are formed, which can be used in agriculture for the production of organo-mineral fertilizers. A particularly beneficial effect is achieved by using these fertilizers on soils that are poor in humic substances.

In addition to potassium-nitrohumate, potassium nitrate, KNO<sub>3</sub>, is also produced as a byproduct in the neutralization reaction of oxidized lignite, which has been used as an ingredient in fertilizers for a very long time. Fertilizers that contain KNO<sub>3</sub> are known as "mixed fertilizers" and it is usual to enrich them with other nutrients in order to meet all the conditions necessary for the proper growth and development of plants.

## II. EXPERIMENTAL

### 2.1. Materials and methode

In this research, lignite from the surface mine "Šikulje" was used, which is located in the lignite mine "Kreka", Bosnia and Herzegovina. The lignite used in the experiment was taken from the roof layer of the excavation and crushed to a granulation size of 0-15 mm. For the purposes of the experiment, the lignite was additionally sieved to a granulation size of 0-5 mm, and to achieve the best results, it was previously dried in an oven at a temperature of 105°C. The parameters of oxidized lignite neutralization using KOH as a neutralizing agent during the process of synthesis of potassium nitrohumate were examined, with special reference to the content of humic acids, potassium and nitrogen in the synthesized potassium nitrohumate [13]. The tested parameters are:

- a) influence of KOH concentration
- b) the influence of the duration of neutralization and

c) influence of the volume of the KOH solution

In all samples of potassium nitrohumate, potassium content was determined using the standard ICP-OES method and nitrogen content using the Kjeldahl method. In addition, in all tested samples, the content of humic acids was determined using the acetate method. Examination of raw lignite included: technical analysis, chemical analysis of ash, determination of humic acid content and elemental analysis (Table 1.).

Table 1. Chemical analysis of the investigated lignite

Proximate analysis, %									
Moisture	Ash	Combustible substances	Volatile matter	C <sub>fix</sub>	Coke	Total sulfur			
49,13	10,19	40,68	22,10	18,58	28,77	0,51			
Elemental analysis, % m/m									
C	H		S	N					
38,75	6,07		0,93	0,57					
Chemical analysis, % m/m									
Organic matter	pH (H <sub>2</sub> O)	pH (KCl)	EC (mS/cm)	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>
57,26	5,27	4,55	2,87	52,00	9,40	20,20	7,05	4,25	5,66

### III. RESULTS AND DISCUSSION

#### 3.1. Enrichment of raw lignite with humic acids by the oxidation with HNO<sub>3</sub>

The content of humic acids in coals varies. Based on the works of numerous researchers, it was determined that most coals contain a small amount of humic acids, even below 10%. Raw lignite can be enriched with humic acids by an oxidation process, such as air exposure, treatment with acidic oxidizing agents (e.g. HNO<sub>3</sub>) or treatment with some oxidizing agents in a basic environment (e.g. air, hydrogen peroxide, potassium permanganate, etc.). The advantage of lignite oxidation using nitric acid is that, in addition to enrichment with humic acids, there is also an increase in nitrogen content. Our research shows that the investigated lignite contains 17.5% of total humic acids. In order to increase the content of humic acids, lignite samples were oxidized using 125 mL of 20% HNO<sub>3</sub> for 24 hours at room temperature. After the end of the reaction, the resulting product was evaporated in a water bath and then dried in an oven at 105 °C. The product was analyzed for the content of humic substances fractions, and the results are shown in Figure 2.

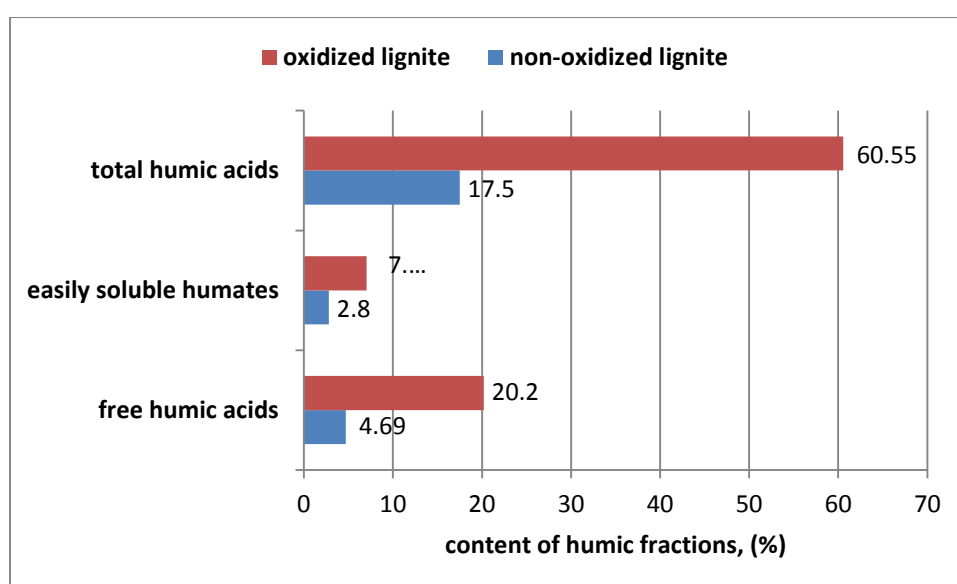


Figure 2. Content of humic substances fractions

An increase in the content of all fractions of humic substances can be observed. The content of total humic acids in oxidized lignite is 60.55%, which is 245% more than in untreated lignite (17.5%). There are similar relationships with the remaining two fractions of humic substances. The content of free humic acids in oxidized lignite is 20.2%, which is 330% more than untreated lignite (4.69%). There is also a significant increase in the content of easily soluble humates, which is 246% more in oxidized lignite (7.03%) than in untreated lignite (2.03%). In Figure 3. the results of determining the structure by the FTIR method, i.e. the content of functional groups in untreated (parent substrate) lignite and lignite treated with 20% HNO<sub>3</sub> are presented.

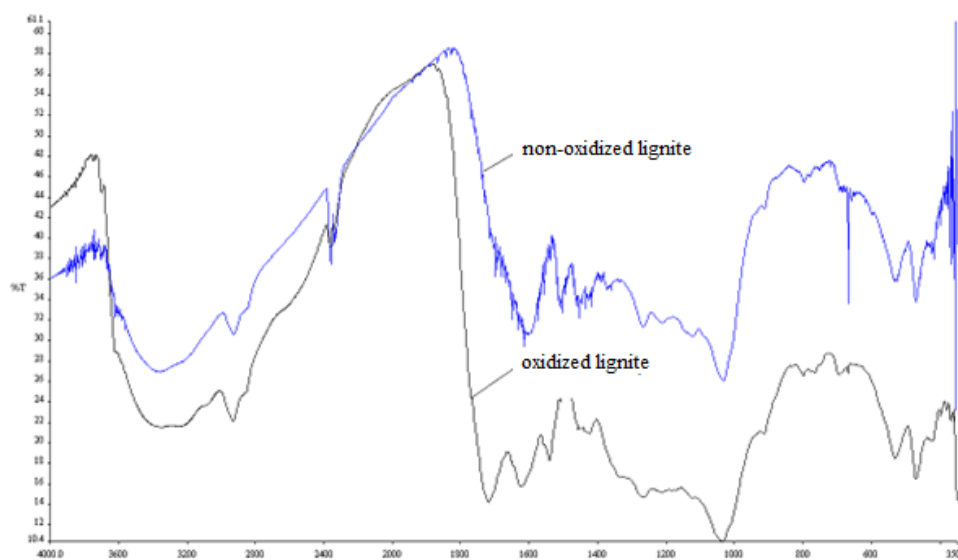


Figure 3. FTIR spectra of non-oxidized and oxidized lignite

The FTIR spectrum of the used lignite (Figure 3) shows a typical infrared characterization of organic matter in younger coals, which is confirmed by literature data, including aliphatic C-H valence bands at 2924 cm<sup>-1</sup> and 2856 cm<sup>-1</sup>, C=C or C=O valence bands. aromatic ring at 1610 cm<sup>-1</sup> and 1505 cm<sup>-1</sup>, as well as aliphatic C-H valence band at 1455 cm<sup>-1</sup>, 1370 cm<sup>-1</sup> and 822 cm<sup>-1</sup>[15,16].

In order to obtain potassium nitrohumate, oxidized lignite is subjected to a neutralization process. Neutralization of oxidized lignite was carried out with KOH solutions of different concentrations (0.1; 0.2; 0.3; 0.4; 0.5; 0.7; 1.0 mol/L) at a temperature of 20°C, during of 24 h at a volume of KOH solution of 125 mL. When obtaining potassium nitrohumate, both the content of humic acids and the content of biogenic elements, potassium and nitrogen, are important. Five solutions of potassium nitrohumate were obtained, which were evaporated to dryness in a water bath, and then dried in an oven at 105°C. The content of humic acids, potassium oxide and nitrogen was determined in the obtained potassium nitrohumate products.

Wafaa at all. results indicated increases of OM and CEC under effect of either bentonite or potassium humate; no significant effect was obtained among rates of nitrogen. Results showed positive responses of available N, P and K compared to control treatments. Also, results revealed significant positive responses of total yield (straw and grains) along with total content of nutrient elements. From the present study, soil conditioner (combination of bentonite and potassium humate) was favorable for soil parameters of chemical soil properties of sandy soil which reflects on increase soil fertility [17].

### 3.2. Determination of optimal conditions for the synthesis of potassium nitrohumate based on lignite coal

#### 3.2.1. Examination of the influence of KOH concentration on the content of total humic acids, nitrogen and K<sub>2</sub>O

The influence of the concentration of KOH on the content of the investigated fractions was examined in such a way that the concentration of potassium hydroxide was changed, while the other neutralization parameters were kept constant. The results of determining the content of total humic acids in potassium nitrohumate samples (Figure 4) show that the most humic acids (32.38%) were isolated from the samples treated with 1M KOH solution, and the least humic acids (20.83%) contain samples treated with 0.1 M KOH. Based on these results, it can be concluded that with an increase in the concentration of potassium hydroxide, there is a slight increase in the concentration of humic acids in potassium nitrohumate. The nitrogen content in the examined samples of potassium nitro-humate ranges from 1.14% to 3.28%. The highest concentration of nitrogen (3.28%) was recorded in the sample obtained by neutralizing oxidized lignite with a 0.3 M KOH solution.

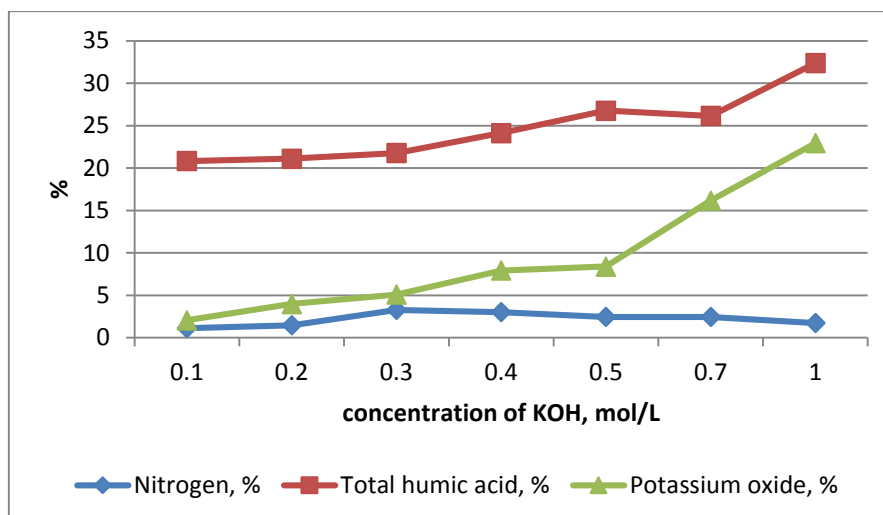


Figure 4. The influence of KOH concentration on the content of humic acids, nitrogen and potassium oxide in potassium nitrohumate

In addition, for the KOH solution concentration interval (0.4-1.0 mol/L) there is an inverse proportionality between the nitrogen content in potassium nitrohumate and the KOH concentration. With the increase in the concentration of KOH, there is an increase in the content of potassium oxide in potassium nitrohumate, so that at a concentration of KOH of 1.0 mol/L it reaches a maximum (22,95%). These results are in line with similar research conducted on lignite from the Kosovo mining basin [6].

The results of testing the influence of KOH concentration on the neutralization of oxidized lignite showed that the best neutralization effects, in terms of  $K_2O$  content, are achieved with the use of 1 M KOH. However, at this concentration of KOH, the  $N_2$  content is quite low (1.74%), so for further research, 0.7M KOH was taken, at which the content of total humic acids is near to the maximum. At this concentration of KOH, the optimal contents of the examined components were achieved. The results representing the minimum and maximum values are excluded from consideration, due to the reduction of the percentage of error in the analysis of such samples.

In Figures 5. and Figure 6. the results of the FTIR spectra of potassium nitrohumate as a function of different concentration of KOH, are presented. The analysis was performed for the purpose of identifying the resulting product. According to literature data, a positive match was established in the structure of the resulting potassium nitrohumate [18].

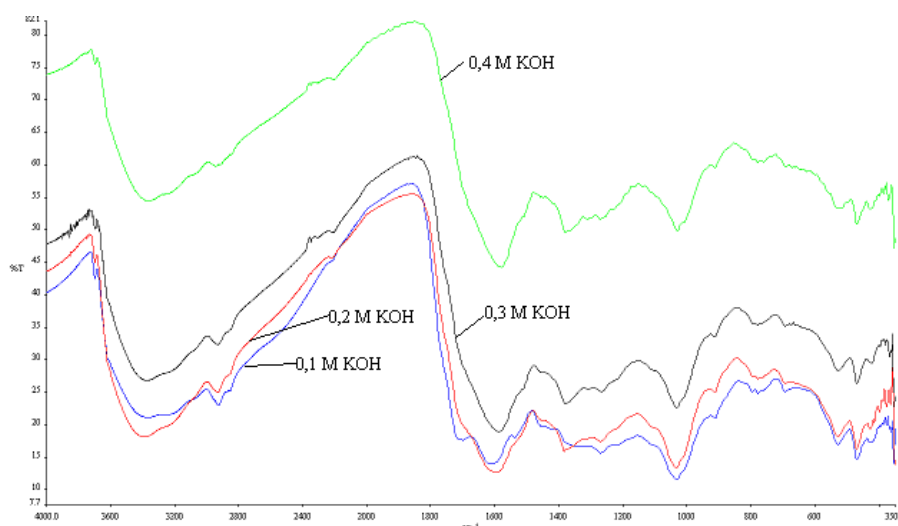


Figure 5. FTIR spectra of potassium nitrohumate as a function of different concentration of KOH (0.1-0.4 M KOH)

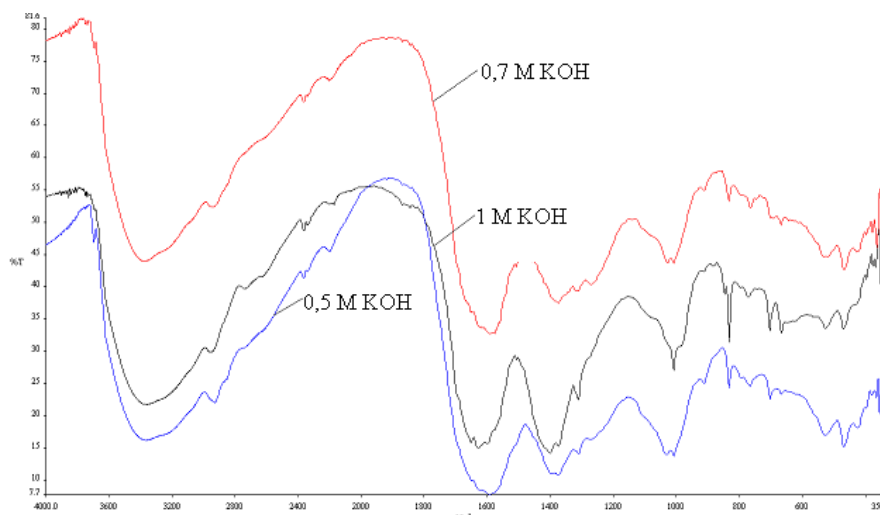


Figure 6. FTIR spectra of potassium nitrohumate as a function of different concentration of KOH (0,5-1 M KOH)

### 3.2.2. Examination of the influence of the duration of neutralization on the content of total humic acids, nitrogen and K<sub>2</sub>O

The investigation of the influence of the duration of the reaction on the neutralization of oxidized lignite was carried out in such a way that in five samples, each with 10 g of oxidized lignite, the neutralization was time-limited to 3, 6, 12, 16 and 24 hours, and the other parameters were kept constant (concentration KOH - 0.7 mol/L; volume of KOH solution - 125mL and temperature 20°C). Five solutions of potassium nitrohumate were obtained, which were evaporated to dryness in a water bath, and then dried in an oven at 105°C. The content of humic acids, potassium oxide and nitrogen in the obtained potassium nitrohumate products was determined. The results of the determination are shown in Figure 7. show that the content of humic acids is the highest in samples with a neutralization time of 24h (27.58%) and the lowest in samples with a neutralization time of 3h (19.81%).

At different durations of neutralization of oxidized lignite, potassium nitrohumate products with different contents of biogenic elements, potassium and nitrogen were obtained. The highest content of potassium oxide (36.76%) is found in samples of potassium nitrohumate where the neutralization process lasted 24 hours, and the lowest content of potassium oxide was recorded in samples where the neutralization process lasted 6 hours (30.47%). It can be generally concluded that with the increase in the duration of the neutralization process, there is an increase in the content of humic acids and potassium oxide.

In the case of nitrogen content, the situation is somewhat different. Namely, the maximum content was recorded in samples where neutralization lasted 16 hours (2.47%). With the increase in the duration of neutralization to 24 hours, the nitrogen content decreases slightly (2.42%), which represents a negligible difference, while the lowest value has the samples where the neutralization lasted 3 hours (1.45%).

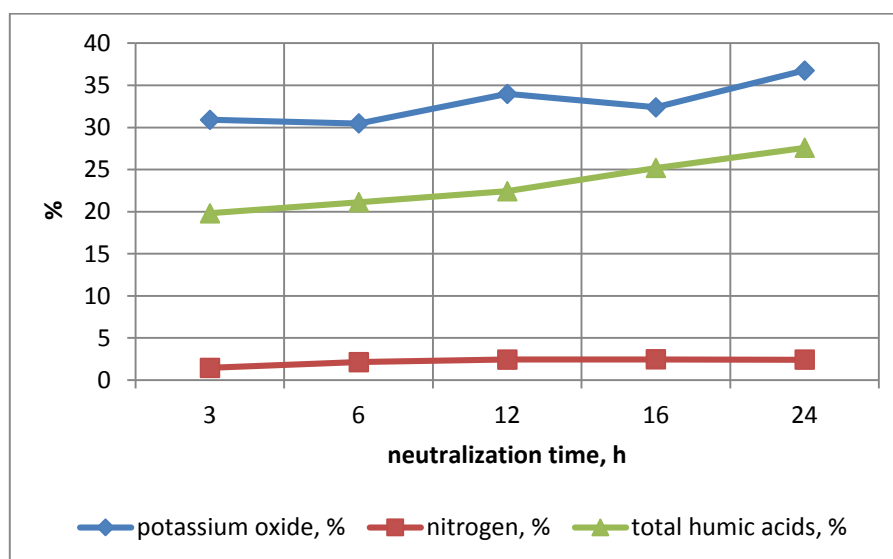


Figure 7. The influence of the duration of neutralization on the yield of humic acids, nitrogen and potassium oxide in potassium nitrohumate

Examining the duration of the neutralization process of oxidized lignite, it was concluded that the maximum content of humic acids and potassium oxide is obtained during neutralization in a period of 24 hours, while the maximum concentration of nitrogen is obtained during the neutralization period of 16 hours. Given that the nitrogen content during 24-hour neutralization is slightly lower than the maximum content during 16-hour neutralization (only 2.02% lower value), it can be considered that the optimal duration of neutralization is 24 hours. For this reason, further investigation of lignite oxidation as a function of different volumes of 0.7M KOH solution was followed with a neutralization time lasting 24 hours.

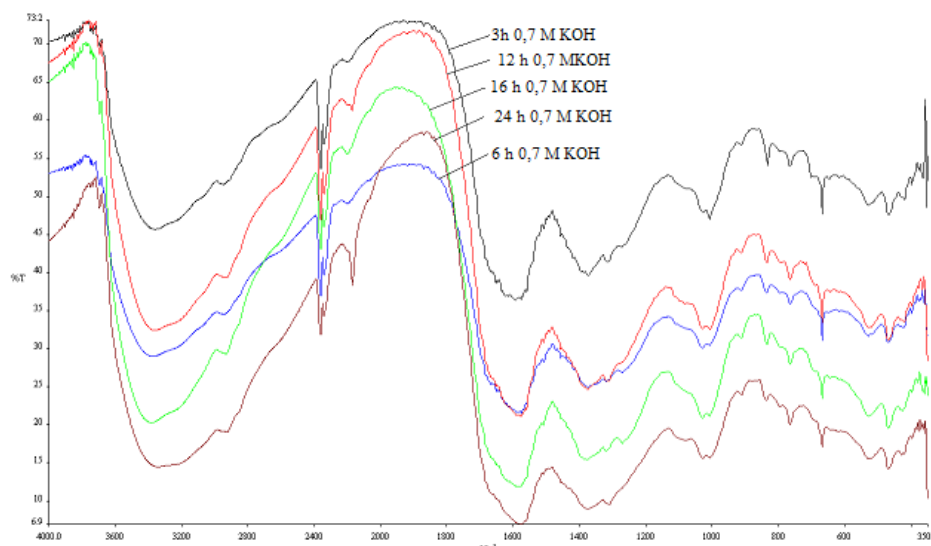


Figure 8. FTIR spectrum of potassium nitrohumate as a function of different neutralization time (3-24h)

### 3.2.3. Examination of the influence of KOH volume on the content of total humic acids, nitrogen and K<sub>2</sub>O

The influence of the volume of the KOH solution on the neutralization of oxidized lignite was examined in such a way that in five samples, with 10 g of oxidized lignite each, the volumes of KOH were changed (50, 100, 150, 200 and 300 mL) and the other parameters were kept constant (concentration of KOH - 0.7 mol/L; volume of KOH solution of 125 mL and temperature 20°C). In this way, seven solutions of potassium nitrohumate were obtained, which were evaporated to dryness in a water bath, and then dried in an oven at 105°C. The content of humic acids, biogenic elements, potassium and nitrogen in the potassium nitrohumate products obtained was determined. The results of these tests (Figure 9) show that the change in the volume of the KOH solution affects the content of humic acids, as well as the content of biogenic elements potassium and nitrogen.

Examination of the influence of the volume of KOH solution on the content of humic acids in potassium-nitro-humate showed that the content of humic acids ranges from 16.63% to 25.55% and that it is inversely proportional to the increase in the volume of 0.7 M KOH. The most humic acids were obtained at a KOH volume of 50 mL (25.55%) and the least at a KOH volume of 300 mL. The possible cause of this phenomenon may be that with a larger volume of KOH, intermediate reactions took place in which, at a KOH volume of 50 mL, the stoichiometric relationship between the acid residues of HNO<sub>3</sub> oxidized lignite and the KOH solution used in the neutralization process was satisfied.

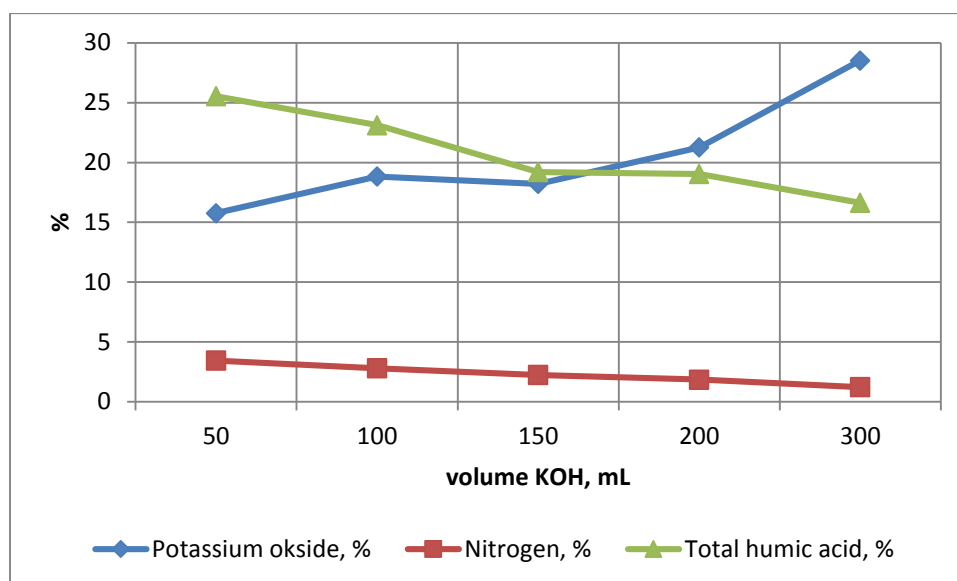


Figure 9. Effect of KOH volume on the yield of humic acids, nitrogen and potassium oxide in potassium nitrohumate

The potassium content in potassium-nitro-humate products ranges from 15.76% at a volume of 50 mL to 28.53% at a KOH volume of 300 mL. It can be observed that with the increase in the volume of KOH comes an increase in the potassium content, where a part of the detected potassium comes from  $KNO_3$ , which is formed as a byproduct of the lignite oxidation reaction with nitric acid. A similar situation as with the content of total humic acids is also with the nitrogen content, which ranges from 1.22% to 3.44%, where the highest nitrogen content was recorded at a KOH volume of 50 mL (3.44%) and the lowest at volume of KOH of 300 mL (1.22%).

According to the results of the analysis of the influence of the volume of KOH (Figure 9.), it can be concluded that the volume of 100 mL of 0.7 M KOH solution is optimal for the content of humic acids (which is only 9.47% lower than with a volume of 50 mL). Also, the volume of 100 mL of 0.7M KOH can be considered optimal for the potassium content (if we exclude the maximum values from consideration to eliminate the error) and the nitrogen content (by 22.4% less than the nitrogen content at the KOH volume of 50 mL).

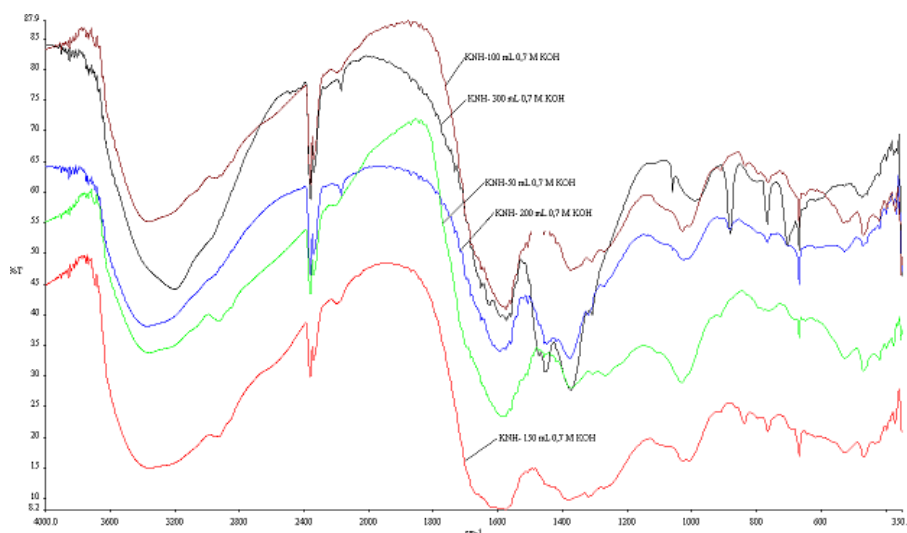


Figure 10. FTIR spectrum of potassium nitrohumate as a function of different KOH volume (50-100 mL 0,7M KOH)

#### IV. CONCLUSION

The content of total humic acids in oxidized lignite is 60.55%, which is 245% more than in untreated lignite (17.5%). The content of free humic acids in oxidized lignite is 20.2%, which is 330% more than untreated lignite (4.69%). There is also a significant increase in the content of easily soluble humates, which is 246% more in oxidized lignite (7.03%) than in untreated lignite (2.03%).



The FTIR spectrum of the used lignite shows the typical infrared characterization of organic matter in younger coals, including aliphatic C-H valence bands at 2924 cm<sup>-1</sup> and 2856 cm<sup>-1</sup>, C=C or C=O valence bands of the aromatic ring at 1610 cm<sup>-1</sup> and 1505 cm<sup>-1</sup>, as well as aliphatic C-H valence bands at 1455 cm<sup>-1</sup>, 1370 cm<sup>-1</sup> and 822 cm<sup>-1</sup>.

The results of testing the influence of KOH concentration on the neutralization of oxidized lignite showed that the best effects, in terms of K<sub>2</sub>O content, are achieved with the use of 1 M KOH. However, at this concentration of KOH, the N<sub>2</sub> content is quite low (1.74%), so for further research, 0.7M KOH was taken, at which the content of total humic acids is close to the maximum. At this concentration of KOH, the optimal contents of the examined components were achieved.

Examining the duration of the neutralization process of oxidized lignite, it was concluded that the maximum content of humic acids and potassium oxide is obtained during neutralization in a period of 24 hours, while the maximum concentration of nitrogen is obtained during the neutralization period of 16 hours. Given that the nitrogen content during 24-hour neutralization is slightly lower than the maximum content during 16-hour neutralization (only 2.02% lower value), it can be considered that the optimal duration of neutralization is 24 hours.

According to the results of the analysis of the influence of the volume of KOH, it can be concluded that the volume of 100 mL of 0.7 M KOH solution is optimal for the content of humic acids (which is only 9.47% lower than with a volume of 50 mL). Also, the volume of 100 mL of 0.7M KOH can be considered optimal for the potassium content (if we exclude the maximum values from consideration to eliminate the error) and the nitrogen content (by 22.4% less than the nitrogen content at the KOH volume of 50 mL).

Based on everything that has been said and in accordance with the specific requirements for the content of individual researched elements, a different arrangement of the researched parameters can be taken.

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