

BROWN COAL OXIDATION BY NITRIC ACID WITH THE NITROGEN–HUMIC FERTILIZERS PRODUCTION

Shayakhmet Moldabekov

South Kazakhstan state university named after M.Auezov
Chemical-technological faculty
160012 Kazakhstan, Shymkent city,
Ryskulbekov street 3a, flat #9
moldabekov39@mail.ru
+77017825447

Kurmanbek Zhantasov

South Kazakhstan state university named after M.Auezov
Chemical-technological faculty
160008 Kazakhstan, Shymkent city, Zhylykshiev street 11,
k_zhantasov@mail.ru
+77017825447

Orazaly Balabekov

South-Kazakhstan State pedagogic institute
160000 Kazakhstan, Shymkent city,
Akademgorodok street 35
k_zhantasov@mail.ru, +77017825447

Ongarkul Koblanova

South-Kazakhstan State pedagogic institute
160008 Kazakhstan, Shymkent city, Kazbek-bi street 32a, flat#13
k_zhantasov@mail.ru, +77017825447

Marina Yeskendirova

South Kazakhstan state university named after M.Auezov
Chemical-technological faculty
160011 Kazakhstan, Shymkent city, Adyrbekov street 63
yesmm12@mail.ru, +77017587814

Dina Zhantasova

South Kazakhstan state university named after M.Auezov
160008 Kazakhstan, Shymkent city, Zhylykshiev street 11,
k_zhantasov@mail.ru, +77017825447

Kamshat Bazhirova

South Kazakhstan state university named after M.Auezov
Chemical-technological faculty

Manap Zhantasov

South Kazakhstan state university named after M.Auezov
Mechanical-building faculty
160008 Kazakhstan, Shymkent city, Zhylykshiev street 110,
manapjan_80@mail.ru, +77017562487

Abstract

Data on oxidation of brown coal by nitric acid and production nitrogen-humic fertilizers are given at various temperatures with extraction of humic acids. According to the suggested method humates are produced by hyperfine dispersion of coals containing humic acids in the presence of a chemical additive such as sodium carbonate or potassium carbonate.

Key words: brown coal nitric acid, oxidation, mixing, temperature, concentration, liquid, liquid product, loose product, yield of humic acids.

1 Introduction

The way of mineral fertilizers manufacture from solid combustible minerals [18] allows to receive the high quality humates for one stage with the high degree of humic acids extraction from coals. According to the suggested method humates are produced by hyperfine dispersion of coals containing humic acids in the presence of a chemical additive such as sodium carbonate or potassium carbonate.

2 The primary research tasks

For the purpose of simplification of a humic plant growth stimulant production process and the deep transformation of an organic part of a brown coal into water-soluble products, the brown coal is processed by the concentrated solution of alkaline metal hydroxide at a higher temperature [19]. But the maximum efficiency of the humic fertilizers is shown at combination with phosphorus fertilizers.

3 Experimental part

The most perspective way – is the ordinary brown coal oxidation in the industrial conditions, and the most accessible oxidizer is the nitric acid. This leads to the resource base increase for the coal-humic compounds production.

The brown coal oxidation by nitric acid was performed in a vessel with a mixer at 30-60⁰C. Nitric acid amount and concentration influence on the humic acids yield was studied in the first set of experiments. The humic acids were extracted from the distilled water washed oxidized coal by the 1% alkali water solution followed by the 10% hydrochloric acid precipitation at pH 6,1–6,2.

The experiments have shown (Fig.1) that nitric acid norm and concentration increase provides for the humic acid yield increase (% for the coal dry mass).

But there is a limit for any initial acid concentration when the dose increase of acid added to the coal doesn't provide for the humic acids yield increase. The addition of 500 kg (mng) HNO₃ in the form of the 30% acid solution for 1 ton of coal the humic compounds yield is equal to 30% or 300 kg for 1 ton of the dry coal. If we added the same acid amount but as the 40% acid solution to the coal the humic compounds yield increased up to 33%. If we added the same amount of the nitric acid of the 56% concentration the humic compounds yield was increased up to 50%.

The product may be liquid, solid, or pulp depending on the added nitric acid dose and concentration. The dotted curve line is denoting the conventional border line between the liquid and dry product (Fig.1). In order to produce the loose product the optimal dose of the added nitric acid is in the intersection of the dotted curve line and the curve, corresponding to the acid concentration.

Fig.2 illustrates the humic acid yield at different ratio of the coal and nitric acid for acids with the different concentration (solid phase ratio to the liquid phase – S:L). It is evident from the figure that if want to have the loose product with the maximal humic acids content we should take the minimal values of S: L and maximal nitric acid concentration.

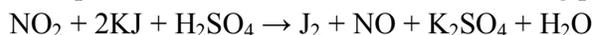
It is shown in the Fig.3 that humic acids yield is 1 g for 1 g of the 56% concentration HNO_3 (in terms of the mono hydrate). If we add the same quantity of the 40% concentration HNO_3 the humic acid yield will be 0,75 g for 1g of the HNO_3 (in terms of the mono hydrate). Humic acids yield coefficient is decreasing with the nitric acid concentration decrease.

The investigation process has testified that the brown coal oxidation is accompanied by the intensive nitrogen oxides liberation into the gas phase. So, it is necessary to provide for the complete utilization of the liberated nitrogen oxides in case the coal humic fertilizers are produced on a regular basis. As a consequence we have managed two problems: 1) the qualitative and quantitative composition of gases which liberate at oxidation of brown coal by the nitric acid was determined; 2) a method of nitrogen oxides utilization out of the gases was developed.

The laboratory setup for recovery and analysis the gases was mounted; it is presented in Fig. 4. The setup contains the three-neck reaction flask 1 with a thermometer and a drop funnel, adsorbing system 4-10, aspirators 2, 11, 17 and decanters 3, 12, 16. The system pressure is measured by the manometer 13.

Coal sample is loaded into the reaction flask and the nitric acid is added from the drop funnel accounting 0,4kg (for mono hydrate) for 1 kg of coal. As far as the acid is added to the coal (the mixture is carefully stirred) the liberated gases (NO_2 , NO , CO_2 and other) are sent to the aspirator filled with the distilled water; a layer of the mineral oil 19 is on the water surface, it prevents the gas mixture from the water solution. The water amount, which was displaced from the aspirator, allows to determine the gas volume which is liberated due to the coal and nitric acid mutual interaction. The liberated gases rate was controlled by the water manometer 13, while keeping the pressure on the definite level. The whole system was blown by nitrogen before the testing.

The gas mixture from the aspirator is given in the first absorbing vessel filled with the 20% acidified solution of potassium iodide; the reaction was taking place in the vessel:



The liberated free iodine was titrated by sodium hyposulphite in the presence of starch. The iodine quantity is equivalent to the NO_2 content in the gas.

Further the gas, purified from the NO_2 was sent to the adsorbing vessels filled with the saturated solution $\text{Ba}(\text{OH})_2$, which interacts with the carbon dioxide. The formed BaCO_3 solution was collected into the vessel 18 in which the hydrochloric acid and the indicator were added from the drop funnel. The liberated carbon dioxide was sent to the aspirator 17 filled with water having the mineral oil film on the surface. The carbon dioxide quantity was estimated according to the quantity of the water flowing out of the aspirator.

Gas, purified from NO_2 and CO_2 was collected into the aspirator 11, where nitrogen oxide (II) NO was oxidized up to nitrogen oxide (IV) NO_2 by the oxygen from the oxygen cylinder 14. Transformation of NO

into NO₂ occurs instantly; as a result the vacuum is formed in the aspirator. In order to keep the regular pressure water was introduced into the aspirator again, earlier it was introduced into the vessel 12.

The residual unabsorbed gas volume was determined after the nitrogen oxides total absorption, the aspirator and vessel 12 contents was mixed and titrated with alkali, then it was be recalculated for NO:



The gas analysis testified that 1 kg of the brown coal oxidation by the 56% nitric acid (the ratio S:L = 1,4, which corresponds to 400 kg of HNO₃ (mng) for 1 ton of the dry coal) produces 30,0 l of gases with the following composition: NO₂ – 5,12%; NO – 41,29 %; CO₂ – 13,80%; N₂ and others – 39, 79 % (mainly nitrogen).

Material balance of the coal oxidation by the nitric acid for the nitrogen at ratio 1:0,4 for the mono hydrate testifies that 39, 15% of the nitric acid transfers to the oxidized coal as nitro compounds and other nitrogen compounds; 36,1% of the nitric acid is reduced, oxidizing organic substances of the coal and transforming into the elementary nitrogen; 2,88 % of the nitric acid nitrogen is transformed into NO₂, and 21,42% - into NO. The latters are to be caught.

The second series of experiments is dedicated to the waste gases utilization process.

The same brown coal was also used as an sorbent for the adsorption of the nitrogen oxides. After nitrogen oxides adsorption the coal is given into the reactor for the final oxidation by the nitric acid and its processing into the standard product.

Such scheme of the gas purification provides for the preliminary coal oxidation by the gaseous nitrogen oxides. It leads to the ratio S:L decrease in the main reactor and finally to the sharp lowering of the nitric acid consumption in the humic fertilizer production.

Angren coal absorbing capacity determination testified that the coal is capable to absorb up to 14-15% of the nitrogen oxides (in terms of nitrogen).

In this case we should oxidize NO into NO₂, because the coal is capable to interact only with the highest nitrogen oxides. Meanwhile the gas which is liberated during the brown coal oxidation by the nitric acid, doesn't contain the oxygen. That is why it is necessary before the nitrogen oxides absorption to add oxygen or air to the gas for the preliminary NO oxidation. Interaction of the gaseous nitrogen oxides with the coal also leads to formation of the humic acids.

4 Results and discussion

Research results are presented in Fig.5. The experiments showed that if the coal interacts with the nitric acid we can have the more intensive humic acids yield in comparison with its interaction with the equivalent nitrogen dioxide amount. In the first case we have 1,2 g of humic acids for 1 g of HNO₃, in the second case – 0,7–0,9 g. In order to have the full adsorption of the nitrogen oxides the coal saturation degree shouldn't exceed 2-3 % in the recalculation for nitrogen. The reaction rate is rather high. The gas purification by coal should be fulfilled in the multishelf apparatus with the fluidized bed, because at purification in the apparatuses with the fixed sorbent layer the coal undesirable local overheat occurs.

At the oxidation of one ton of the Angren coal 30,0 m³ gas is liberated into the gas phase, including 1,54 m³ of NO₂; 12,4 m³ of NO; 4,15 m³ of CO₂; 12,0 m³ of N₂ and others.

Oxidation of 12,4 m³ of NO needs addition of the 8,61 m³ of oxygen (with the oxygen excess equal to 1,3) or 41 m³ of air. Then the exhaust gases entering on the purification will comprise:

$30,0 + 41,0 = 71,0 \text{ m}^3$ for 1 ton of the oxidized coal, including:

NO ₂	1,54 m ³ or 2,19 %
NO	12,4 m ³ or 17,41 %
CO ₂	4,15 m ³ or 5,80 %
O ₂	8,61 m ³ or 12,1 %
N ₂	44,3 m ³ or 62,5 %
Total:	71,0 m ³ – 100 %

It was calculated that the coal amount needed for the adsorption of the nitrogen oxides volume, mentioned above comprises 21 % of the total coal mass, presented for the oxidation. Meanwhile, 0,97 g of humic acids is formed for 1 kg of the nitric acid in the form of the nitrogen oxides.

Humic fertilizers are the most efficient when they are combined with the phosphorus fertilizers. Combined applying of the phosphorus fertilizers with the natural coal, containing the considerable amount of the humic acids, inhibits the phosphorus fixation process, it increases the content of the phosphate mobile forms in the soil and even it transfers the unavailable forms of P₂O₅ in the natural phosphates into the available forms for the plants [1-5]. We decided to examine the behavior of the mixture of the produced nitrogen-humic fertilizer (or nitro-humic compound) combined with the natural phosphorite of the Karatau coalfield.

The Angren coal oxidation was fulfilled by the 56% nitric acid, in the ration 1:1, in the reactor with the stirrer at the room temperature during 1 hour. We produced a product with the following content: humic acids – 55,36 mg-eq/g, COOH – 5,76 mg-eq/g, phenol OH – 6,14 mg-eq/g.

Interaction of nitro hamates and the new compound with the Karatau phosphorite was studied at different initial substances ratio. The samples were placed into a volumetric flask of the 200 ml capacity and 100-120 ml of the distilled water was added. The flasks were shaken on a vibrating table during 6 hours, then the solution volume was diluted with the water up to the mark, stirred and left for 17-18 hours at the room temperature. Then the solutions were filtrated and the amount of the water soluble forms of P₂O₅ (weight method) and CaO (volume method) was checked in the filtrate. The precipitates with the filters were washed by the distilled water, transported to the same volumetric flasks, dissolved in the Peterman solution and determined in the solutions the amount of the citrate soluble forms of P₂O₅ and CaO. The results are given in the Table 1.

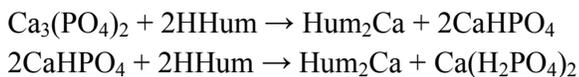
Table 1 – Nitro Humic Compound and Karatau Phosphorite Interaction Results

Determined components, %	Phosphorite composition	Mixture composition at different weight ratios phosphorite : nitro humic compound		
		1:0,25	1:0,5	1:1
P ₂ O ₅ total	25,01			
CaO _{total}	38,41			
P ₂ O ₅ water	-	0,35	1,85	3,76
CaO _{water}	0,49	0,56	1,04	1,54

Ca(H ₂ PO ₄) ₂ ·H ₂ O	-	0,62	3,28	6,67
P ₂ O ₅ _{citrate}	0,48	0,84	1,76	2,30
CaO _{citrate}	0,42	3,70	4,96	7,48
Ca(HPO ₄) ₂ ·2H ₂ O	1,16	2,11	4,26	5,67
CaO in humate	-	3,01	3,57	5,67
P ₂ O ₅ _{available}	0,48	1,22	3,61	6,06
P ₂ O ₅ _{water} : P ₂ O ₅ _{total}	-	1,40	7,40	15,03
P ₂ O ₅ _{available} : P ₂ O ₅ _{total}	1,92	4,88	14,43	24,23
CaO _{available}	0,91	4,26	6,00	9,02
CaO _{water} : CaO _{total}	1,28	1,48	2,71	4,01
CaO _{available} : CaO _{total}	2,37	11,09	15,62	23,48
pH	7,85	7,64	6,72	5,50

The experimental data show that addition of the nitro humic compounds to the natural phosphates provides for the appearance and increase of the 1) water soluble forms of P₂O₅ and CaO, which testifies to the formation of the mono calcium phosphate and 2) citrate soluble forms, which is caused by the dicalcium phosphate formation. Besides, increase of the nitro humic compound addition in the mixtures provides for the calcium humate amount increase, formed as a result of interaction. Binding of the calcium ions by the humic acids into the sparingly soluble salts, and the pH medium lowering provide for the dissolution of the new portions of the Karatau phosphorites.

As far as the main ingredient part of the natural phosphorites is tribasic calcium phosphate, and that of the nitro humic compound – humic acids, we may suppose the following reaction mechanism between them:



So, we have stated that nitro humic compound addition to the Karatau phosphorite provides for the P₂O₅ transition into the forms easily assimilated by the plants, it is accompanied by the formation of the slow acting organic fertilizer – calcium humate. It permits to use the nitro humic compounds as a humic fertilizer and as a substance which mobilizes the phosphorus when it is applied with the phosphorus fertilizers. So, there will be no necessity to have a stage of the preliminary phosphate raw materials decomposition by the mineral acids [6].

Soil structure is of great importance for the crop yield level rising. Unfortunately all the irrigated soils of the Central-Asian region, excluding some hydromorphic types, are structureless according to the structural classical point of view. All of them comprise less than 40 % of the water-stable aggregates of the 0,25 mm size, meanwhile this factor should be 70-80 % in the structural soils. In the desert zones on the takyr soils, for example in Kara-Kalpak, the content of the aggregates with the above mentioned sizes is lowered up to 5-10 % and even to null [7].

The perennial herbs sowing at the high level of agricultural technics had been the only means of the structure improvement and humus increase in the soil till the beginning of the seventies of the past century. I.V. Turin brilliantly characterized the then situation, he said: “If we had found the economically advantageous way of the artificial structure formation, considerably more perfect than structure formation under the influence of the perennial herbs, - we would have had a great revolution in our agricultural technics; its sequences might have been comparable with the revolution, which was caused in its time in the Western Europe by the wide application of the mineral fertilizers, which provided for the increase of the

average crop yield, reached 15 centner per hectare owing to clover, up to 25-30 centner per hectare, i.e. doubled" [7].

This is the reason why the artificial soil amendments search was undertaken all over the world. The Uzbek chemists supervised by the academician of the Academy of Sciences of Uzbekistan K.S. Akhmedov contributed greatly to the problem solving. They created the series of poly electrolytes of the "K" series, poly electrolytes of the "PAA" series, poly electrolytes of the "KMC" series, poly electrolytes of the "GU" series, poly electrolytes of the "poly imine" series [8-14]. All of them are perfect soil amendments [15, 16]. It is enough to say that at application of the K-preparation (0,15 % of the soil weight) to the typical sierozem the amount of the water-stable aggregates larger than 0,25 mm reaches 90,99 % [14].

Chirchik Production Association 'Electrochimprom' and Production Association "Navoiazot" industrially produced the preparations of "K" series. But unfortunately they have not reached the agriculture sphere. They are taken away by the drilling technicians to stabilize the liquid mud at drilling of oil-and-gas holes. They are very expensive for the agriculture. Their production needs such deficient and expensive products as polyacrylonitrile, acrylic acid, meta-acrylic acid, acrylonitrile, nitron fiber wastes and others.

So, we decided to create a new artificial soil structure former aiming the accessible and cheap initial materials. We have offered a mode of production of the artificial soil structure former, which includes the interaction of ammonium humates with an organic reagent at a high temperature; a distinctive feature of this mode is using the calurea and formaldehyde as an organic reagent with the aim to improve the quality of the produced structure former at the following component ratio, mass parts:

Ammonium humates	4,0–12,5
Calurea	1,9–1,5
Formaldehyde	0,5–1,5

The temperature is 85-95⁰C, duration of the process is 3-3,5 hours.

Example 1. Ammonium humates are preliminarily produced by the humate-containing raw materials processing (wastes of the brown coal briquette factories) with the 5% ammonia solution with the following separation of the insoluble residue by centrifuging. 150 ml of the 3,5 % ammonium humate solution (12,5 weight parts) are loaded into the reactor, add calurea 0,42 g (1 weight part), the reaction mixture is heated up to 85-95⁰C and add the formaldehyde 0,63 g (1,5 weight parts). The reaction mixture is heated 3 hours at 85-95⁰C. The reaction completion is determined by the free calurea absence. The reaction result is the solution, containing 5,9 g of the structure former (94% yield from the initial reagents).

The table 2 shows the influence of the initial reagents ratio and conditions of their interaction on the produced structure former quality.

Example 2. The soil sample is processed by the 2,5 % solution of the produced structure former up to the complete soil moisture capacity in the amount of 0,5 % of the dry substance to the dry soil weight. Then the soil sample is dried up to the air-dry condition and according to the G.I. Pavlov's method we determined the amount of the water-stable aggregates having the particle size more than 0,25 mm.

The offered method of the structure former production on the basis of ammonium humates, calurea and formaldehyde permits to produce the soil structure former by the one stage process; at the optimal application 0,2-0,5 % to the soil weight its exploration will provide for the increasing of the water-stable aggregates content in the soil up to 60-80 %, increasing of the humus content and the cotton crop yield up to 5,1-8,6 centner of the raw cotton on the hectare.

Table 2 – Experiment conditions influence on the structure former quality

Humate concentration, %	Sample of humate in terms of a dry substance, g	Amount of HNO ₃ in the solution, g	Calurea amount, g	Formaldehyde, g	Reaction temperature, °C	Reaction time, hr	Amount of the water-stable aggregates 0,25 mm, %	Free calurea content, g
3,5	5,0	2,5	0,42	0,63	95	5	67	-
5,5	5,0	2,5	0,42	0,63	95	5	64	-
10,0	5,0	2,5	0,42	0,63	95	5	1,5	1,9
5,0	25,0	7,5	2,20	3,30	85	5	64	-
5,0	25,0	7,5	2,20	3,30	80	5	2,0	1,8
5,0	25,0	7,5	2,20	3,30	95	3,5	64	-
5,0	25,0	7,5	2,20	3,30	95	3,0	62	0,4
5,0	25,0	7,5	2,20	3,30	95	2,5	22	0,8
5,0	25,0	7,5	2,20	4,40	95	3,5	2,0	1,6
5,0	25,0	7,5	6,25	8,40	95	3,5	68	-
-	-	7,5	2,20	3,30	95	5,0	1,9	1,9

The table 3 shows the water-stable aggregate amount dependence on the structure former dose and the soil type.

Table 3 – Efficiency of the structure former depending on its dose and soil type

Soil sample	Amount of the structure former to the soil weight, %	Amount of the water-stable aggregates, %
Powdered (less than 0,25 mm)	0,25	68
	0,50	80
Powdered (less than 0,25 mm)	0,25	65
	0,50	78
Cloddy (0-3 mm)		
Cloddy (0-3 mm)		

5 Conclusions

The brown coal oxidation process by the 56 % nitric acid was studied.

It was shown, that introduction of 8-20 % ammonium humates solution in the ammophos pulp make possible to receive a new high-performance fertilizer.

The offered method allows to produce a soil amendment on the basis of ammonium humates, calurea and formaldehyde by the one stage process. At the optimal application 0,2-0,5 % to the soil weight its exploration will provide for the increasing of the water-stable aggregates content in the soil up to 60-80 %, increasing of the humus content and the cotton crop yield up to 5,1-8,6 centner of the raw cotton on the hectare.

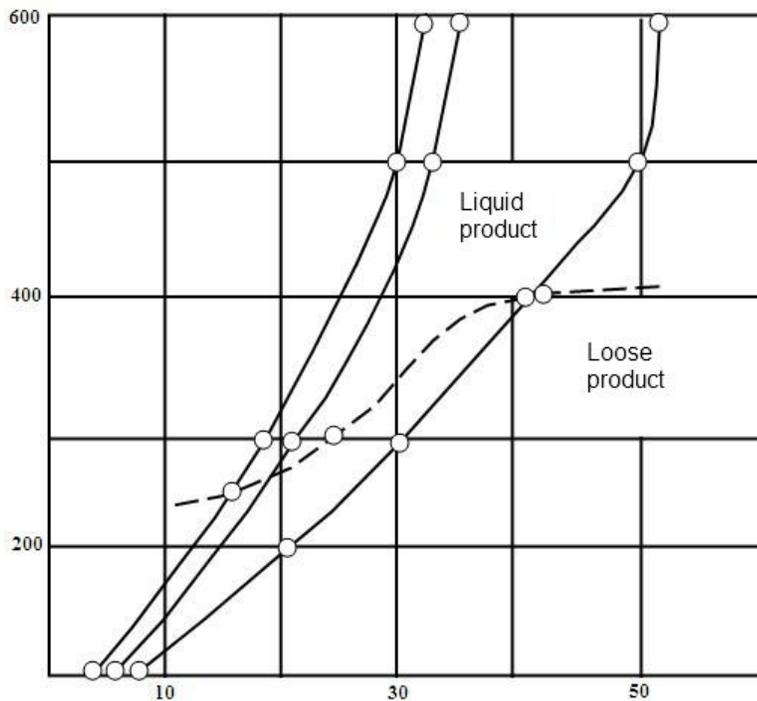
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List of figures

Fig.1 – Humic acid yield depending on the added HNO_3 amount:
1 – 30% HNO_3 ; 2 – 40% HNO_3 ; 3 – 55% HNO_3



Humic acid yield, weight %

Fig.2 – Influence of the ratio S:L on the humic acid yield: 1 – 55% HNO₃; 2 – 40% HNO₃; 3 – 30% HNO₃; 4 – 20% HNO₃

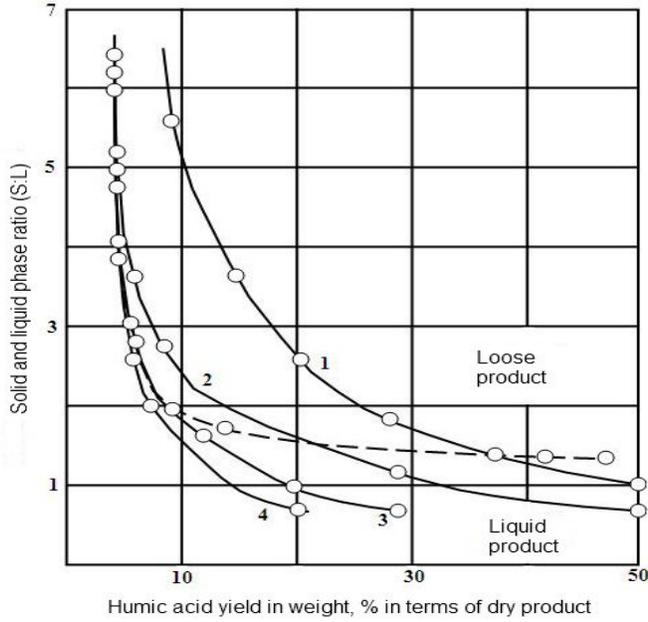


Fig.3 - Humic acids yield for a unit of the consumable HNO₃ (mono hydrate): 1 – 55% HNO₃; 2 – 40% HNO₃; 3 – 30% HNO₃; 4 – 20% HNO₃

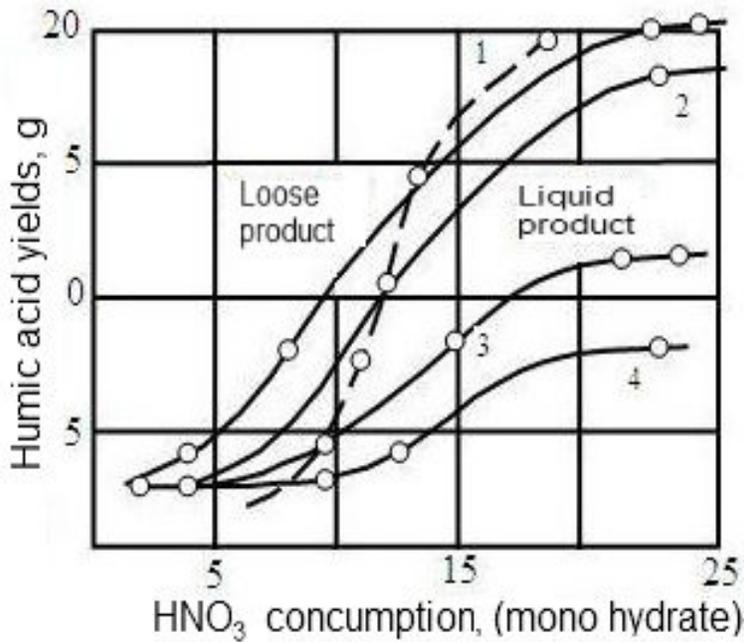


Fig.4 – A scheme of the laboratory setup for determination of the gas mixture composition, formed due to the interaction of the nitric acid with the coal

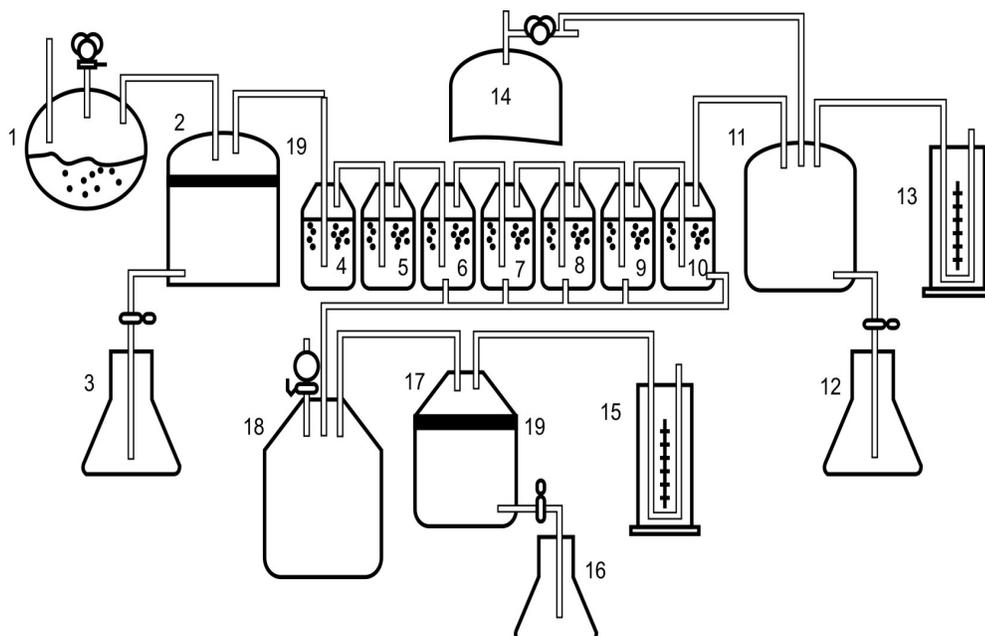


Fig. 5 – Humic acid yield at coal oxidation by the gaseous nitrogen oxides: 1 – calculation in %; 2 – calculation in grams; 3 - humic acid yield at the coal oxidation by the nitrogen oxides (for comparison), calculation in grams.

