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Research Article

Extraction Of Humic Acid from Local Brown Coal and Synthesis of Its Vinyl Ether

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ABSTRACT:

This article presents research on the extraction of humic acid from brown coal samples obtained from the Angren and Apartak coal deposits of Uzbekistan and the Karakyche deposit of Kyrgyzstan, as well as the heterogeneous-catalytic synthesis of its vinyl ether. The coal samples were pre-oxidized with nitric acid, followed by alkaline extraction to isolate the humic acid. The effect of oxidation conditions (acid concentration, mass ratio, and time) on the humic acid yield was investigated. The vinyl etherification of humic acid was carried out in the gas phase with acetylene in the presence of a $Zn(CH_3COO)_2/Al_2O_3$ catalyst. The functional composition of the obtained products was confirmed by IR spectroscopy.

Keywords: *Humic Acid, Brown Coal, Oxidation, Alkaline Extraction, Vinylation, Heterogeneous Catalysis, Nitric Acid, Vinyl Ether.*

INTRODUCTION

The ongoing depletion of oil and gas reserves is driving the chemical industry to seek alternative raw material sources. Brown coal plays a particularly important role in this regard, as it is relatively inexpensive and available in large reserves within our country. Uzbekistan possesses major coal deposits such as Angren, Shargun, and Boysun, and extracting high-value-added chemical products from them — not only fuel — is a strategic priority [1,2].

Humic acids are important biologically active compounds found in brown coal, with wide applications in agriculture, pharmaceuticals, ecology, and industry. However, the organic acids required by the chemical industry are currently largely imported. The production of humic acid from local brown coal would address this problem and contribute to economic independence [3,4].

The scientific novelty of this work lies in the fact that the vinyl ether of humic acid was synthesized for the first time using a heterogeneous-catalytic method in the gas phase with acetylene in the presence of a $Zn(CH_3COO)_2/Al_2O_3$ catalyst. The aim of this study is to optimize the conditions for extracting humic acid from local brown coals and to synthesize a new chemically modified product — the vinyl ether.

MATERIALS AND METHODS

Research Objects

Three coal samples were used in the study: Angren coal deposit (grade 2BR-B2), Apartak coal deposit (grade 2BOMSSH-B2), and the Karakyche coal deposit of Kyrgyzstan. The main technical characteristics of the samples are presented in Table 1.

Table 1. Main technical characteristics of the coal samples

Coal Deposit	Moisture, %	Ash Content, %	C Content, %	O Content, %
Angren	13.95	68.92	70.12	23.67
Apartak	11.50	76.18	72.11	20.91
Karakyche	12.69	11.3–18.4	76.21	19.90

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Humic Acid Extraction Procedure

The coal samples were ground to a particle size of 0.050 mm in a ball mill. 500 ml of a 5% NaOH solution was added to 100 g of the coal sample, and the mixture was heated at 80 °C for 2 hours. H_2SO_4 was added to the filtered solution to acidify it to pH = 2. The resulting precipitate was separated using a vacuum filter, washed with ethanol to remove hymatomelanic acid, and dried at 30–40 °C [5,6].

Pre-oxidation of Coal with Nitric Acid

The coal samples were oxidized with 30%, 40%, and 50% HNO_3 solutions at coal-to-acid mass ratios of 2:1, 1:1, and 1:2 for durations of 5, 10, 20, and 40 minutes. At the end of each experiment, the humic acid yield was determined as a percentage.

Catalyst Preparation

The heterogeneous catalyst was prepared by impregnation: a saturated solution of 30 g $Zn(CH_3COO)_2$ was impregnated onto 70 g of Al_2O_3 (or activated carbon) granules and dried at 130–150 °C for 2 hours. This produced $Zn(CH_3COO)_2/Al_2O_3$ and $Zn(CH_3COO)_2$/activated carbon catalysts.

Vinylation Process

The vinyl ether of humic acid was synthesized in a vertical quartz reactor under gas-phase flow conditions. Acetylene gas was purified and fed into the reactor via a dosimeter. The temperature range studied was 200–380 °C. The humic acid-to-acetylene molar ratio was varied from 3:1 to 1:5. The product was analyzed by IR spectroscopy (IRTracer–100).

RESULTS AND DISCUSSION

Humic Acid Yield by Alkaline Extraction

The amount of humic acid extracted directly from unoxidized coal samples by alkaline extraction was determined. The Angren coal yielded 20.19 g of humic acid per 100 g (20.2%), Apartak coal yielded 12.95 g (13.0%), and Karakyche coal yielded 15.08 g (15.1%). The high humic acid yield from Angren coal is attributed to its high oxygen content (23.67%).

Effect of Acid Concentration and Mass Ratio

Oxidation of coal with HNO_3 significantly increased the humic acid yield. The results obtained for Angren coal are presented in Table 2.

Table 2. Effect of acid concentration and mass ratio on humic acid yield (Angren coal, 30 minutes)

Coal:Acid Ratio	HNO_3 30%, HA Yield %	HNO_3 40%, HA Yield %	HNO_3 50%, HA Yield %
2:1	45.5	60.0	63.0
1:1	51.0	66.0	71.0
1:2	67.8	75.0	78.0

As shown in Table 2, increasing the acid concentration from 30% to 50% and changing the mass ratio from 2:1 to 1:2 raised the humic acid yield in the Angren sample from 45.5% to 78.0%. A similar trend was observed for the Apartak (42.5→76.4%) and Karakyche (40.8→74.9%) samples. The increase in acid concentration is attributed to deeper destruction of the coal's organic polymer structure.

Effect of Oxidation Time

Extending the oxidation time from 5 to 40 minutes also significantly increased the humic acid yield. With 50% HNO_3 at a 1:1 mass ratio and 40 minutes of oxidation, yields of 81.7% for Angren coal, 80.5% for Apartak, and 79.2% for Karakyche were obtained. These results demonstrate that coal from all three deposits is capable of producing high humic acid yields under optimal conditions.

Vinylation of Humic Acid

The effects of temperature, catalyst type, and molar ratio on the yield of humic acid vinyl ether were studied. The optimal temperature with the $Zn(CH_3COO)_2/Al_2O_3$ catalyst was 340 °C, at which a yield of 44.5% was obtained. With the activated carbon-based catalyst, a yield of 27.3% was achieved at 340 °C. The higher efficiency of the Al_2O_3-based catalyst is associated with its well-developed porous surface and its superior ability to retain zinc acetate.

Regarding the effect of the initial molar ratio: at a humic acid-to-acetylene ratio of 1:4–1:5, a maximum yield of 81.2% was achieved using the $Zn(C_5H_{11}COO)_2/Al_2O_3$ catalyst. The excess

acetylene enables full activation of the reactive sites on the catalyst surface.

Table 3. Effect of temperature and catalyst type on the yield of humic acid vinyl ether

Temperature, °C	Yield (Activated Carbon), %	Yield (Al_2O_3), %	Note
200	13.2	26.6	Low yield
240	15.8	28.9	
280	19.1	33.1	
300	24.5	39.7	
340	27.3	44.5	Optimal temperature
380	15.7	27.3	Decomposition observed

IR SPECTROSCOPY RESULTS

The IR spectra of the obtained humic acid samples showed the following major absorption bands: 2960 cm^{-1} (CH_3 stretching vibration), 2874 cm^{-1} (CH_2 stretching vibration), 1724 cm^{-1} (C=O carboxyl group), 1607 cm^{-1} (carbonyl group), 1470 cm^{-1} (aromatic ring), 1300–1000 cm^{-1} (C–O bonds), 3310 cm^{-1} (–OH groups), 883 cm^{-1} (CH_2 deformation vibration). These data confirm that humic acid is a polyfunctional organic compound capable of undergoing vinylation reactions.

CONCLUSION

1. Humic acid was extracted in yields of 12.95–20.19% from brown coals of Uzbekistan (Angren, Apartak) and Kyrgyzstan (Karakyche) by alkaline extraction.

2. Pre-oxidation of coal with HNO_3 allowed the humic acid yield to be increased by 3–4 times. The optimal conditions were: HNO_3 concentration of 50%, oxidation time of 40 minutes, and mass ratio of 1:1, under which the yield reached 78–81.7%.

3. The vinyl ether of humic acid was synthesized for the first time by a heterogeneous-catalytic method at 340 °C with a yield of 44.5% using a $Zn(CH_3COO)_2/Al_2O_3$ catalyst. At a molar ratio of 1:4–1:5 (humic acid:acetylene) and using the $Zn(C_5H_{11}COO)_2/Al_2O_3$ catalyst, the maximum yield reached 81.2%.

4. IR spectroscopy confirmed the presence of carboxyl, hydroxyl, aromatic, and methylene functional groups in the humic acid composition.

5. The obtained results demonstrate the feasibility of producing high-value-added products intended for the agricultural and pharmaceutical industries through deep chemical processing of local coal raw materials.

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