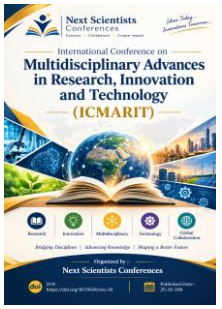
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Synthesis And Analysis Of Naphthalene Sulfonate–Formaldehyde Resin Based On Pyrolysis Oil

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ABSTRACT

This article examines the physico-chemical and technological basis of the sulfonation of pyrolysis oil, a by-product of the Ustyurt Gas Chemical Complex (operated by Uz-Kor Gas Chemical LLC), with 96% sulfuric acid. The possibility of obtaining a functional product containing sulfo groups by sulfonating the aromatic and reactive hydrocarbon components of pyrolysis oil is considered. Based on a systematic analysis of the effect of the ratio of the starting components and the reaction temperature on the course of the process and the properties of the final product, the optimal technological conditions for sulfonation were determined.

KEYWORDS: Pyrolysis oil, sulfonation, sulfuric acid, sulfo group, chemical modification, IR spectroscopy, reaction temperature, component ratio, secondary raw materials.

INTRODUCTION

Processing secondary raw materials in chemical production not only reduces the volume of waste but also makes it possible to obtain value-added products from it. At the Ustyurt Gas Chemical Complex, pyrolysis distillate, pyrolysis oil and resinous streams are formed alongside polymer production. Owing to their content of aromatic compounds, these streams are a promising local feedstock for organic synthesis.

This work focuses on the sulfonation of the aromatic components of pyrolysis oil with sulfuric acid. The introduction of sulfo groups changes the chemical functionality of the feedstock, and the subsequent condensation of the resulting intermediate with formaldehyde yields a material of the NSF (naphthalene sulfonate–formaldehyde) type. The main objective of the study is therefore to select the optimal sulfonation conditions and to evaluate the instrumental and physico-chemical characteristics of the final product.

Pyrolysis oil (a secondary raw material of the chemical complex) contains up to 50% naphthalene. The heavy oils separated from it are usually divided into fractions: a light fraction containing benzene and toluene, and a heavy fraction (180–250 °C) consisting of ethylbenzene, xylenes and other aromatic compounds, as well as indene, naphthalene and other cyclic aromatic compounds. Indole and α- and β-methylnaphthalenes can be obtained from the high-boiling fraction (240–255 °C); seven of the ten possible dimethylnaphthalenes have been isolated from the 260–270 °C fraction, and on cooling the 280–290 °C fraction a large amount of mixed crystals precipitates, consisting mainly of acenaphthene, diphenyl oxide and fluorene.

2,3,6- and 1,3,7-Trimethylnaphthalenes have been isolated from residual petroleum oil. Skatole is present together with its 2-, 5- and 7-isomers; its isolation is based on the fact that none of these four compounds forms a carboxylic acid when their sodium derivatives react with carbon dioxide. Physical properties of naphthalene: density 1.14 g/cm^3, melting point 80.26 °C, boiling point 217.7 °C, solubility in water 30 mg/L, flash point 79–87 °C, molar mass 128.17052 g/mol, vapour pressure (at 80 °C) 1040 Pa.

The sulfonation experiments were carried out in a 250 mL three-necked glass flask equipped with a paddle stirrer with adjustable speed and an oil bath. A measured amount of pyrolysis oil was placed in the reactor and heated to 70 °C. When the set temperature was reached, the required amount of sulfuric acid was added to the reaction mixture at a constant rate over 15–20 seconds. The use of sulfuric acid made it possible to avoid an excessive rise in the temperature of the reaction mixture; the reaction temperature was then raised to 160 °C and held at this level for 3 hours. Temperature changes during the reaction were recorded. At the end of the reaction, the mixture was rapidly cooled to 110 °C, after which a 36% formalin solution was added slowly. The resulting product was neutralized.

Studying the sulfonation rate is complicated by the low solubility of pyrolysis oil in sulfuric acid. In this heterogeneous system, the effect of mass transfer on the reaction rate was eliminated by intensive stirring. To confirm that the rate measurements were not affected by mass-transfer limitations, the conversion of sulfuric acid was measured at different stirring speeds.

Because of the limited solubility of pyrolysis oil in sulfuric acid, mass transfer may affect the rate of the process. The stirring regime was therefore studied separately. At rotation speeds above 10 rev/s, the conversion of sulfuric acid remained practically unchanged, which made it possible to use a regime in which the effect of mass-transfer limitations was reduced in the subsequent experiments.

No.	T, °C	Ms	Mn	c
1	155	119.6	100.0	1.5
2	160	90.8	128.0	0.8
3	165	119.5	100.0	1.5
4	165	195.5	100.0	1.5
5	165	119.0	100.0	1.5

Table. Experimental conditions for the sulfonation of pyrolysis oil

The sulfonation experiments were carried out at initial molar ratios in the range of 1–1.5% and at temperatures of about 160–165 °C. In the experiments with 96% sulfuric acid, the formation of monosulfonated naphthalene derivatives was observed. The analysis showed that the maximum content of naphthalene sulfo derivatives was 1.4% by mass, and the resin content was about 0.1%.

The experimental results show that temperature and reagent ratio in the sulfonation process should not be considered separately; they should be treated as interrelated process parameters. Raising the temperature increases reactivity, but excessive activity may increase the likelihood of side reactions. Therefore, when selecting the process parameters, the criterion should include not only the yield of the sulfonated product but also the quality of the NSF product obtained by subsequent condensation.

From a practical point of view, this approach can help increase the value of pyrolysis oil as a secondary raw material. To further improve the process, it is advisable to study the component

ratio over a wider range, to confirm the molecular structure by additional instrumental methods and to evaluate the properties of the product in practical applications.

Conclusion

The study experimentally evaluated the possibility of obtaining a product based on naphthalene sulfonate–formaldehyde (NSF) resins by sulfonating pyrolysis oil with 96% sulfuric acid, followed by condensation of the sulfonated product with a 36% formalin solution. The sulfonation stage was carried out at 155–165 °C (the main experiments at 160–165 °C), and the ratio of the starting components was varied within 1–1.5%.

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