

SYNTHESIS OF DIAMINE BASED ON DOMESTIC WASTE

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Abstract. In this article, a research on obtaining diamine based on the processing of household waste containing polyethylene terephthalate was conducted. At first, the wastes were washed and cleaned and subjected to alkaline hydrolysis, and trephthalic acid was obtained by neutralizing the resulting solution with hydrochloric acid. The obtained results were studied and analyzed using IR-spectra.

Keywords: household waste, trephthalic acid, p-phenylenediamine, alkaline hydrolysis.

Introduction

As we know, today all over the world, the amount of waste containing polyethylene terephthalate has exceeded one million tons, and these numbers continue to grow day by day. In the last few decades, these wastes can be reduced by recycling them. Carbonic hydrolysis of polyethylene terephthalate was first described by Waters in 1950 [1]. It is known that PET is very resistant to weak alkaline solutions, resistant to concentrated alkaline solutions at room temperature and begins to decompose only at the boiling point [2] This is due to the high packing density of PET not only in crystalline but also in amorphous parts. But this is an approximate qualitative indicator of polymer stability.

Only as a result of repeated hydrolytic degradation, the polymer can be separated into fragments corresponding to one monomer unit. The relief of the surface is formed for a certain period of time and does not change after that [3]. According to some researchers, the peculiarity of the hydrolysis reaction of PET with sodium hydroxide is its spontaneous acceleration, which is explained by the activity of the amorphous phase and the increase in the number of acid groups.

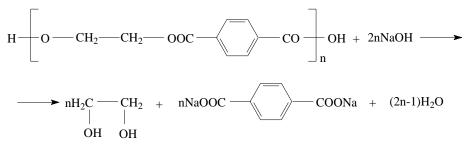
High-purity p-Phenylenediamine (p-PDA) is used primarily as a monomer in the production of some polycondensation synthetic polymers, antioxidants for petroleum and polymer products, and as a vulcanization accelerator. Currently, p-



PDA is not produced in Russia and comes mainly from China. The most important industrial methods for producing p-PDA are: the reduction reaction of nitro compounds (reduction of nitro compounds with cast iron shavings according to Bechamp, sulfur alkalis according to Zinin, zinc dust, hydrogen, etc.) [4]; carboxylation of aniline in the presence of alkali metal alcoholates, followed by treatment with a nitrating mixture and then hydrogenation with hydrogen on a palladium catalyst (DuPont, USA), as well as chemical transformations along the chain: chlorination of benzene \rightarrow nitration of chlorobenzene \rightarrow production of 1,4dinitrobenzene \rightarrow hydrogenation on a palladium catalyst to p-PDA (Mitsubishi Chem, Japan). The presented multi-stage methods for obtaining p-PDA are technologically complex, energy-intensive, and are accompanied by a large number of harmful effluents and gas emissions. The purpose of this work is to study the patterns of synthesis of p-PDA of monomeric purity based on domestic raw materials - terephthalic acid diamide.

Experimental part

The hydrolysis reaction of PET with sodium hydroxide is as follows:

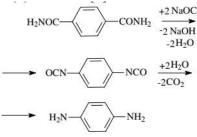


1750-1800 rpm to obtain terephthalic acid from secondary PET. A reactor equipped with a high-speed mechanical stirrer and a thermometer was used. Hydrolysis of polyethylene terephthalate is carried out in the following sequence. For the study, we mainly used secondary PET. The process was carried out for 2 hours at a temperature of 95-100°C using a 40% sodium hydroxide solution. An aqueous solution of sodium terephthalate was formed as a reaction product. The resulting solution was then diluted with water and filtered to remove unreacted PET. The resulting solution was neutralized with sulfuric acid. The formed terephthalic acid was separated in the form of a white powdery precipitate because it was not well soluble in water. The precipitate was filtered and then washed 3-4 times with boiling water. We dried the finished product in a drying oven at a temperature of 55-



60°C until it reached a constant mass. The resulting terephthalic acid was used as a raw material for the synthesis of organic pigments, which are the main focus of our research.

Synthesis p-FDA. The synthesis of p-FDA (3) is carried out by the regrouping of Hoffmann's diamide terephthalic acid (DATFK) (1) with aqueous solutions of sodium hypochlorite in an alkaline medium through the stage of formation of isocyanate (2) according to [5].



The initial reagents were DATPhK with a mass fraction of the main substance of 98–99%, aqueous solutions of NaOCl with a mass fraction of active chlorine of 5–20%, and aqueous solutions of NaOH with a mass fraction of the main substance of 9–46%.

The synthesis of p-PDA was carried out in a 3-liter glass reactor equipped with a thermometer, reflux condenser, stirrer, dropping funnel, and heat exchange jacket.

The calculated amounts of DATPhK, NaOCl, and NaOH were placed in the reactor at a temperature of 0-5 °C, after which the stirrer was turned on and the reaction mixture was maintained at 10 °C for 3 h. Then it was slowly heated to 38–42 °C. At 38–42 °C, an exothermic rearrangement reaction begins. The temperature quickly rises and reaches 65–85 °C after 1 min. By resuming cooling, we ensured that the reaction temperature during the temperature jump did not exceed 85 °C. The solution, which had acquired a brownish tint, was heated for another 30 min at 60 °C. Then it was heated to 80 °C for 15 min and maintained at this temperature for 1 h. In this case, the final reaction mass was a transparent water-salt solution of a brown tint with a mass fraction of p-PDA of 5–18%. p-PDA was extracted from the water-salt solution in three stages. Initially, azeotropic distillation of water was carried out. For this, toluene was added to the solution at a weight ratio of reaction mass: toluene equal to 1:3 and azeotropic distillation of water was carried out at a temperature of 120–130 °C until the latter was completely absent from the reaction mixture (the presence of water worsens the quality of the target product).





Results and Discussion

The structure of the substance obtained as a result of this reaction was studied and analyzed using the IR spectrum (Fig. 3.1).

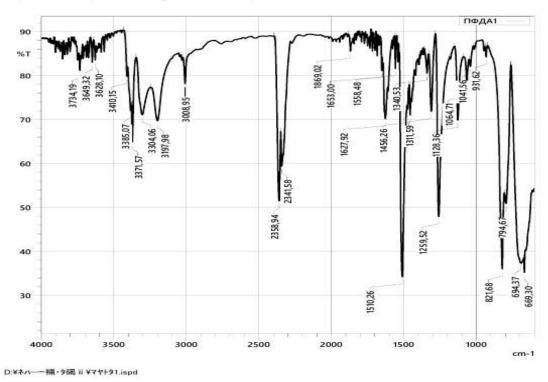


Figure 3.1. IR spectrum of p-phenylenediamine

In the IR spectrum analysis, symmetric and asymmetric valence vibrations of N-H bonds of two amine groups are observed in the 3197, 3304, 3371, 3385, 3410 cm⁻¹ regions. Deformation vibrations of the amine group are observed in the region of 794 cm⁻¹. In addition, we can observe the deformation vibration of the C-N-H bond in the 1627 cm⁻¹ area and the valence vibrations of the C-H bonds in the aromatic ring in the 3008 cm⁻¹ area. Deformation vibrations of C-H bonds along the plane of the ring are observed in the 1128, 1064 cm⁻¹ region, and out-of-plane vibrations are observed in the 694 cm⁻¹ region. Valence vibrations of C-C bonds in the aromatic ring are observed in the 1456, 1510 cm⁻¹ regions, two corresponding peaks in the 1667-2000 cm⁻¹ region and ring deformation vibrations in the 821 cm⁻¹ region are observed due to the distribution in the para state. Valence vibrations are observed in the area of 1340s m⁻¹, deformation vibrations in the area of 1259cm⁻¹.



Conclusion

As a result of the research, the analysis of the processing of household waste and the production of diamines based on it was conducted. The obtained results were studied with IR-spectra and their approximate formulas were proposed.

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