



SYNTHESIS AND STRUCTURAL ANALYSIS OF DIVINYL ETHER OF SALICYLIC ACID

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ABSTRACT

The article discusses the relevance of the chemistry of acetylene compounds, its derivatives, such as vinylacetylene, its interaction with salicylic acid, the conditions for the synthesis of vinylacetylene ester. The article also presents the results of the spectral (IR) analysis of the synthesized salicylic acid ester and the interpretation of the spectra of functional groups and the conclusions based on them.

KEY WORDS: acetylene, vinylacetylene, salicylic acid, salicylic acid divinyl ester, IR spectroscopy, DMSO (dimethyl sulfoxide), DMFA (dimethylformamide).

INTRODUCTION

Acetylene derivatives used in various sectors of the national economy and the chemical industry have an important role today. These are anti-inflammatory, antipyretic drugs, biologically active substances, insecticides and herbicides in agriculture, anti-corrosion inhibitors, etc [1,2,15].

The important role and high demand for compounds and derivatives of acetylene in various fields requires a thorough study of known and synthesis of new derivatives aimed at various sectors of the national economy. More than a hundred years have passed since the discovery of the Favorsky reaction, interest in the chemistry of acetylene derivatives does not lose its significance. During this period, a very large amount of work has been done in this area. A large number of acetylenic aldehydes and ketones, arylvinyl esters have been obtained, which are widely used in various fields.

In this regard, it is appropriate to note the scientific works of W. Reppe, B. A. Trofimov and J. J. Keynesper, which are devoted to the synthesis of vinyl esters of carboxylic acids [1,3,18]. F.M.Kimmich studied the technology of the interaction of benzoic acid with acetylene with the participation of a heterogeneous platinum catalyst, E.Drent proposed a technology for the synthesis of vinyl esters from branched carboxylic acids and acetylene with the participation of Ru-Cd-Cu salts at high pressure, M.L. Davis studied the process of cyclopropanization of vinyl ethers, S. Harrison - the process of polymerization of vinyl ethers, etc [4,5,17].

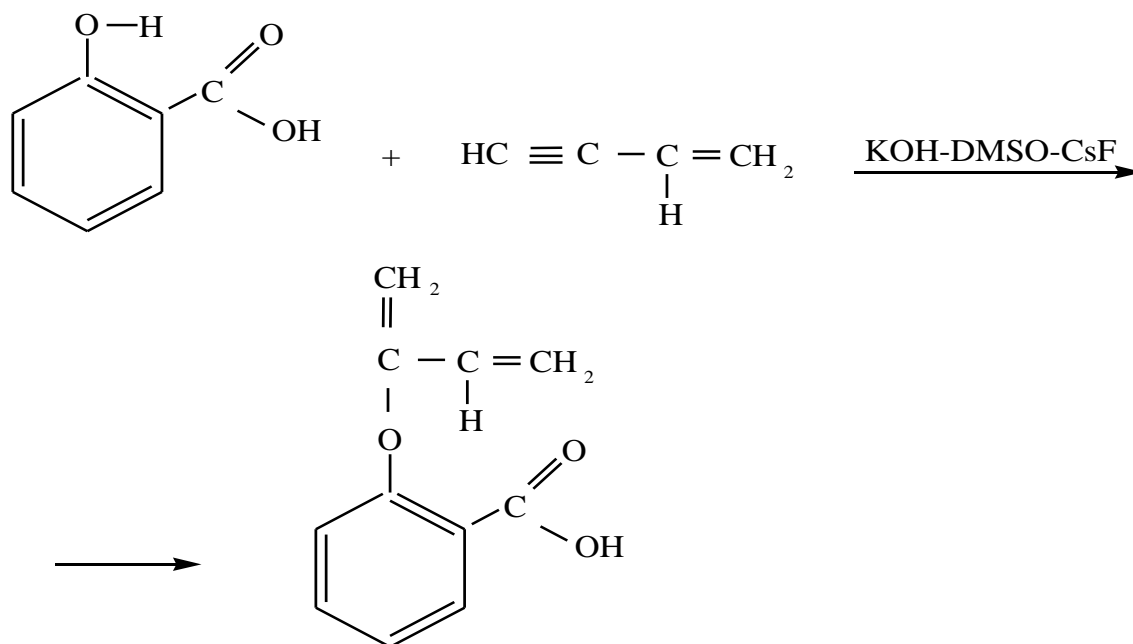
MATERIALS AND METHODS

The high interest in the chemistry of acetylene and its derivatives was the reason for our research in this area. The aim of our research was to study the process of synthesis of vinyl esters of salicylic acid by a homogeneous catalytic reaction and its derivatives with vinylacetylene, as well as the synthesis of vinyl esters of salicylic acid and its derivatives from vinylacetylene without catalysts, as well as to conduct X-ray diffraction analysis of the synthesized compounds to study the electronic structure of the synthesized compounds, in the subsequent study of their composition and properties, the development of technology for the production of divinyl ester of salicylic acid, as well as the identification of their scope [5,6,20].

Synthesis of complex divinyl ester of salicylic acid was carried out in the following order: 0.08 mol of 2-hydroxybenzoic acid is added to 100 ml of 35% potassium hydroxide solution and placed in a magnetic stirrer until a homogeneous solution is formed. The synthesized and cooled (to -3 °C) solution of vinylacetylene in o-xylene (or chlorobenzene) is combined with a Drexel flask with a solution of 2-hydroxybenzoic acid. When a solution of vinylacetylene is heated, the gas released from it passes into a solution of 2-hydroxybenzoic acid. The process continues for 55-60 minutes until the vinylacetylene solution is saturated



with 2-hydroxybenzoic acid (up to a maximum of 1.5 hours). After completion, the resulting reaction mixture was cooled, dissolved in 150 ml of water and extracted with diethyl ether. The extraction product is dried with anhydrous calcium chloride, and the solvent is distilled off in a rotary evaporator. In this case, β -divinyl ester of 2-hydroxybenzoic acid is obtained in crystalline form with the yield of the reaction product up to 66%. The molecular weight of β -divinyl ester of 2-hydroxybenzoic acid is 204 g/mol, the boiling point is 285.2 °C and with a melting point of 71.9 °C [7,8,19]. The process of interaction of salicylic acid with vinylacetylene to form salicylic acid divinyl ester can be expressed using the following equation:



Unlike the synthesis of this drug by other methods, the proposed method for synthesizing divinyl ester of salicylic acid is distinguished by its performance under mild conditions, that is, the synthesis is carried out at low pressure, temperature and without the participation of a catalyst. The synthesis is carried out in an alkaline medium. The study systematically studied the conditions for optimizing the reaction of salicylic acid with vinyl acetylene using the strongly alkaline systems KOH-DMSO and KOH-DMFA. [8,9,10,].

RESULTS

The most reliable way to determine the composition of a substance, the presence of certain functional groups and bonds in it is IR - spectroscopic analysis of a substance. We obtained IR spectra of both salicylic acid and divinyl ester of salicylic acid. The following are the results of the obtained IR - spectra of compounds: [11,12].

In the IR spectra of salicylic acid (Fig.-1), the absorption spectrum of the $\text{C}=\text{O}$ group is observed in the region of 1722 cm^{-1} , in the region of 1420-1330 cm^{-1} , stretching vibrations of acidic hydrogen of the hydroxyl $-\text{OH}$ group of phenol are observed and in the region of 3439 cm^{-1} there is a stretching vibration of the hydroxyl group.

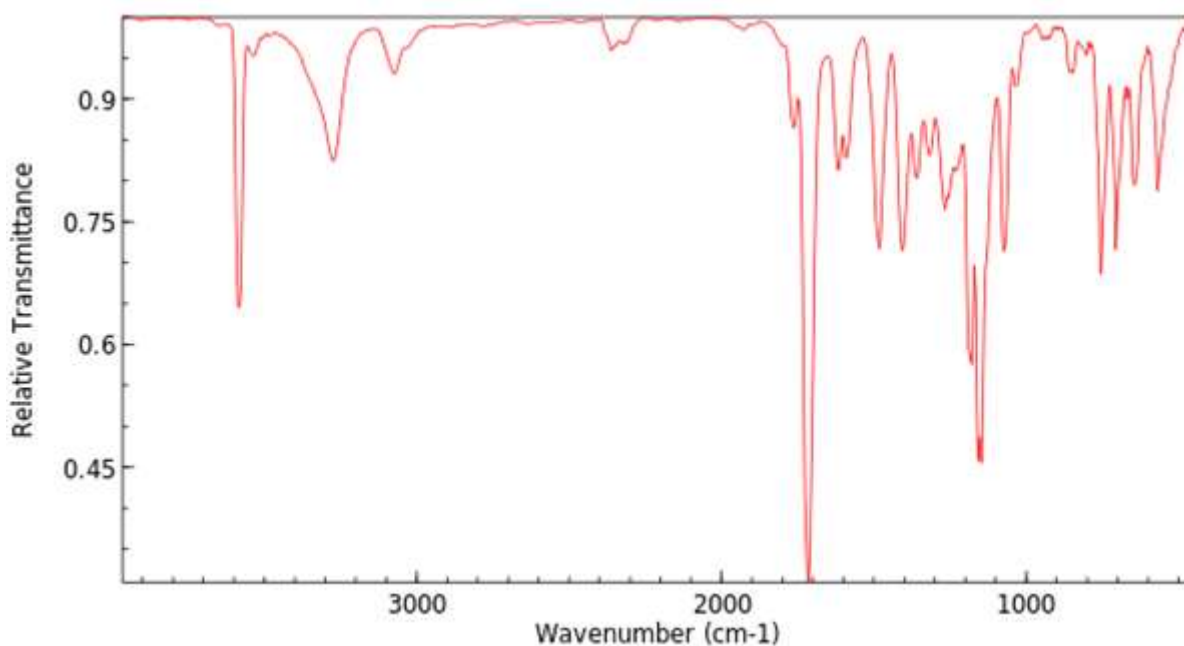


fig.-1. IR spectra of salicylic acid.

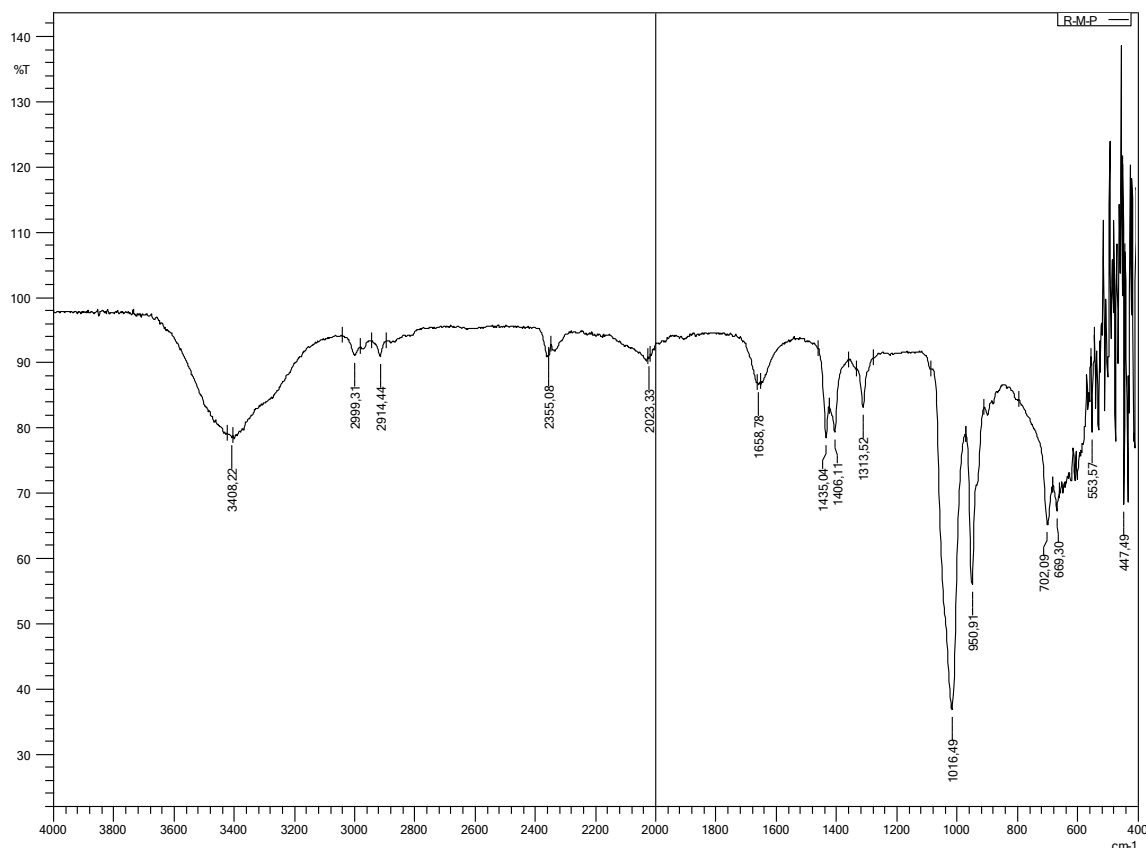


fig-2. IR spectra of divinyl ester of salicylic acid.

Interpretation of the stretching vibrations of the divinyl ester of salicylic acid (Fig-2), in contrast to salicylic acid, has bending vibrations in the region of 885 cm^{-1} related to the $=\text{CH}_2$ group, in the $2930\text{-}1409\text{ cm}^{-1}$ $\equiv\text{CH}$ group, in the region of 1075-



1020 cm^{-1} =C-O-C groups in the region of 1652 cm^{-1} stretching vibrations of the vinyl -CH=CH₂ group, which state that a chemical interaction has occurred between salicylic acid and vinylacetylene [13-21].

CONCLUSION

So, the data obtained indicate that, in fact, the reaction product, divinyl ester of salicylic acid, was formed from the reagents, as evidenced by the results of IR spectroscopy of salicylic acid and divinyl ester of salicylic acid.

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