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Research Article

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Synthesis and characterization of Benzoic Acid

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Abstract

In this paper, tests of benzoic acid synthesis, melting point test, benzoic acid content test and determination of benzoic acid identification by IR technique were performed. The results showed that the tested samples meet the specified quality requirements of official pharmacopeial monographs [5].

Keywords: benzoic acid, additives, preservatives, synthesis

1. Introduction

Benzoic acid is a white, solid, aromatic acid, used in a wide range of cosmetic products as a pH regulator, and in a wide range of cosmetic products. It is one of the preservatives and is under the European approval number e 210. Preservatives are added to stabilize the product, extend its shelf life, and prevent spoilage or microbiological contamination [1]. Preservatives are grouped into different groups depending on their source, mode of synthesis, structure, and role [2]. Additives and preservatives must not adversely affect the nutritional value of food or cosmetic products, they must comply with several regulations prescribed by the competent authorities. Additives are marked with an E-number as a confirmation of toxicological evaluation and classification of each additive. The most common preservatives are sorbic acid, propionic acid and benzoic acid, and the most common preservative in cosmetics is paraben. They can be associated with allergic reactions of the body, migraine and asthma attacks, obesity, and behavioral disorders, especially in children [3, 4].

2. Materials and Methods

Materials and Equipment

The following reagents were used: sodium hydroxide, benzaldehyde, ethanol, sodium permanganate, hydrochloric acid, charcoal, phenolphthalein 1% in ethanol and samples of benzoic acid. All the chemicals and reagents used were of analytical grade. A Shimadzu UV-Visible double beam spectrophotometer (Shimadzu, Japan) with matched 1 cm quartz cells was used for the measurements, balance Mettler Toledo, Buchi B- 545 Melting point, and IR spectrometer Shimadzu.

Method

On samples of benzoic acid obtained in the laboratory, benzoic acid synthesis tests, melting point tests, determination of benzoic acid content and determination of benzoic acid identification by IR technique were performed, following the monograph from the European Pharmacopoeia Preparation.

Synthesis of Benzoic acid

In a 100 ml Erlenmeyer flask, dissolve 1.25 g of solid sodium hydroxide in 25 ml of distilled water while stirring. Then weighed 2 g of potassium permanganate and divided it into 4 portions. Transfer the first portion to the Erlenmeyer flask containing the sodium hydroxide solution. When the potassium permanganate has dissolved, add 1.25 g of benzaldehyde to the reaction mixture. During the reaction, a slow loss of the violet color of the reaction mixture was noticeable. When the reaction mixture becomes colorless (a portion of potassium permanganate has reacted), insert a new portion of potassium permanganate from the test tube. In the same way, we add the third and fourth portions of potassium permanganate. Heat the Erlenmeyer flask with the reaction mixture in a water bath to a temperature of 70˚C and occasionally stir with a glass rod. After adding the fourth portion, stir the reaction mixture for a few more minutes, then stop heating and cool the Erlenmeyer flask to room temperature. The oxidation reaction is stopped, that is, excess potassium permanganate is removed by adding 1.5 ml of ethanol.

Figure 1: Synthesis of Benzoic acid- step I

Filter the cooled mixture into a clean glass. The filtrate contains benzoic acid in the form of a soluble sodium salt (sodium benzoate). We add concentrated hydrochloric acid in drops until an acidic environment is reached in the filtrates (we use litmus paper). After that, we add another 2-3 ml of concentrated hydrochloric acid, during which free benzoic acid precipitates.

Figure 2: Synthesis of Benzoic acid- step II

The precipitated benzoic acid is filtered on a Buchner funnel, and the precipitate is washed with a little distilled water. The crude product, benzoic acid, is recrystallized from water.

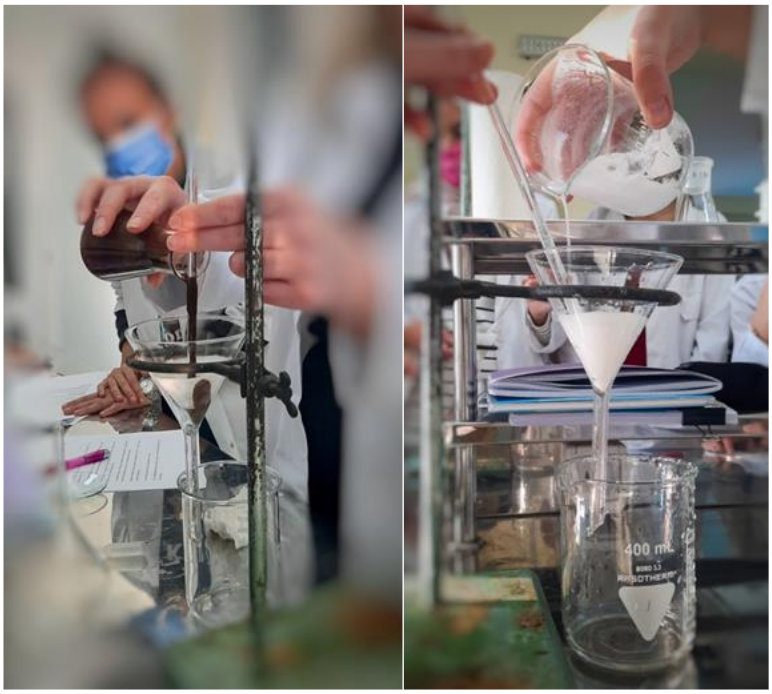

Figure 3: Synthesis of Benzoic acid- step III

After drying the benzoic acid, we weigh our product on a paper tray and determine the melting point.
The benzoic acid content was tested according to the Essay described in the European Pharmacopoeia.
Preparation and standardization of sodium hydroxide
1 M sodium hydroxide: Dissolve 42 g of sodium hydroxide, R in a vacuum without the presence of carbon dioxide R and make up to 1000.0 ml with the same solvent.
Standardization of sodium hydroxide
Titrate 20.0 ml of sodium hydroxide solution with 1 M hydrochloric acid using the prescribed indicator in the determination in which 1 M sodium hydroxide is used.
Indicator - phenolphthalein
0.1 M sodium hydroxide: Add 100.0 ml of 1 M sodium hydroxide to 1000.0 ml with water, without the presence of carbon dioxide R.
Standardization
The titration is performed as described for 1 M sodium hydroxide using 0.1 M hydrochloric acid (European Pharmacopoeia).
Determination of the identification of benzoic acid - IR technique
The solid sample for recording the IR spectrum is prepared in the form of a KBr lozenge or as a suspension in paraffin oil. A KBr lozenge is prepared by mixing the sample with dry potassium bromide (1 mg sample: 100 mg KBr). It is necessary to grind the mixture well, so that there is no excessive scattering of radiation on the crystals of the sample and potassium bromide. The sample is made into thin lozenges about 1 mm thick and 1 cm in diameter. The KBr lozenge is placed in a suitable holder and the IR spectrum is recorded. KBr does not absorb radiation in the mid-IR range, so this technique of solid sample preparation is most often used (European Pharmacopoeia 2.2.24).

3. Results & Discussion

Synthesis

The first step of the reaction is the nucleophilic addition of the hydroxyl group to the carbonyl group of the aldehyde. The resulting addition intermediate is a reducing agent because it releases a hydride ion that is added to the carbonyl group of another benzaldehyde molecule, thus reducing it. This step is, therefore, oxidation-reduction and alkoxide and benzoic acid are formed. The resulting benzoic acid, as a stronger acid, protonates the alkoxide (stronger base) and in this step the reaction takes place in one direction, unlike the previous steps, which are reversible. Namely, a pair of weaker acid (benzyl alcohol) and weaker base (benzoate) is formed, the equilibrium reaction is on the side of the product. Benzoate is separated from benzyl alcohol by extraction and converted into benzoic acid with the help of strong mineral acid. Synthesis results are given in table 1.

Table 1: Synthesis results

Sample	Synthesized	Yield
A	0.75	50.13
B	0.66	44.12
C	1.01	67.51
D	0.48	32.09
E	0.29	19.39

Melting point

In this work, we had two samples, where both showed satisfactory results according to the monograph from the European Pharmacopoeia. The requirement according to the European Pharmacopoeia is 121˚C-125˚C. The result for Sample 1 is 122˚C. The result for Sample 2 is 121.9˚C.

Assay of Benzoic acid- Volumetric determination of benzoic acid content

Sample	***m (g)***	***V (ml)***	***Vs-Vblank***	***%***
1	0.1004	7.6	7.6	99.16
2	0.1002	7.6	7.5	98.70
			AVG	**98.93**

Figure 4: Volumetric determination of benzoic acid content

Results of benzoic acid identification - IR technique

After preparing the KBr lozenge and recording the IR spectrum, the interpretation of the spectrum of the sample and the spectrum of the standard was started. Figure 5 shows the match between the spectra of the synthesized benzoic acid sample and the benzoic acid standard, result 100%

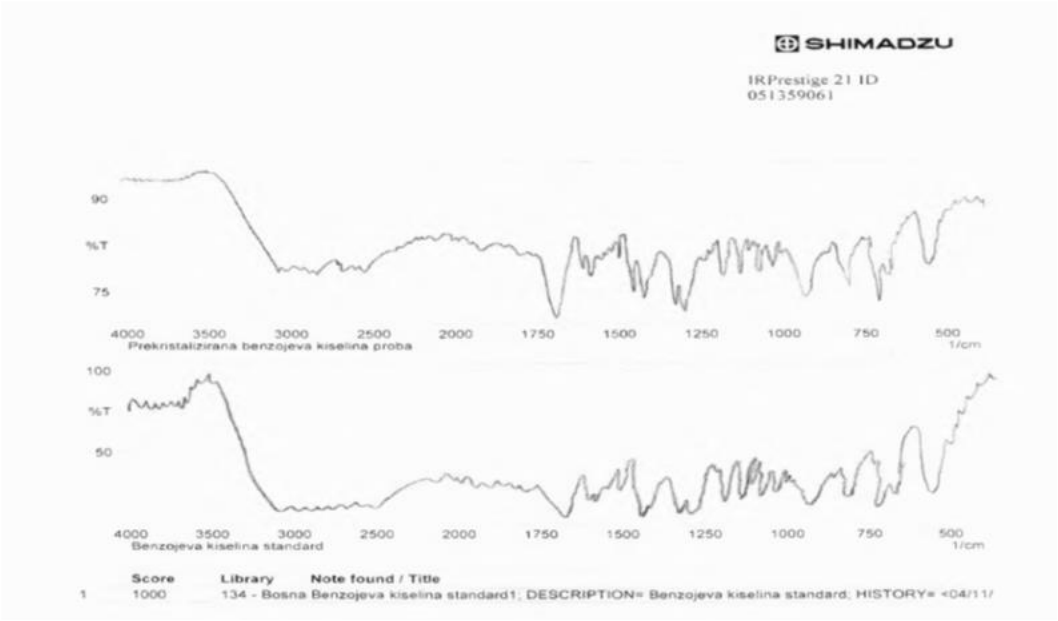

Figure 5: IR identification of benzoic acid

4. Conclusion

After the experimental work and the processing of the results, we can conclude that the tested sample of benzoic acid corresponds to the requirements prescribed by the European Pharmacopoeia, which in this paper includes the following: synthesis of benzoic acid, the test results showed that the amount of the sample, the yield results we calculated, correspond to the requirements; the results of determining the melting point correspond to the requirements of the benzoic acid monograph from the European Pharmacopoeia, determination of the content of benzoic acid, the test results showed that the sample of benzoic acid is of appropriate quality, that the amount of content corresponds to the requirements of the European Pharmacopoeia and through qualitative analysis, IR identification, we also confirmed the primary identification of the synthesized benzoic acid sample.

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