

ИССЛЕДОВАНИЕ ЭКСТРАКЦИИ СОРБИНОВОЙ И БЕНЗОЙНОЙ КИСЛОТ БЛОК-СОПОЛИМЕРОМ «ПЛУРОНИК»

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Экстракционные системы на основе водорастворимых полимеров представляют собой вариант «зеленой» экстракции, отличающийся применением безвредных, экологически безопасных реагентов. Актуальность работы обусловлена возрастающими требованиями к экстрагентам, способным обеспечить практически полное извлечение сорбиновой и бензойной кислот из пищевых объектов с целью их последующего количественного определения. Работа посвящена изучению экстрагирующей способности блок-сополимера «Плуроник» по отношению к сорбиновой и бензойной кислотам и установлению закономерностей межфазного распределения в исследованных системах. Неионогенные поверхностно-активные блок-сополимеры этиленоксида и пропиленоксида обеспечивают высокие количественные показатели межфазного распределения широкого класса органических веществ. В небольших количествах сорбиновая и бензойная кислоты в составе продуктов питания не оказывают негативного влияния на здоровье человека, но повышенное содержание консервантов вызывает патологии организма. Поэтому разработка новых методик извлечения и определения сорбиновой и бензойной кислот в водных средах с целью дальнейшего контроля их содержания в пищевых объектах является актуальной задачей аналитической химии. В статье приводятся рассчитанные коэффициенты распределения и степень извлечения сорбиновой и бензойной кислот с применением в качестве экстрагента блок-сополимера «Плуроник» и высаливателя сульфата аммония. Установлены концентрации аналитов и экстрагента, а также соотношение объемов водной и органической фаз, при которых достигаются максимальные экстракционные характеристики. В работе применен электрофоретический анализ водной фазы после экстракции, установлены параметры количественного определения сорбиновой и бензойной кислот. Предложены схемы взаимодействия блок-сополимера с аналитами за счет водородных связей, учитывающие особенности строения «Плуроника» и извлекаемых веществ.

Ключевые слова: сорбиновая и бензойная кислоты, экстракция, блок-сополимер, «Плуроник», капиллярный электрофорез

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STUDY OF EXTRACTION OF SORBIC AND BENZOIC ACIDS USING BLOCK COPOLYMER “PLURONIC”

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Extraction systems based on water-soluble polymers are a variant of “green” extraction, characterized by the use of harmless, environmentally friendly reagents. The relevance of the work is due to the increasing requirements for extractants capable of ensuring almost complete extraction of sorbic and benzoic acids from food objects for the purpose of their subsequent quantitative determination. The work is devoted to studying the extracting ability of the Pluronic block copolymer in relation to sorbic and benzoic acids and establishing the patterns of interphase distribution in the studied systems. Nonionic surfactant block copolymers of ethylene oxide and propylene oxide provide high quantitative indicators of interfacial distribution of a wide class of organic substances. In small quantities, sorbic and benzoic acid in food products do not have a negative effect on human health, but high levels of preservatives cause pathologies in the body. Therefore, the development of new methods for the extraction and determination of sorbic and benzoic acids in aqueous media with the aim of further monitoring their content in food objects is an urgent task of analytical chemistry. The article presents the calculated distribution coefficients and the degree of extraction of sorbic and benzoic acids using the Pluronic block copolymer and ammonium sulfate as an extractant. The concentrations of analytes and extractant, as well as the ratio of the volumes of aqueous and organic phases at which maximum extraction characteristics are achieved, have been established. Electrophoretic analysis of the aqueous phase after extraction is used in the work. Parameters for the quantitative determination of sorbic and benzoic acids were established. Schemes for the interaction of the block copolymer with analytes due to hydrogen bonds are proposed, taking into account the structural features of Pluronic and the extracted substances.

Key words: sorbic and benzoic acids, extraction, block copolymer, Pluronic, capillary electrophoresis

INTRODUCTION

The modern food market is almost impossible to imagine without the use of preservatives, thickeners, stabilizers, flavoring additives, and dyes. In small quantities, the listed substances do not have a negative effect on human health, while a significant part of manufacturers do not comply with established standards for controlling the content of additives in food products. Unregulated content of these components can cause not only allergic reactions, but also cause the development of chronic pathologies [1]. Benzoic acid and its salts are not always harmless to human health, even in the minimal quantities in which they are present in food products. In small quantities with frequent use,

this preservative can provoke allergic reactions [2]. If the permissible limit is exceeded, more critical consequences can occur: impairment of cognitive functions, neurotransmission, development of acidosis, convulsions and hyperpnea [3]. Sorbic acid and its derivatives are less toxic to humans, however, there are cases in which these substances initiated allergic reactions, including dermatitis [4]. When entering the human body in large quantities, food additives of this group can destroy vitamin B12, the deficiency of which can cause the development of anemia, as well as various neurological disorders [5].

The requirement for the content of preservatives is regulated by the Customs Union [6]. The maximum permissible concentration of sorbic and benzoic

acids and their salts in soft drinks is 300 mg/dm³ and 150 mg/dm³, respectively [7].

Currently, there are various instrumental methods for determining the components of food additives, including chromatographic, spectrophotometric and electrophoretic techniques [8]. Considering that most food products have a complex multicomponent matrix, there is a need for a preliminary sample preparation stage, which ensures the isolation and concentration of analytes. Liquid-liquid extraction is most widely used for these purposes, but the classic version of extraction requires large volumes of toxic solvents and is characterized by complexity and time-consuming performance [9].

Due to their unique physicochemical, structural and technological properties, water-soluble polymers are promising extractants for the extraction of organic compounds of various classes, including sorbic and benzoic acids, from aqueous solutions [10-12]. Such extractants are environmentally safe, non-toxic, and at the same time they provide high quantitative extraction rates. Among water-soluble polymers, a

special place is occupied by nonionic surface-active block copolymers of ethylene oxide (EO) and propylene oxide (PO) with the composition (EO)_x–(PO)_y–(EO)_x, produced under the Pluronic trademark [13,14]. These polymers are among the most popular modern auxiliary substances in various fields of chemistry and medicine. They are used as micellar containers for the delivery of drugs, artificial blood components, and compositions for the treatment of wounds and burns [15-17].

The purpose of this work is to study the interfacial distribution of benzoic and sorbic acids in extraction systems based on the Pluronic block copolymer.

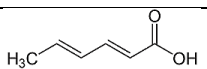
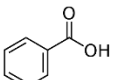
EXPERIMENTAL PROCEDURE

The reagents used in the work were “Sorbic acid” and “Benzoic acid” of chemically pure grade, some of the characteristics of which are given in Table 1. “Pluronic R-123” was chosen as the extractant, which is an alternating triblock copolymer, in the macro chain of which there are 2 blocks of 20 monomer units of ethylene oxide and a block of 70 units of propylene oxide separating them (Fig. 1).

Table 1

Some physicochemical characteristics of sorbic and benzoic acids

Таблица 1. Некоторые физико-химические характеристики сорбиновой и бензойной кислот

Substance	Structural formula	Molar mass, g/mol	Solubility in water, g/dm ³	T _{кип} , °C	pKa (at 25 °C)
Sorbic acid		112,13	0,16 (20 °C)	228	4,77
Benzoic acids		122,12	3,44 (25 °C)	250	4,21

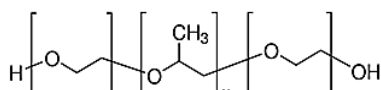


Fig. 1. Structure of the block copolymer "Pluronic"
Рис. 1. Структура блок-сополимера «Плуроник»

To conduct the experiment, a solution of sorbic and benzoic acids was prepared with concentrations of 0.001-0.025 mg/cm³ and 0.01-0.035 mg/cm³, respectively, in a saturated solution of ammonium sulfate, which ensures the most complete release of the polymer into a separate phase [11, 18]. The concentrations of the Pluronic block copolymer in aqueous solutions were 3-6 mg/cm³. 10 cm³ of sorbic and benzoic acid solutions and 2-4 cm³ of an aqueous polymer solution were placed in graduated test tubes with a capacity of 25 cm³. The separation time of the extraction system was no more than 5 min. To establish interfacial equilibrium and complete separation of the system, the extraction system was centrifuged at 5000 rpm for 10 min. The ratio of the volumes of equilibrium aqueous and

organic phases r ($r = V_B/V_o$) was measured.

The water-salt phase was separated from the organic phase and analyzed by capillary electrophoresis: voltage +20 kV, temperature 24±1 °C, water samples: hydrodynamic under a pressure of 30 mbar for 15 s [11, 19]. Two electropherograms of each portion of the prepared sample were recorded, and sorbic and benzoic acids in the sample were identified and determined. The determination of sorbic and benzoic acids is carried out at a wavelength of 254 nm, the leading electrolyte is sodium tetraborate, 10 mM, pH 9.2. Distribution coefficients (D) and the degree of recovery (R, %) of analytes from a water-salt solution were calculated using known equations [20].

RESULTS AND ITS DISCUSSION

Despite the fact that many methods are known for the determination of benzoic and sorbic acids in food, pharmaceutical and cosmetic products, the development of simple, highly effective sample preparation methods remains an urgent task. Extraction of analytes

with the Pluronic block copolymer is a variant of “green” chemistry and is a promising direction in the development of analytical chemistry [21-23].

In Table 2 and 3 present the results of a single extraction of sorbic and benzoic acids using the Pluronic R-123 block copolymer. For both acids, the highest degree of extraction (up to 96%) is achieved at an extractant concentration of 5 mg/cm³ and a ratio of aqueous and organic phases of 10:4. The concentration of sorbic acid is 0.010 mg/cm³, benzoic acid is 0.025 mg/cm³, increasing the concentrations of analytes does not lead to an increase in the degree of extraction.

Under these conditions, separate electrophoretic determination of the two acids was carried out. Let us note that the determination of benzoic and sorbic acids in alcoholic and non-alcoholic drinks by the method of capillary electrophoresis is regulated by GOST R 53193_2008 [24].

Table 2
Extraction characteristics of sorbic acid when extracted with Pluronic (n = 3, P = 0.95)

Таблица 2. Экстракционные характеристики сорбиновой кислоты при извлечении «Плуронином» (n = 3, P = 0,95)

Sorbic acid concentration, mg/cm ³	D	R, %
r = 10:2		
0.001	29±3	85.2
0.003	37±3	88.1
0.005	45±3	90.0
0.007	47 ±3	90.4
0.009	51±3	91.1
0.010	58±3	92.1
0.015	49±3	90.1
0.025	42±4	89.4
r = 10:3		
0.001	10±1	76.9
0.003	12±1	80.1
0.005	13±1	81.2
0.007	16±2	88.9
0.009	25±2	89.2
0.010	27±2	90.1
0.015	32±2	91.4
0.025	13±1	81.2
r = 10:4		
0.001	25±5	90.1
0.003	29±5	92.1
0.005	34±5	93.2
0.007	38±5	93.8
0.009	43±6	94.5
0.010	49±6	95.1
0.015	44±5	94.6
0.025	38±5	93.8

Table 3

Extraction characteristics of benzoic acid when extracted with Pluronic (n = 3, P = 0.95)

Таблица 3. Экстракционные характеристики бензойной кислоты при извлечении «Плуронином» (n = 3, P = 0,95)

Concentration of benzoic acid, mg/cm ³	D	R, %
r = 10:2		
0.01	84±6	94.4
0.015	88±6	94.6
0.02	80±6	94.1
0.025	93±7	95.1
0.03	89±7	95.4
0.035	87±7	95.3
r = 10:3		
0.01	23±2	87.4
0.015	28±2	89.5
0.02	25±2	88.3
0.025	41±4	92.6
0.03	43±3	92.9
0.035	35±3	91.4
r = 10:4		
0.01	51±3	95.3
0.015	53±4	95.5
0.02	56±3	95.7
0.025	61±4	96.1
0.03	49±3	95.1
0.035	47±3	94.9

Based on the obtained extraction results, it can be assumed that the interfacial interaction in the sorbic (benzoic) acid – “Pluronic” systems is carried out due to hydrogen bonds with the participation of carboxyl groups of acids (Fig. 2 and 3). In the main chain, ethylene oxide units can form hydrogen bonds with water molecules. The absence of side substituents makes Pluronic macromolecules flexible enough to interact with water, which is determined by the hydrophilicity of polyethylene oxide chains and the hydrophobicity of polypropylene oxide (due to the presence of methyl substituents) [14, 25, 26].

Pluronics, like most amphiphilic compounds, in aqueous solution at certain concentrations and temperatures form various supramolecular associates, incl. micelles. At low concentrations, pluronics exist in the form of unassociated molecules, which are ideal coils [14]. With increasing concentration of pluronic in the solution, micelles are formed, which are in a state of equilibrium with single molecules. In aqueous solutions, the molecules of block copolymers EO and PO aggregate into micelles, the core of which is formed by a polypropylene oxide block, and the crown is formed by hydrated polyethylene oxide blocks [16].

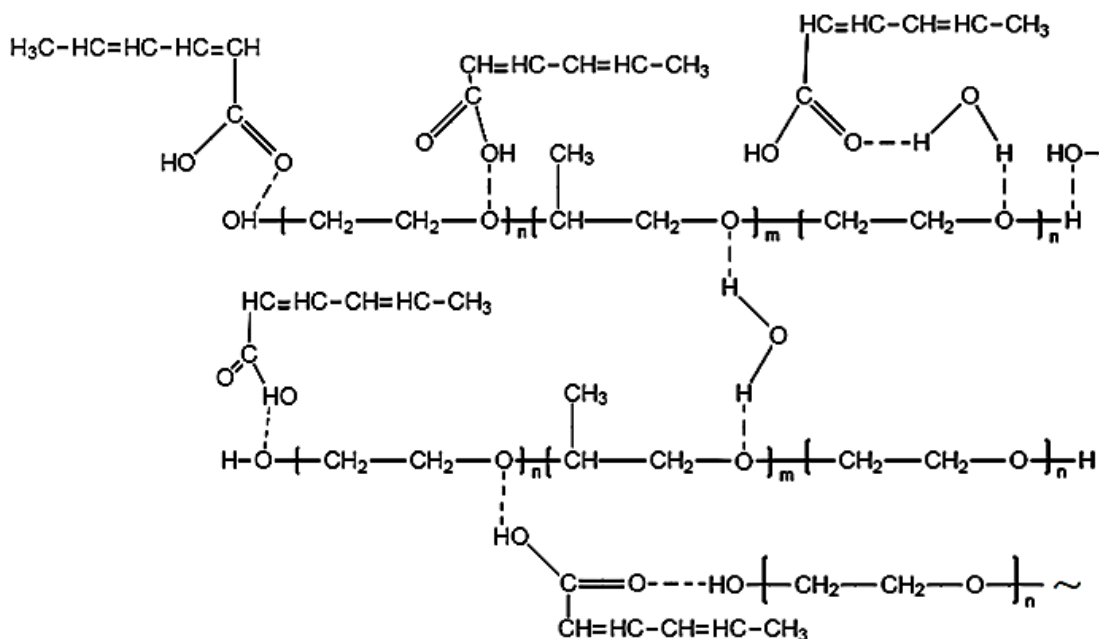


Fig. 2. Scheme of interaction in the sorbic acid – “Pluronic” system
 Рис. 2. Схема взаимодействия в системе сорбиновая кислота – «Плуроник»

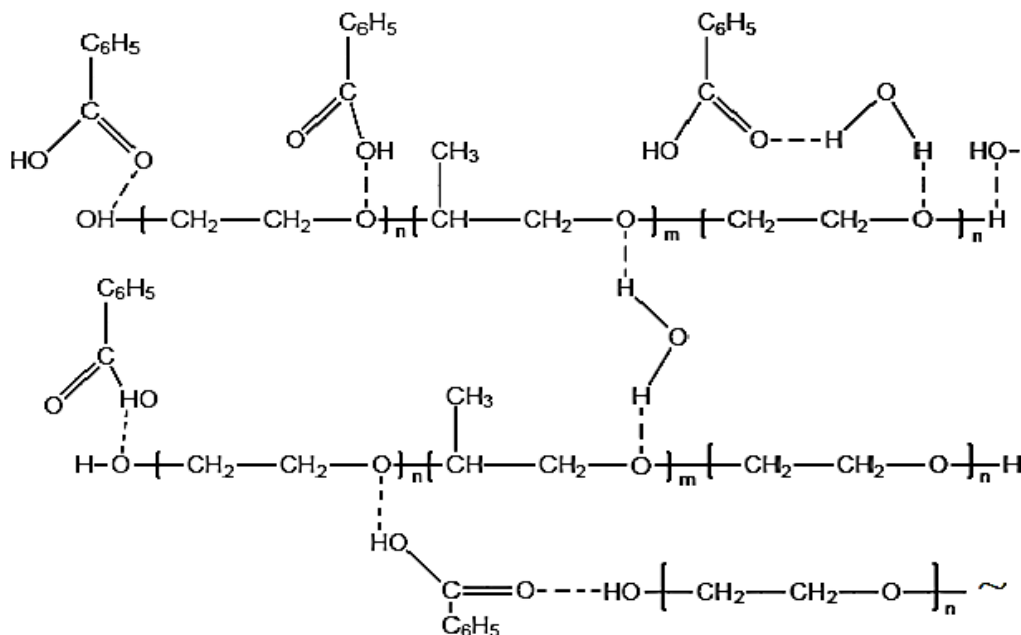


Fig. 3. Scheme of interaction in the benzoic acid – “Pluronic” system
 Рис. 3. Схема взаимодействия в системе бензойная кислота – «Плуроник»

CONCLUSIONS

The extracting ability of the block copolymer “Pluronic-R123” in relation to sorbic and benzoic acids has been studied in the work. The distribution coefficients and degree of recovery of analytes in extraction systems based on water-soluble polymer were calculated. The concentrations of “Pluronic” and acids, the ratio of phase volumes at which maximum extraction characteristics are achieved, have been established.

Based on the structure of the block copolymer, a mechanism of interfacial interaction in the Pluronic systems – sorbic (benzoic) acid – has been proposed. Determination of analytes after extraction from aqueous media was carried out by capillary electrophoresis.

The authors declare the absence a conflict of interest warranting disclosure in this article.

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