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## ANALYSIS OF PRESERVATIVES CONTENT IN FOOD

### ANALIZA ZAWARTOŚCI KONSERWANTÓW W PRODUKTACH SPOŻYWCZYCH

**Abstract:** The aim of this work was the determination of benzoic acid and sorbic acid contents in selected beverages and processed fruit and vegetable products. The analyses of these preservatives in 34 different food products (ketchups, carbonated and non-carbonated beverages, beverage concentrates) were performed. In this purpose, the conditions of analyzed compounds extraction from the food samples were optimized and method of analysis by high performance liquid chromatography method (HPLC) was developed.

**Keywords:** food preservatives, benzoic acid, benzoates, sorbic acid, sorbates, beverages, fruit and vegetable products

Majority of foods available in the market contain different types of additives, among which preservatives are playing an important role. These additives, applied in order to maintain food quality and prolong storage time, often make consumers anxious about its safety. The very popular preservatives are benzoic acid and sorbic acid, used mostly in the form of the well soluble sodium, potassium or calcium salts [1]. Analytical methods used for their determination are based mainly on UV spectrophotometry, gas chromatography (GC) and high performance liquid chromatography (HPLC) [2]. In many cases prior to the analysis sample preparation is required, in order to isolate the analytes from the complex food matrix. The aim of this work was the analysis of benzoic and sorbic acid content in selected beverages and processed fruit and vegetable products. In this purpose, the conditions of analyzed compounds extraction from the food samples were optimized and method of analysis by high performance liquid chromatography method (HPLC) was developed.

### Materials and methods

The analyses of benzoic and sorbic acid used as the preservatives in different food products available in the home market (ketchups, carbonated and non-carbonated beverages, beverage concentrates) were performed.

Analytes were extracted from the food matrix by chloroform, after previous salting out using sodium chloride solution. In order to optimize this step, the influence of different factors during sample preparation was studied (solvent volume, multiplication, pH, filtration). Finally, 2.5 g of ketchup or 15 cm<sup>3</sup> of beverage were placed in a measuring flask, filled to a 25 cm<sup>3</sup> volume with saturated sodium chloride solution and well mixed. Carbonated beverages were degassed previously by using water aspirator. Next, 20 cm<sup>3</sup> of obtained solution was placed in separatory funnel and extracted four times by using 5 cm<sup>3</sup> of chloroform. All extracts were connected and filled up to a 25 cm<sup>3</sup> volume with methanol. Before the analysis, samples were centrifuged.

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The analyses were performed by high performance liquid chromatography (HPLC) method. The reversed phase mode was applied, with non-polar octadecylsilane (C18) stationary phase and polar mobile phase with acetic buffer as part of the composition. LC-5B liquid chromatograph equipped with UV-VIS detector and glass column (150 x 3.3 mm I.D.) filled with SEPARON SGX C-18 (7 µm) stationary phase was used. The monitoring wavelength was 254 nm. The composition of eluent was optimized in order to achieve good separation of benzoic acid, sorbic acid and chloroform. Methanol - water - acetic buffer pH 4.4 (40:40:20) mobile phase was used with a flow rate of 0.5 cm<sup>3</sup>/min. The analyses were performed at room temperature.

The limits of detection in proposed method are 4.0 mg/dm<sup>3</sup> for benzoic acid and 0.12 mg/dm<sup>3</sup> for sorbic acid. These values are much below the permitted limits detailed for the individual foods, which are in most cases between 150÷1000 mg/dm<sup>3</sup> or mg/kg for benzoic acid and 200÷2000 mg/dm<sup>3</sup> or mg/kg for sorbic acid [3].

## Results and discussion

The performed analyses of 9 different types of ketchups (K) confirmed that in agreement with the producer's information, two of them were without preservatives. In the remaining 7 ketchups sodium salt of benzoic acid was applied. The level of benzoic acid in one of them (1776 mg/kg) was found to be high above the maximum allowed limit for this group of products (1000 mg/kg). The content of benzoic acid in other samples ranged from 226 to 1009 mg/kg (Table 1).

Preservatives contents in ketchups (K)

Table 1

Product	Benzoic acid [mg/kg]	Sorbic acid [mg/kg]	Summary content (benzoic + sorbic acid) [mg/kg]
K-1	416	-	416
K-2	941	-	941
K-3	226	-	226
K-4	1776	-	1776
K-5	660	-	660
K-6	1009	-	1009
K-7	695	-	695
K-8	-	-	-
K-9	-	-	-

The results of the analyses of the non-carbonated (NCB) and carbonated beverages (CB) are collected in Tables 2 and 3, respectively. All analyzed non-carbonated beverages contained benzoic acid in the range of 73÷174 mg/dm<sup>3</sup>, and additionally in two samples sorbic acid (27 and 30 mg/dm<sup>3</sup>) was found. Most of the analysed carbonated beverages contained benzoic acid (16÷406 mg/dm<sup>3</sup>) or its mixture with sorbic acid (73÷169 mg/dm<sup>3</sup>), in one sample only sorbic acid was applied (169 mg/dm<sup>3</sup>). The amount of benzoic acid in two samples was above the maximum allowed limit for beverages - 150 mg/dm<sup>3</sup>. The limit for sorbic acid in beverages was set at 300 mg/dm<sup>3</sup> when it is used separately or 250 mg/dm<sup>3</sup> when it is applied together with benzoic acid. These values were not exceeded in none of the analysed samples of beverages.

Table 2

Preservatives contents in non-carbonated beverages (NCB)

Product	Benzoic acid [mg/dm <sup>3</sup> ]	Sorbic acid [mg/dm <sup>3</sup> ]	Summary content (benzoic + sorbic acid) [mg/dm <sup>3</sup> ]
NCB-1	142	-	142
NCB-2	73	27	100
NCB-3	73	30	103
NCB-4	141	-	141
NCB-5	128	-	128
NCB-6	174	-	174
NCB-7	132	-	132

Table 3

Preservatives contents in carbonated beverages (CB)

Product	Benzoic acid [mg/dm <sup>3</sup> ]	Sorbic acid [mg/dm <sup>3</sup> ]	Summary content (benzoic + sorbic acid) [mg/dm <sup>3</sup> ]
CB-1	149	84	233
CB-2	133	73	206
CB-3	136	-	136
CB-4	92	-	92
CB-5	117	-	117
CB-6	138	-	138
CB-7	78	-	78
CB-8	132	-	132
CB-9	123	-	123
CB-10	129	-	129
CB-11	-	169	169
CB-12	93	88	181
CB-13	16	169	185
CB-14	406	-	406

The analyses of selected beverage concentrates (BC) showed that calculated summary content of benzoic and sorbic acid in these products (after dilution according to the producer's recommendation) ranges between 76 and 189 mg/dm<sup>3</sup>. These values, collected in Table 4, are below the maximum summary content limit of these two acids - 600 mg/dm<sup>3</sup>.

Table 4

Preservatives contents in beverage concentrates (BC)

Product	Benzoic acid [mg/dm <sup>3</sup> ]	Sorbic acid [mg/dm <sup>3</sup> ]	Summary content (benzoic + sorbic acid) [mg/dm <sup>3</sup> ]	Recommended dilution of product (v:v)	Summary content (benzoic + sorbic acid) in diluted product [mg/dm <sup>3</sup> ]
BC-1	919	-	919	1:10	92
BC-2	857	-	857	1:10	86
BC-3	-	304	304	1:4	76
BC-4	166	588	754	1:4	189

The obtained data indicate that the preservatives content in foods exceeded the permitted maximum levels only accidentally.

## Conclusions

The analyses of benzoic and sorbic acid content in 34 different food products (ketchups, carbonated and non-carbonated beverages, beverage concentrates) were performed by high performance liquid chromatography. The proposed method enables the simultaneous and selective analysis of these popular preservatives in different types of products.

On the basis of the obtained results it was found that the content of the analyzed preservatives in most cases was below the permitted level. In one ketchup sample and two beverages samples, amount of benzoic acid significantly exceeded the allowed values.

## References

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## ANALIZA ZAWARTOŚCI KONSERWANTÓW W PRODUKTACH SPOŻYWCZYCH

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**Abstrakt:** Przedmiotem badań była analiza zawartości kwasu benzoowego oraz kwasu sorbowego w wybranych napojach i produktach owocowo-warzywnych. Oznaczono zawartość tych konserwantów w 34 artykułach żywnościowych: ketchupach, napojach gazowanych i niegazowanych oraz zaprawach do napojów. W tym celu dobrano warunki ekstrakcji oznaczanych związków z próbek artykułów spożywczych oraz opracowano metodę ich analizy za pomocą wysokosprawnej chromatografii cieczowej (HPLC).

**Słowa kluczowe:** konserwanty, kwas benzoowy, benzoiany, kwas sorbowy, sorbiniany, napoje, produkty owocowo-warzywne