



Test systems and a method for express detection of synthetic food dyes in drinks



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ARTICLE INFO

Article history:

Received 31 May 2012

Received in revised form

23 March 2014

Accepted 26 March 2014

Available online 3 April 2014

Keywords:

Synthetic dyes

Drinks

Adulteration

Adsorption

Spectrophotometry

ABSTRACT

In this study, new test systems and express method were developed for the rapid detection of synthetic food dyes in drinks including alcoholic drinks, fruit juices, soft drinks and other non-alcoholic drinks. The detection is based on selective adsorption of synthetic food dyes from a drink by the specific adsorbent contained in the developed test system. The method allows screening of samples for synthetic food dyes in the presence of natural colors by differential spectrophotometry following adsorption. The said adsorbent provides adsorption of synthetic food dyes and substantially does not interact with natural dyes of drinks. Therefore, when a drink contains synthetic dyes, the color of the sample changes, whereas the color of a drink having only natural dyes stays substantially the same. This test can be used as a first step analysis for screening. So, only in a case when the test showed the presence of synthetic dyes in a drink, further laborious identification and quantitative determination of the revealed dyes could be performed. The use of such an approach reduces the time and costs required to perform the analysis.

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1. Introduction

Quality control of drinks has a significant value, especially today when recent technologies provide manufacturing of drinks using a large amount of different cost-effective artificial ingredients.

The use of these artificial ingredients causes a lot of adulteration issues. For example, such popular drinks as wines and juices are often adulterated using synthetic food dyes instead of producing a drink using natural raw materials. It should be noted that the presence of synthetic dyes not only indicates the adulteration, but also shows potential health risks (Kobylewski & Jacobson, 2010). Recent studies show that such effects as allergy, asthma, hyperactivity and even cancer can be linked with the intake of synthetic food dyes (Hashem, Atta, Arbid, Nada, & Asaad, 2010; Kobylewski & Jacobson, 2010; Tripathi, Khanna, & Das, 2007). Therefore, strict control of synthetic food dyes in drinks that are positioned to be natural is very important.

Among the most popular methods that have been used for detection of synthetic food dyes are thin layer chromatography, high performance liquid chromatography, capillary electrophoresis and nuclear magnetic resonance methods (Socaciu, 2008). Many

studies are directed to improve these methods in order to simplify the detection procedure (Alves, Brum, de Andrade, & Netto, 2008; Baranowska, Zydron, & Szczepanik, 2004; Minioti, Sakellariou, & Thomaidis, 2007; Kartsova et al., 2009; Patsovskii, Rudometova, & Kamentsev, 2004; Serdar & Knežević, 2009; Tripathi et al., 2007; Tuzimski, 2011). Some of these methods are standardized in different countries for use in official control of synthetic dyes.

Nevertheless, the said methods still have some challenges in implementation related to the laborious sample preparation, high costs of the equipment and use of the toxic chemicals. In some cases, these challenges are reasonable, especially when the precise quantitative analysis of a drink is needed. However, it is still difficult for a consumer to differentiate the natural drink from the low quality synthetic one.

It is therefore highly important to develop test systems that are simple in use as well as to provide a method that can easily show the presence or absence of synthetic dyes in a drink. Having such a screening method, further precise identification and quantitative analysis using labor-consuming standard methods could be performed only for the samples where the synthetic dyes presence was detected. Thus, the expenses needed for the analysis could be significantly reduced.

Based on the aforesaid, the goal of our research was to develop novel test systems based on the selective adsorption of synthetic dyes. We proposed the detection method to include mixing of a

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drink with the specifically developed adsorbent in the test system and comparing the color characteristics of the initial drink and the supernatant after adsorption. In order to correlate the differences in color characteristics with the presence or absence of synthetic dyes in a drink, the special Index was intended to be developed.

2. Materials and methods

2.1. Synthetic food dyes

In this study, the following standard sulfonated synthetic dyes were used: Tartrazine (E102), Chinoline Yellow (E104), Sunset Yellow FCF (E110), Carmoisine (E122), Amaranth (E123), Ponceau 4R (E124), Erythrosine (E127), Red 2G (E128), Allura Red AC (E129), Indigo Carmine (E132) and Brilliant blue FCF (E133). These synthetic dyes having major dye content of at least 85 g/100 g were produced by Roha Dyechem Pvt. Ltd. (Mumbai, India) and Sensient Colors UK Ltd. (King's Lynn, UK).

2.2. Drinks

More than 200 samples of different drinks comprising wines, juices, cocktails and models thereof were used in the research. Among the drinks studied were red dry wine Cabernet Sauvignon (Barton & Guestier, Blanquefort, France), red dry wine "Chianti Ruffino" (Ruffino, Pontassieve, Italy), homemade red dry wine "Isabella", red semi-dry wine "La Bifora" (Toskovini S.P.A., Brescia, Italy), red semi-sweet wines "El Paso. Cabernet" and "El Paso. Merlot" (Igristie Vina, Saint Petersburg, Russia), red dry wine "Bodegas Nobles" (Exvima Exportación S.L., Chinchilla de Monte-Aragón, Spain), red dry wine "Beaujolais. Jean Deville" (Jean Deville, Romaneche-Thorins, France), cherry nectar "NICO" (Multon, Saint Petersburg, Russia), low-alcohol cocktail "Blazer" (Rudo-Akva, Ryazan, Russia). Additionally, the drinks involved in the research included drink samples given for testing and identification referred according to their labeling codes, e.g., "Wine sample 1", "Juice sample 1", etc.

2.3. Adsorbent Riosorb

Adsorbent Riosorb was specially developed in our previous study to provide high adsorption activity towards synthetic food dyes and low adsorption activity towards natural drink dyes (Komissarchik & Nyanikova, 2010). Riosorb was prepared as a chitosan containing adsorbent from the biomass of Mucorales fungi.

2.4. Model solutions preparation

Model solutions of the above listed synthetic dyes were prepared by dissolving a predetermined amount of a dye in water, water-ethanol solutions, in natural apple juice, in dry white wine Punto Nino Chardonnay (Laroche Vina Punto Alto, Casablanca Valley, Chile), sweet white wine Cavic (RPB SOCIEDAD ANONIMA, Maipú, Argentina) or in the above-identified red wines. Optionally, the natural Caramel colorant E150 was added to the solutions in order to adjust the values of color intensity and tint to those of the natural drinks since the selective adsorption was proposed to be evaluated in samples with similar initial color intensity and tint. The amounts of dyes were recalculated based on the major dye content.

2.5. Synthetic dyes adsorption

Adsorption was performed in the test systems, the test systems being plastic 12 ml test tubes comprising the predetermined

amount of the adsorbent. A drink sample of 10 ml was poured into the test system, the test tube was closed and the mixture was thoroughly shaken on a Laboratory shaker type 358S (Elpan, Lubawa, Poland) at room temperature. Then the supernatant was separated by 5 min centrifugation at $4185 \times g$ and used for the further color measurement.

2.6. Color characteristics measurement

The color characteristics selected for use in the analysis were color intensity (I) and tint (T) measured according to the following formulas as set forth in the Commission Regulation (EEC) No 2676/90:

$$I = (D_{420} + D_{520} + D_{620})/L \quad (1)$$

$$T = D_{420}/D_{520} \quad (2)$$

where D_{420} , D_{520} and D_{620} are the optical densities of a sample measured at the wavelengths of 420 nm, 520 nm and 620 nm, respectively; L is optical path length in the sample, cm.

The Synthetic Dyes Index was calculated as:

$$SDI = \left| \frac{I_0 - I}{I_0} \cdot \frac{T_0 - T}{T_0} \right| \cdot 100 \quad (3)$$

where I_0 and T_0 are the color intensity and the tint of an initial drink, respectively; I and T are the color intensity and the tint of the supernatant after adsorption.

2.7. Sample preparation for the identification and quantitative analysis of synthetic food dyes

The sample preparation was performed in accordance with the State standard of Russia – GOST R (GOST R 52470-2005, 2006) as follows. To 50 ml of a drink 1 ml of glacial acetic acid was added. The mixture was heated in a water bath at 80–90 °C for 5 min and then cooled to the room temperature. The mixture was passed through the solid phase extraction cartridge filled with 500 mg of Al_2O_3 (activated, acidic, Brockmann I, Sigma Aldrich Co. LLC, St. Louis, MO, USA) pre-treated with 25 ml of glacial acetic acid. The desorption was performed by eluting with 25 ml/100 ml aqueous ammonia solution until the full discoloration of the adsorbent in a cartridge was achieved. The eluate was collected and evaporated to dryness in a water bath at 80–90 °C, cooled and dissolved in water to give the sample solution for further analysis.

2.8. Thin layer chromatography conditions

The TLC analyses of drinks were performed in accordance with the State standard of Russia – GOST R (GOST R 52470-2005, 2006). 0.5 μ L of a sample prepared using the procedure as described in 6.2 was spotted on the Silufol plate (10 cm \times 10 cm). The mobile phase was prepared of pyridine: isoamyl alcohol: isobutanol: ethanol: 25 ml/100 ml aqueous ammonia in ratio 3:3:3:4:4 (v/v). For the identification, R_f values were compared with that of the standard dyes obtained in the same conditions.

2.9. Capillary electrophoresis conditions

The analysis by capillary electrophoresis method was performed on CAPEL[®]-105 instrument equipped with a spectrophotometric detector (produced by Lumex LLC, Saint Petersburg, Russia). A quartz capillary was used, the capillary having the following characteristics: internal diameter of 75 μ m, length of 60 cm, effective

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