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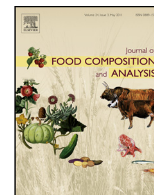

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2 Original Research Article

3 Determination of furfural and hydroxymethyl furfural from baby 4 formula using dispersive liquid–liquid microextraction coupled with 5 high performance liquid chromatography and method optimization by 6 response surface methodology

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ABSTRACT

A simple, rapid and efficient method was developed using dispersive liquid–liquid microextraction (DLLME) coupled with high performance liquid chromatography (HPLC) for extraction and determination of furfural (*F*) and hydroxymethyl furfural (HMF) in baby formula. The effects of different variables on the extraction efficiency such as the volume of extraction and disperser solvents, pH and salt effect were studied and optimized simultaneously using (RSM) based on central composite design (CCD). Under optimum conditions, a mixture of ethanol (disperser solvent) and 1-octanol (extraction solvent) was rapidly injected in to the sample solution (after adding 2 g salt and adjusting pH to 6.5). Limit of detection for *F* and HMF were 0.7 and 1.8 ng g^{-1}, respectively. The inter-day relative standard deviation (RSD%) were 4.9 for HMF and 3.9 for *F* and also inter-day RSD% were in the range of 5.2–8%. The results showed that DLLME-HPLC is a very fast, simple, sensitive and accurate analytical method for the determination of *F* and HMF in baby formulas. Finally, The ability of the proposed method to determine *F* and HMF in different baby formulas in Iran was studied and suitable result was obtained.
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12 1. Introduction

13 Q2 Heat treatment is common way for preserving foods and 14 making them edible. Any heat treatment going beyond the 15 requisite can modify the food constituents and thus adversely 16 affect the taste and nutritional value (Crews and Castle, 2007). One 17 reaction that takes place in foods during heating and storage and 18 has an important influence on the appearance and taste of foods is 19 Maillard (Antonelli et al., 2004; Guerra-Hernández et al., 2002; 20 Habibi et al., 2012). Baby formulas are enriched with compounds 21 (vitamin A, iron, and lactose) that increase their susceptibility to the Maillard reaction (Ferrer et al., 2002). The Maillard reaction 22 induces formation of the furanic compounds furfural (*F*) and 23 hydroxymethyl furfural (HMF) during heating. Some studies have 24 postulated various sources and mechanisms for *F* and HMF 25 formation (Ramírez-Jiménez et al., 2000). Several possible origins Q3 26 are thermal degradation of carbohydrates or reaction between 27 reducing sugar and amino acids (Maillard reaction), thermal 28 oxidation of poly-unsaturated fatty acids, and thermal decompo- 29 sition of ascorbic acid and its derivatives (Becalski and Seaman, 30 2005; Märk et al., 2006; Crews and Castle, 2007; Limacher et al., 31 2007; Perez Locas and Yaylayan, 2004). The contents of undesir- 32 able compounds such as *F* generated at advanced stages of the 33 Maillard reaction are used to evaluate the intensity of the thermal 34 treatment in baby formula (Ferrer et al., 2002, 2005). Another 35 compound is HMF, a known indicator for the loss of quality 36

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because of excessive heat treatment or storage of sugar-containing foods (Wagner and Beil-Seidler, 2007).

Up to now, many methods such as HPLC (Albalá-Hurtado et al., 1997; Gökmen and Senyuva, 2006; Liu et al., 2012), gas chromatography (GC) (Teixidó et al., 2006), spectroscopy (Motiyenko et al., 2007), micellar electrokinetic capillary chromatography (Morales and Jiménez-Pérez, 2001) and colorimetric methods (Porretta and Sandei, 1991) have been used for analysis of *F* and HMF. Among these techniques, HPLC has been widely used to determine *F* and HMF since this technique does not require derivatization step.

In the analysis of samples containing complex tissue, sample preparation is a critical step in the overall analytical process. Applying this stage considerably decreases interference co-extraction species and enhances the extraction efficiency. We used primary extraction with NaOH solution for extraction of HMF and *F* from baby formula. In this step, the target analytes (*F* and HMF) release from matrix sample and come to extraction solvent. After this step, clean-up and preconcentration for determination of trace amount of *F* and HMF are necessary (Akpınar et al., 2011; Rezaee et al., 2010). Conventional LLE (Teixidó et al., 2008) and solid-phase extraction (SPE) were used for the clean-up and concentration of *F* and HMF in different solid samples (Teixidó et al., 2006). LLE is time-consuming, expensive and hazardous to health due to the high volume of toxic solvents used. SPE needs less solvent, but is still time-consuming, and often requires a concentration stage that presents disadvantages such as losses in the evaporation step, risk of contamination and loss of sensitivity.

Microextraction techniques have been characterized as a promising basis for a new generation of sample preparation techniques and have recently received much attention (Ghambarian et al., 2013; Seidi et al., 2013). Microextraction techniques have many advantages such as simplicity of operation, rapidity, low cost, high recovery, high enrichment factor and wide application prospects in trace analysis (Arvand et al., 2013; Campillo et al., 2012; Chaichi et al., 2012; Enteshari et al., 2013; Ong et al., 2008). Headspace solid-phase microextraction (HS-SPME) (Bononi and Tateo, 2009; Giordano et al., 2003; Ozolina et al., 2011; Petisca et al., 2013), liquid phase microextraction (HS-LPME) (Abu-Bakar et al., 2014), have been used in the study of *F* and HMF in different foods. In 2006, a microextraction technique named dispersive liquid–liquid microextraction (DLLME) was developed by Assadi and co-workers (Rezaee et al., 2006). DLLME is a miniaturized liquid–liquid extraction (LLE) that uses microliter volumes of the extraction solvent. For DLLME, water-immiscible extraction solvent dissolved in a water miscible dispersive solvent is rapidly injected into the aqueous solution by syringe (Andruch et al., 2012; Chiang and Huang, 2008; Fattahi et al., 2007; Kokya et al., 2012; Ramezani et al., 2014; Rezaei et al., 2008; Wu et al., 2009, 2011; Zang et al., 2009). This technique is distinguished by minor extractant phase consumption, high potential to pre-concentrate target analytes and ease of use. Also this method has been successfully employed for the analysis of complex matrix such as fruit juice samples, beverage and smoked fish (Biparva et al., 2012; Kamankesh et al., 2013; Ghasemzadeh-Mohammadi et al., 2012).

In the present study, sensitive and efficient analytical method (DLLME) followed by HPLC-UV was applied for extraction and determination of *F* and HMF from baby formula. The extraction was carried out in a binary system composed of extraction solvents (1-octanol and ethanol) and the sample solution. Several experimental parameters influencing the extraction performance of the proposed method were investigated and optimized by CCD. The proposed method was successfully applied to the analysis of trace amount of *F* and HMF in baby formula and suitable results were obtained.

2. Materials and methods

2.1. Reagent, material and standards

Furfural and hydroxymethyl furfural were purchased from Sigma–Aldrich (Steinheim, Germany) at purity higher than 99%. L-Octanol, ethanol, acetic acid, ammonium phosphate, and sodium chloride (analytical grade), sodium acetate, glacial acetic acid, acetonitrile, potassium hexacyanoferrate, zinc acetate dehydrate and water (HPLC-grade) were obtained from Merck (Darmstadt, Germany). For preparation of carrez solution I, 10.6 g of potassium hexacyanoferrate was dissolved in 100 mL distilled water. Carrez solution II was prepared with mixing of 21.9 g of zinc acetate with 3 mL of acetic acid, and then the volume was adjusted to 100 mL by distilled water. Stock standard solutions of *F* and HMF were prepared at a concentration of 2000 $\mu g\ mL^{-1}$ in HPLC grade methanol. Mixed standard solution was made at concentration of 100 $\mu g\ mL^{-1}$. Working solutions (1–200 $ng\ mL^{-1}$) were prepared by diluting stock solution with double distilled water for linear range assay. Stock and working solutions were refrigerated at 4 °C. The addition of standard solution is carried out in the tested samples due to the evaluation of relative recovery and accuracy. All solvents were analytical reagent grade or HPLC grade. The pH of the solutions was adjusted by hydrochloric acid (2 $mol\ L^{-1}$).

2.2. Instrumentation

The chromatographic analysis was carried out with a Cecil CE-4900 HPLC (Cambridge, UK) equipped as follows: two CE-4100 pumps, multiple solvent delivery unit, vacuum degasser, mixing chamber, six-port valve (Rheodyne, USA), CE-4200 UV-Vis detector (Cambridge, UK). An ODS column (250 mm × 4.6 mm, 5 μm) from Phenomenex (Torrance, CA, USA) was used for separation of the analytes. The injection volume was 20 μL, and the column temperature was 25 °C (ambient temperature). Separation of *F* and HMF was achieved using an acetate buffer (0.2 $mol\ L^{-1}$, pH = 3): acetonitrile (85:15) as mobile phase at a flow rate of 0.8 $mL\ min^{-1}$. The effluent was monitored at 284 nm for both *F* and HMF.

2.3. Sample preparation

5 g of milk powder was mixed with 5 mL of distilled water in conical flask and spiked with 100 μL of HMF and *F* standard (50 $ng\ g^{-1}$) by the standard addition method. Then 100 μL of standard solution was added and this mixture was thoroughly stirred to obtain a very homogeneous sample for 2 min. 2 g of this sample was weighted and transferred to the another conical flask and 9 mL of NaOH (0.01 $mol\ L^{-1}$) was added to hydrolyze and saponify the sample. In this step, pH adjusted to 3 and 1 mL carrez solution I and 1 mL carrez solution II were added to the sample solution to precipitate the protein (Ghasemzadeh-Mohammadi et al., 2012). After shaking (for 2 min), the closed container was centrifuged for 5 min in 4000 rpm and the upper aqueous phase was separated and filtered. Finally, 9 mL of sample solution was transferred to another conical flask (after adding 2 g salt and adjusting pH to 6.5) where the DLLME process was performed.

2.4. Dispersive liquid–liquid microextraction (DLLME)

A solution consisting of 650 μL of ethanol (disperser solvent) and 60 μL of 1-octanol (extraction solvent) was rapidly injected into the 9 mL of sample solution. The mixture was thoroughly shaken using a flat shaker for 2 min. In this step, *F* and HMF were extracted into fine droplets of 1-octanol and the solution become cloudy. The produced cloudy solution was centrifuged for 2 min at

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