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Improved Method for the Determination of Hydroxymethylfurfural in Baby Foods Using Liquid Chromatography−Mass Spectrometry

VURAL GÖKMEN*,† AND HAMIDE Z. ŞENYUVA§

Department of Food Engineering, Hacettepe University, 06532 Beytepe, Ankara, Turkey, and Ankara Test and Analysis Laboratory, Scientific and Technical Research Council of Turkey, Ankara 06330, Turkey

An improved analytical method for the rapid, reliable, and sensitive determination of hydroxymethylfurfural (HMF) in baby foods is described. It entailed aqueous extraction from food matrix with simultaneous clarification using Carrez I and II reagents, solid-phase extraction cleanup using Oasis HLB, and analysis by liquid chromatography−mass spectrometry. A narrow-bore column allowed fast chromatographic separation with good resolution of HMF and matrix coextractives. In positive atmospheric pressure chemical ionization conditions, precursor and compound-specific ions were sensitively detected in selected ion monitoring mode. Sample preparation with efficient cleanup followed by fast chromatographic analysis allowed the analysis to be completed in <20 min. Recovery ranged between 91.8 and 94.7% for spiking levels of 0.25, 1.0, and 5.0 mg/kg HMF in cereal-based baby foods. The method was shown to be successful when using liquid chromatography coupled to ultraviolet detection at 285 nm.

KEYWORDS: Hydroxymethylfurfural; HMF; LC-MS; SPE cleanup; baby foods

INTRODUCTION

Baby foods available in the market are mainly based on milk, fruit, and cereal and combine a set of factors that makes them highly sensitive to Maillard reactions. During the manufacture of baby foods, severe heat treatment can be applied; moreover, these products are usually stored for long periods of time. These processes may cause damage to constituents and decrease in the nutritional value. The quality of baby foods is of considerable importance, because babies may be obtaining all of their nutrients from a small number of foods; therefore, overprocessing may affect the nutritional status adversely.

Chemical indicators for assessing the quality of overprocessed foods have proved to be useful. Hydroxymethylfurfural (HMF) is recognized as an indicator of quality deterioration in a wide range of foods. It is formed as an intermediate in the Maillard reaction (*1*, *2*) and is also formed during acid-catalyzed dehydration of hexoses (*3*). Formation of HMF in foods is especially dependent on temperature and pH (*4*).

Several high-performance liquid chromatography (HPLC) techniques have been reported for the determination of HMF in various foods (*5*−*13*). These techniques utilize ultraviolet (UV) detection because of the strong absorption of furfurals at ∼280−285 nm. However, many compounds naturally present or formed in foods during processing may also absorb at this wavelength. Poor chromatographic resolution of these compounds may adversely affect the quantification of HMF during UV detection.

In recent years, the presence of HMF in foods has raised toxicological concerns: the compound and its similar derivatives (5-chloromethyl- and 5-sulfidemethylfurfural) have been shown to have cytotoxic (*14*), genotoxic (*15*), and tumoral effects (*16*, *17*). However, further studies suggest that HMF does not pose a serious health risk (*18*), but the subject is still a matter of debate.

In this study, a wide variety of processed baby foods including breads, biscuits, and adopted and follow-on formulas, as well as fruited fresh cheeses and yogurts, were analyzed for HMF using an improved liquid chromatography−mass spectrometry (LC-MS) method. The method entailed aqueous extraction of HMF, solid-phase extraction (SPE) cleanup, and analysis by LC-MS. The separation was performed on a narrow-bore column to shorten the chromatographic run.

EXPERIMENTAL METHODS

Chemicals and Consumables. Methanol, potassium hexacyanoferrate, and zinc sulfate were of analytical grade and obtained from Merck (Darmstadt, Germany). Ultrapure water was used throughout the experiments (Milli-Q system, Millipore, Bedford, MA). Oasis HLB (1 mL, 30 mg) SPE cartridges were supplied by Waters (Milford, MA). The analytical column (ZORBAX Bonus-RP, Narrow Bore RR 2.1 × 100 mm 3.5-μm) was supplied by Agilent Technologies (Wilmington, DE).

* Corresponding author (e-mail vgokmen@hacettepe.edu.tr).
† Hacettepe University.
§ Scientific and Technical Research Council of Turkey.

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Standards. Stock solution of HMF was prepared at a concentration of 1.0 mg/mL by dissolving crystalline standard in distilled water. Working standards were prepared daily by diluting the stock solution to concentrations of 0.05, 0.10, 0.25, 0.50, 1.0, 1.5, and 2.0 μg/mL with distilled water. Carrez I solution was prepared by dissolving 15 g of potassium hexacyanoferrate in 100 mL of water, and Carrez II solution was prepared by dissolving 30 g of zinc sulfate in 100 mL of water.

Sample Preparation. Depending on the sample matrix, the sample was ground (by using a mill, mesh size of 2 mm) or minced (by mincer reducing size). Subsamples of the homogenate were stored at −20 °C in high-density polyethylene bottles with plastic screw-capped lids.

Finely ground sample (1 g) was weighed into a 10 mL glass centrifuge tube with cap. A total of 100 μL of Carrez 1 and 100 μL of Carrez 2 solutions was added, and the volume was completed to 10 mL with 0.2 mM acetic acid. HMF was extracted by mixing the tube for 3 min using a vortex mixer. It was then centrifuged for 10 min at 5000 rpm, at 0 °C. The clear supernatant was cleaned up further by using an Oasis HLB SPE cartridge. Prior to use, the SPE cartridge was conditioned by passing 1 mL of methanol and equilibrated by passing 1 mL of water at a flow rate of approximately two drops per second using a plastic syringe of 2 mL. The excess of water was removed from the cartridge by passing 2 mL of air. One milliliter of aqueous extract was eluted through the preconditioned cartridge at a flow rate of approximately one drop per second using a plastic syringe, and the eluate was discarded. The cartridge was washed by passing 0.5 mL of water. Then the cartridge was dried under a gentle stream of nitrogen. HMF was eluted from the cartridge by passing 0.5 mL of diethyl ether at a flow rate of approximately one drop per second using a plastic syringe of 2 mL. The eluate was collected in a conical bottom glass test tube placed in a water bath at 40 °C (Zymark Turbo Vap LV evaporator) and evaporated to dryness under nitrogen at 3 psig. The remaining residue was immediately redissolved in 1 mL of water by mixing in a vortex mixer for 1 min. Twenty microliters of this test solution was injected onto the HPLC system.

LC-MS Analysis. The quantification of HMF was performed by an Agilent 1100 HPLC system (Waldbronn, Germany) consisting of a binary pump, an autosampler, a diode array detector, and a temperature-controlled column oven, coupled to an Agilent 1100 MS detector equipped with an atmospheric pressure chemical ionization (APCI) interface. The chromatographic separations were performed on an Zorbax Bonus RP Narrow Bore column (2.1 × 100 mm, 3.5 μm) using the isocratic mixture of 0.01 mM acetic acid in 0.2% aqueous solution of formic acid at a flow rate of 0.2 mL/min at 40 °C. Data acquisition was performed in selected ion monitoring (SIM) mode using the following interface parameters: drying gas (N_2, 100 psig), flow of 4 L/min; nebulizer pressure, 60 psig; drying gas temperature, 325 °C; vaporizer temperature, 425 °C; capillary voltage, 4 kV; corona current, 4 μA; fragmentor voltage, 55 eV; dwell time, 439 ms. Ions monitored for HMF were *m*/*z* 109 and 127. The quantification was performed on the basis of the signal response of the ion having *m*/*z* of 109. Chromatograms were also monitored at a detection wavelength of 285 nm.

RESULTS AND DISCUSSION

To date, several types of foods have been subjected to the analysis of HMF by using HPLC with UV detection at 280−285 nm, including spirits and honey (*19*−*21*), wine and other alcoholic beverages (*22*−*24*), fruit juices (*25*−*29*), vinegars (*30*), ultrahigh-temperature-treated milks (*8*), coffee (*31*), breakfast cereals (*32*), breads (*7*), and baby cereals (*33*). The chromatographic conditions used in these studies did not allow a complete baseline separation of the peaks of interfering compounds from the peak of HMF in most cases. Because UV detection is not specific, an accurate detection of HMF will be possible only if a good baseline separation is achieved. On the other hand, MS detection offers more selectivity and accuracy, bringing a significant improvement to the analytical methodology of HMF.

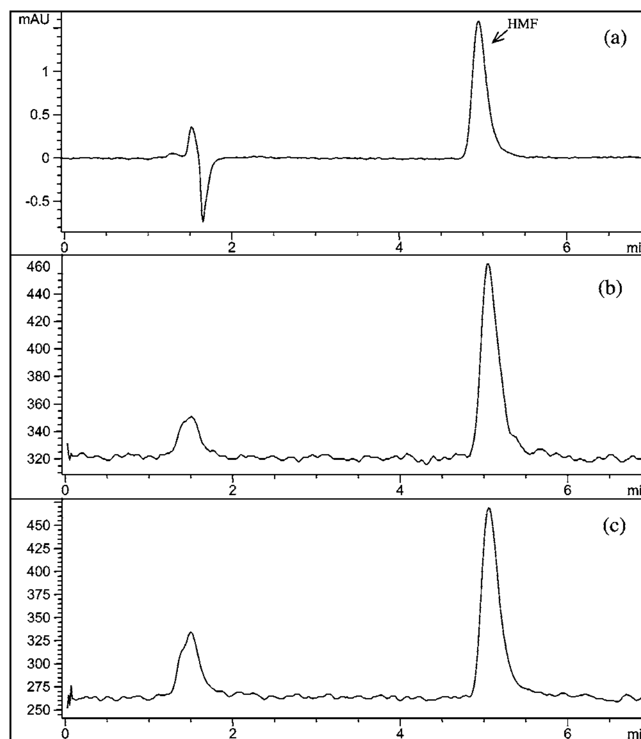

Figure 1. Chromatograms of 0.05 μg/mL of HMF standard: (**a**) 285 nm; (**b**) SIM for *m*/*z* 109; (**c**) SIM for *m*/*z* 127.

Positive APCI-MS analysis of HMF showed both the precursor [M + 1] ion and the compound-specific ion [$C_6H_5O_2$] due to loss of water from the protonated molecule. These characteristic ions having *m*/*z* of 127 and 109 were used to monitor HMF in SIM mode. The ratio of these ions (response of ion 127/response of ion 109 = 1.12) was used to confirm the purity of HMF peak. The signal response was linear over a concentration range of 0.05−2.0 μg/mL for both ions with correlation coefficients of >0.99. On the basis of a signal-to-noise ratio of 3, the limits of detection (LOD) were determined to be 0.005 and 0.006 μg/mL for ions having *m*/*z* 127 and 109, respectively. LC-MS with APCI was found to be a powerful tool that allowed us to determine HMF sensitively and precisely.

Figure 1 shows the chromatograms of HMF at a concentration of 0.05 μg/mL obtained by recording both UV signal at 285 nm and MS signals for ions having *m*/*z* 127 and 109 in SIM mode, simultaneously. Because the column effluent was directed to the MS inlet after passing through the flow cell of the diode array detector, there was a time lag of ∼0.2 min in MS signals. The chromatographic separation of HMF was performed on a Zorbax Bonus RP narrow-bore column. The solution of 0.01 mM acetic acid in 0.2% aqueous solution of formic acid was used as the mobile phase at a flow rate of 0.2 mL/min to increase the ionization yield during MS detection with an adequate separation of HMF in the column from interfering matrix coextractives. Under these conditions, HMF eluted at 5.087 min with good retention time reproducibility (5.09 ± 0.04 min, n = 10). The capacity factor (k') was determined to be 2.33 for HMF on the basis of the hold-up time of 1.55 min.

The usual approach for the extraction of free furfurals from solid food matrices entails extraction with water followed by clarification using Carrez I and II reagents (*7*, *32*, *34*, *35*). Some researchers have used heating in boiling water for 25 min after the addition of oxalic acid into the sample to estimate total HMF (*2*, *6*, *9*, *36*). We initially attempted to use 0.2 mM acetic acid

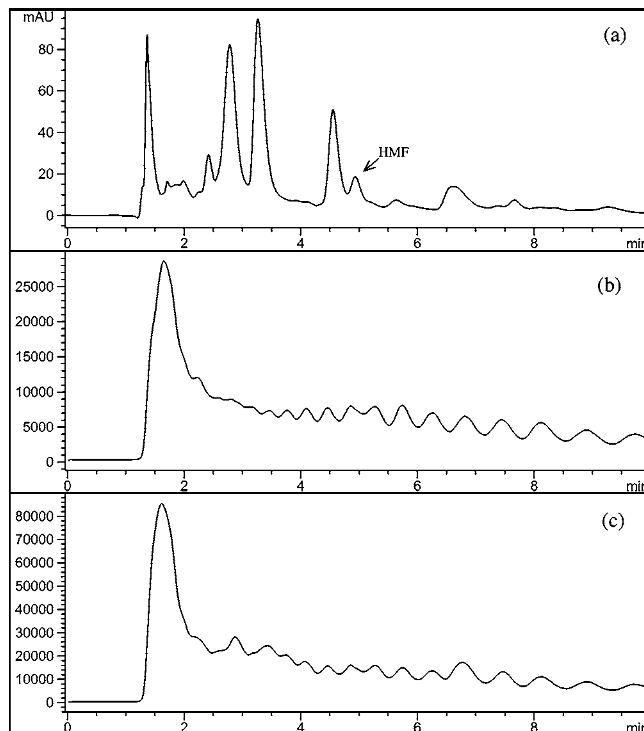

Figure 2. Chromatograms of a cereal-based baby food: (**a**) 285 nm; (**b**) SIM for *m*/*z* 109; (**c**) SIM for *m*/*z* 127.

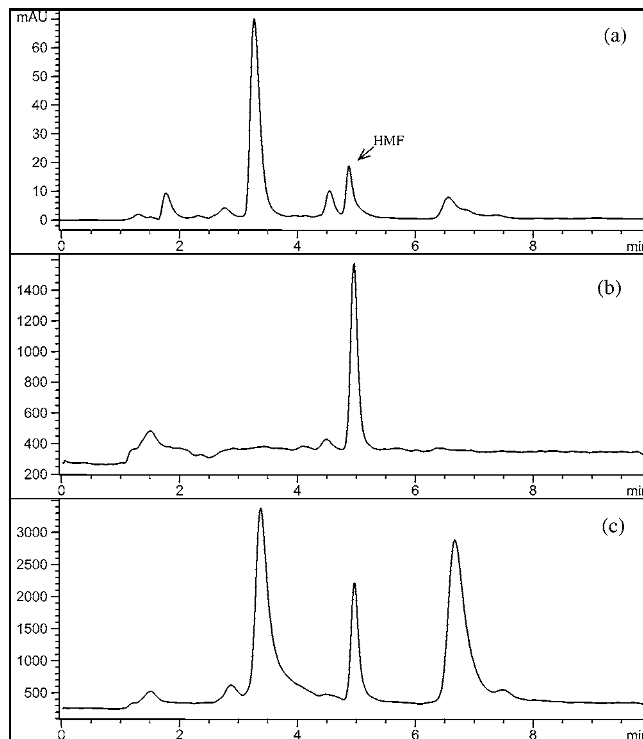

Figure 3. Chromatograms of a cereal-based baby food after SPE cleanup: (**a**) 285 nm; (**b**) SIM for *m*/*z* 109; (**c**) SIM for *m*/*z* 127.

for the extraction of free HMF from appropriately ground food matrix. The extract was simultaneously clarified by adding certain amounts of Carrez I and II reagents during mixing. Therefore, an extract free from the colloids and fat if present was obtained by Carrez clarification and cold centrifugation, respectively. **Figure 2** shows the chromatogram of a cereal-based baby food obtained by recording UV and MS signals, simultaneously. It is clearly seen that there was a significant increase in the baseline signal, which prevented the detection of HMF using MS in SIM mode. Nevertheless, the presence of HMF in this sample was identified on the basis of the detection at 285 nm. However, the peak eluted at ∼4.5 min was not fully resolved from the peak of HMF under the chromatographic conditions applied here. It was confirmed by spectral analysis of the wavelength range between 190 and 350 nm that the peak assigned as HMF was not pure. Further cleanup was, therefore, required for an accurate quantification of HMF in the food sample. Here, an Oasis HLB cartridge packed with a macroporous copolymer of the lipophilic divinylbenzene and the hydrophilic *N*-vinylpyrrolidone was used to clean the extract prior to LC analysis. The clear aqueous extract was passed through a preconditioned cartridge. HMF present in the extract strongly interacted with the sorbent material, whereas much of the coextractives did not. HMF retained in the cartridge was then eluted with diethyl ether. It was determined that 0.5 mL of diethyl ether was sufficient to recover HMF from the cartridge completely. **Figure 3** shows the chromatogram of the sample after cleanup using Oasis HLB. It is clearly seen that SPE cleanup significantly improved the chromatograms by avoiding the coextractives that interfere with HMF during both UV and MS detection. The peak eluted at 4.554 min was the major interference during UV detection at 285 nm. It eluted just before HMF and adversely affected the quantification of HMF. Taking the signal responses at 285 nm into account, 85% of this interfering compound could be eliminated by means of SPE cleanup. A UV scan confirmed the purity of the HMF peak shown in **Figure 3**. It was clear from the results of UV responses that the analyses of extracts without SPE cleanup overestimated the HMF concentrations in the samples. Estimated HMF concentration of the samples was 32% higher when SPE cleanup was not applied.

Table 1. Recovery of HMF from Cereal-Based Baby Food

spiking level, μg/g	recovery, %	RSD, %	*n*
0.25	92.4	3.8	4
1.0	94.7	2.9	4
5.0	91.8	5.1	4

SPE cleanup brought significant improvement for the detection of HMF using MS in SIM mode. The total ion chromatogram indicated the presence of three major peaks in the sample. The HMF peak was identified by comparing both retention time and mass spectral data. The ratio of characteristic ions having *m*/*z* 127 and 109 also confirmed the purity of the HMF peak. The compound-specific ion $[C_6H_5O_2]$ having *m*/*z* of 109 was found to be more selective than the parent compound ion. Therefore, the quantification of HMF was performed using the signal response recorded for this ion.

The accuracy of the method was verified by analyzing spiked cereal-based baby foods. The recovery of HMF was determined by analyzing each of the spiked samples four times for spiking levels ranging from 0.25 to 5.0 μg/g. The mean percentage recoveries exceeded 90% for all levels (**Table 1**).

The method was used to analyze 16 commercial baby food samples (3 milk-based and 13 cereal-based foods). The HMF concentrations of the milk-based foods were <1.0 μg/g. Eight of 13 cereal-based foods were also found to contain a HMF concentration of <1.0 μg/g. There was only a small variation in the HMF concentrations of cereal-based foods with one exception. One of the samples had 57.18 μg/g of HMF, which was significantly higher than the HMF contents of others. The compositions (protein, carbohydrate, and lipid) of baby food

Table 2. HMF Contents of Some Selected Commercial Baby Foods

food type	brand code	compositions as declared on the label, g/100 g of sample			HMF, µg/g
		protein	carbohydrate	lipid	
milk-based foods	A1	6.5	16.0	3.5	0.25
	A2	6.9	16.8	3.5	0.18
	A3	6.5	15.9	3.0	0.43
cereal-based foods	B1	7.1	71.7	16.9	nd
	B2	5.0	73.4	16.5	2.58
	C1	16.0	68.5	8.5	0.40
	C2	15.8	67.6	9.0	0.26
	C3	12.5	76.1	3.5	0.38
	C4	15.5	68.7	8.8	1.07
	D1	15.0	69.7	9.0	1.04
	D2	15.0	69.5	9.0	57.18
	E1	16.1	63.6	9.7	0.18
	E2	11.0	74.0	9.0	0.37
	E3	4.5	73.4	16.0	5.00
	F1	14.3	67.2	11.4	0.17
	F2	1.0	9.0	1.4	0.38

samples based on the label declarations are given in **Table 2**. There were several vitamins and minerals in the samples, but the levels of these minor components were similar in these samples. Therefore, an unusually high concentration of HMF might be due to the differences in the processing conditions.

In conclusion, the growing attention of the scientific community with regard to the potentially toxic effects of HMF requires new efforts to be made to establish new rapid, reliable, and sensitive methods to determine HMF in real matrices. Because their quality is of considerable importance, a variety of baby foods were chosen as the test matrix to develop the analytical method for the determination of HMF. Previous methods usually deal with food items having HMF concentrations that are comparatively higher and utilize extraction procedures which usually do not avoid potential interfering compounds prior to LC analysis. The presence of interferences may be problematic, particularly during the UV detection after LC separation when low concentrations of HMF are being measured in baby foods. In such a case, (i) a rapid separation of HMF from the matrix coextractives in a narrow-bore column, (ii) an efficient cleanup of the extract using SPE, and (iii) a selective detection of HMF using MS may be combined in an analytical method. Here, we described an efficient procedure that removes the interfering coextractives by means of SPE cleanup. This led to an important improvement during the LC analysis of HMF coupled to MS detection in SIM mode, as well as to UV detection at 285 nm. Sample preparation and the subsequent chromatographic run took <20 min to complete. Preliminary analyses showed that the method seems to be applicable for other foods such as honey, jams, tomato paste, and fruit juices, which are known to contain HMF in large amounts.

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