Scientific paper

Spectrophotometric Determination of Trace Amounts of Furfural in Water Samples After Mixed Micelle-Mediated Extraction

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Received: 13-06-2008

Abstract

A new micelle-mediated phase separation method using mixed micelle of the anionic surfactant sodium dodecyl sulfate (SDS) and non-ionic surfactant Triton X-100 for preconcentration of ultra-trace quantities of furfural as a prior step to its determination by spectrophotometry has been developed. The method is based on the extraction of phenylhydrazone derivative, the colored product of the reaction of furfural with phenylhydrazine**,** in acidic media. The phenylhydrazone derivative was concentrated in surfactant rich phase, and then determined spectrophotometrically at 446 nm. The optimal extraction and reaction conditions (e.g. surfactant concentration, reagent concentration and temperature effect) were studied and the analytical characteristics of the method (e.g. limit of detection, linear range, preconcentration factor and enhancement factor) were obtained. Linearity was obeyed in the range of 5.0–450 ng mL^{-1} of furfural and the detection limit of the method was 1.0 ng mL^{-1}. The interference effect of some cations, anions and organic compounds was also tested. The proposed method was successfully applied to the determination of furfural in water samples.

Keywords: Cloud point extraction; spectrophotometric determination; furfural; mixed micelle; preconcentration

1. Introduction

Furfural is an aromatic aldehyde with the chemical formula $C_5H_4O_2$ is used as a solvent in petrochemical refining to extract dienes (which are used to make synthetic rubber) from other hydrocarbons.[1] Furfural is also used as a chemical intermediate in the production of the solvents of furan and tetrahydrofuran. Furfural is a new pesticidal active ingredient intended for use as a fumigant to control root infesting plant parasitic nematodes and fungal plant diseases in greenhouse soil used for growing ornamentals and other non-food commodities.[2] Furfural is toxic and readily absorbed by skin, oral, dermal or inhalation routes of exposure and acute exposure can also damage the liver and kidneys and led to tumors and mutations. The permissible exposure limit (PEL) and the threshold limit value (TLV) for furfural was reported 5.0 μg mL^{-1} and 2.0 μg mL^{-1}, respectively.[3,4] Because of the environmental and toxicological significance of furfural, sensitive and reliable analytical methods are necessary for preconcentration and determination of furfural in samples.

Different methods including gas chromatography,[5–8] high performance liquid chromatography,[9–16] fluorimetry,[17,18] and spectrophotometry,[19–27] have been reported for the determination of furfural.

Spectrophotometric methods offer many appealing characteristics, including simple instrumentation, rapid response times and easy operation. These properties are highly desirable to the future design and development of portable analytical devices capable of quickly responding to trace levels of hazardous compounds in the field. So far, a few spectrophotometric methods have been reported for the determination of furfural. The lowest determination limit for furfural by spectrophotometric method based on reaction with 4-aminophenol in trichloroacetic acid has been reported to be 0.50 μg mL^{-1}.[19] Therefore, for spectrophotometic determination of ultra-trace amounts of furfural a suitable enrichment procedure prior to its determination is necessary. To the best of my knowledge, there is no report on the preconcentration and determination of furfural by cloud point extraction methodology.

The classical liquid-liquid extraction and separation methods are usually time consuming and labor extensive and require relatively large volumes of high purity solvents. Of additional concern is disposal of the solvent used, which creates a severe environmental problem. Cloud point extraction (CPE) is an attractive technique that reduces the consumption of and exposure to a solvent, disposal costs and extraction time.[28,29] The technique is based on the property of most non-ionic surfactants in aqueous solutions to form micelles and become turbid when heated to a temperature as the cloud point temperature. Above the cloud point temperature the micellar solution separates in a surfactant-rich phase of a small volume and in a diluted aqueous phase, in which the surfactant concentration is close to the critical micellar concentration (cmc). Any analyte solubilized in the hydrophobic core of the micelles, will separate and become concentrated in the small volume of the surfactant-rich phase. The small volume of the surfactant-rich phase obtained with this method permits the design of extraction schemes that are simple, cheap, efficient and safe in comparison with liquid-liquid extraction methods.[30,31]

Cloud point (CP) phenomenon is generally observed in nonionic surfactant micellar solutions when the temperature of the system is raised to a certain value, but the use of ionic surfactants (cationic and anionic) in combination with non-ionic surfactant has been documented with an increase in the extraction efficiency of polar organic compounds.[32,33] In this study, to carry out the separation and preconcentration of furfural from mixed micelle-mediated extraction system (mixed-MME),Triton X-100/ SDS, was used. MME is becoming an important and practical application of the use of surfactants in analytical chemistry.[34–36]

In this paper, a cloud point extraction spectrophotometric method for the determination of trace amounts of furfural is described. The method is based on the reaction of furfural with phenylhydrazine and cloud point extraction (CPE) of phenylhydrazone derivative product in mixed surfactant media.

2. Experimental

2. 1. Apparatus

A Hitachi model 3310 UV-Vis spectrophotometer with 1-cm quartz cells (1.0 mL) was used for recording absorbance spectra. All spectral measurements were performed using the blank solution as a reference. A centrifuge with 10 mL calibrated centrifuge tubes (Hettich, Germany) is used to accelerate the phase separation process.

2. 2. Reagents

All chemical reagents used were of analytical reagent grade, and triply distilled water was used throughout the experiments. A standard solution of furfural (1159 μg mL^{-1}) was prepared by dissolving 100 μL furfural (Merck) in water and diluting to the mark with water in a 100 mL volumetric flask. A 0.250 mol L^{-1} phenylhydrazine solution was prepared by dissolving 3.64 g phenylhydrazine hydrochloride in water and diluting to the mark with water in a 100 mL volumetric flask. A 1.0% (w/v) SDS was prepared by dissolving 1.0 g SDS (Merck) in water and diluting to the mark in a 100 mL volumetric flask. A 1.0% (w/v) Triton X-100 was prepared by dissolving 1.0 g Triton X-100 (Merck) in hot water and diluting to the mark in a 100 mL volumetric flask. A 0.2 mol L^{-1} hydrochloric acid solution was prepared by appropriate dilution of concentrated hydrochloric acid (Merck).

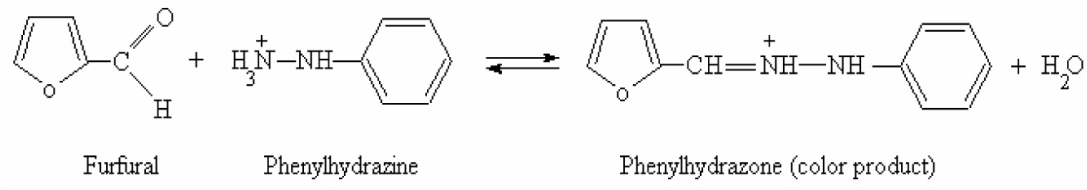
2. 3. Procedure

An aliquot of the solution containing 50–450 ng (0.52–4.69 nmol) of furfural, 1.0 mL of 0.25 mol L^{-1} phenylhydrazine solution, 1.0 mL of 0.2 mol L^{-1} hydrochloric acid solution and 1.0 mL of 1.0% (w/v) SDS were transferred into a 10 mL tube. The solution was diluted to approximately 8 mL with water and was immersed in water bath of 70 °C for 10 min followed by cooling to room temperature. Then 1.0 mL of 1.0% (w/v) of Triton X-100 solution were added. The solution was taken up to the mark with triply distilled water. Separation of the aqueous and surfactant-rich phase was accomplished by centrifugation for 5 min at 3500 rpm. The mixture was cooled in an ice-salt bath to increase the viscosity of the surfactant-rich phase, and the aqueous phase was easily decanted by simply inverting the tube. The surfactant rich phase of this procedure was dissolved and diluted to 1.0 mL with ethanol and transferred to 1.0 mL quartz cell for absorbance measurement at 446 nm.

3. Results and Discussion

The Schiff base and condensation reaction of aromatic aldehydes with amines is the well-known reaction and affording color products that are used for determination of aromatic aldehydes.[37,38] Condensation of furfural with phenylhydrazine affording phenylhydrazone derivative product, proceed according to stoichiometric equation given below:

The effect of SDS as an anionic surfactant on enhancement the rate and equilibrium constants of the condensation reactions of aromatic aldehydes with hydrazine derivatives have been reported.[39–41] SDS micellar media strongly enhance sensitivity the above reaction. Formed phenylhydrazone derivative shows an absorption spectrum with maximum absorbance at 440 nm. It was observed using mixed micelle of the anionic surfactant sodium dodecyl sulfate (SDS) and non-ionic surfactant Triton X-100 in acidic media makes the solution turbid. Therefore, the phenylhydrazone derivative can be extracted by CPE



method. The absorption spectrum of phenylhydrazone derivative in surfactant-rich phase shows a maximum absorbance at 446 nm. After separation of surfactant-rich phase, the absorbance was measured in 446 nm against a reagent blank as the reference (Fig. 1).

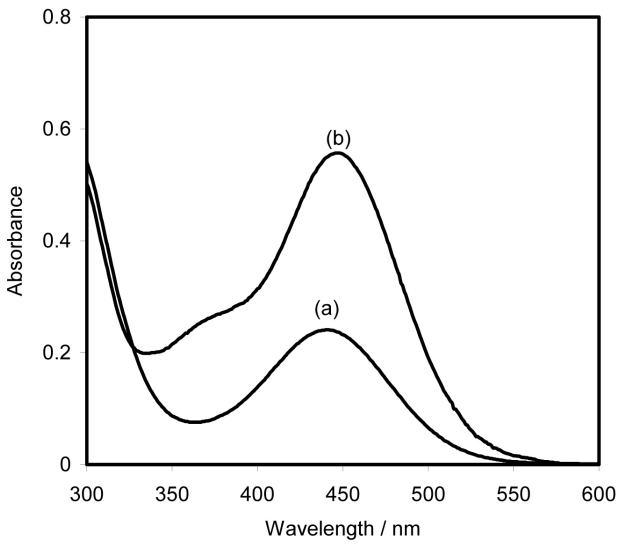


Figure 1. Absorption spectra of phenylhydrazone derivative (a) 2000 ng mL^{-1} furfural before CPE (b) 200 ng mL^{-1} furfural after CPE, Conditions: phenylhydrazine, 2.5×10^{-2} mol L^{-1}; HCl, 0.02 mol L^{-1}; SDS, 0.1% (w/v); Triton X-100, 0.1% (w/v).

3. 1. Optimization of the System

To take full advantage of the procedure, the reagent concentrations and reaction conditions must be optimized. Various experimental parameters were studied in order to obtain optimized system. These parameters were optimized by setting all parameters to be constant and optimizing one each time. This optimization procedure may not lead to the actual optimum, although it certainly leads to an improvement of the analytical method.

The effect of phenylhydrazine concentration on the reaction of furfural with phenylhydrazine was investigated in the range 5.0×10^{-3} –4.0×10^{-2} mol L^{-1}. As Fig. 2 (Curve A) shows, the absorbance increased by increasing phenylhydrazine concentration up to 2.5×10^{-2} mol L^{-1} and remained nearly constant at higher concentrations. Therefore, a concentration of 2.5×10^{-2} mol L^{-1} phenylhydrazine was selected as the optimum.

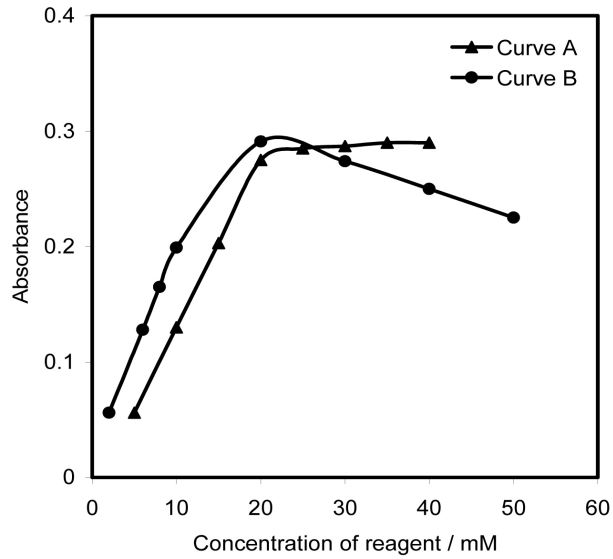


Figure 2. Currve A: Effect of phenylhydrazine concentration on the absorbance system after CPE, **Curve B:** Effect of hydrochloric acid on the absorbance system after CPE, Conditions Conditions: furfural, 100 ng mL^{-1}; SDS, 0.1% (w/v); Triton X-100, 0.1% (w/v).

The effect of hydrochloric acid on the reaction of furfural with phenylhydrazine and also on the cloud point extraction efficiency was studied in the range 0.002–0.05 mol L^{-1}. As Fig. 2 (Curve B) shows, the absorbance increased by increasing hydrochloric acid concentration up to 0.02 mol L^{-1} and decreased at higher concentration. Therefore, a concentration of 0.02 mol L^{-1} hydrochloric acid was used as optimum concentration for the reaction of furfural with phenylhydrazine. The effect of electrolytes on the cloud point while using ionic-nonionic surfactant solutions (Mixed-MME) has been investigated.[42,43] When small amounts of inorganic acids are added to the system, a decrease in the cloud point temperature was noted. In this work, was observed that the addition of hydrochloric acid to the Triton X-100/SDS system reduces drastically the cloud point. Thus allowing phase separation occurs at room temperature.

The effect of temperature on the reaction of furfural with phenylhydrazine was studied in the range 30–85 °C. As Fig. 3 shows, the absorbance increased by increasing temperature up to 70 °C and remained nearly constant at higher temperatures. Therefore, a temperature of 70 °C was used as optimum temperature for the reaction of furfural with phenylhydrazine.

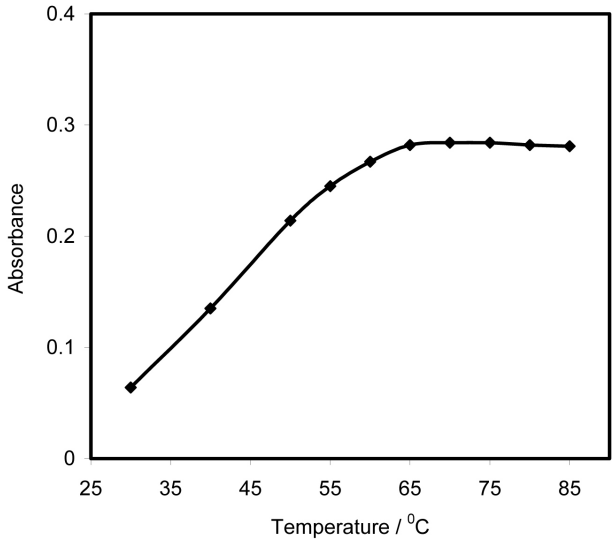


Figure 3. Effect of temperature on the absorbance system after CPE, Conditions: furfural, 100 ng mL^{-1}; phenylhydrazine, 2.5 × 10^{-2} mol L^{-1}; HCl, 0.02 mol L^{-1}; SDS, 0.1% (w/v); Triton X-100, 0.1% (w/v).

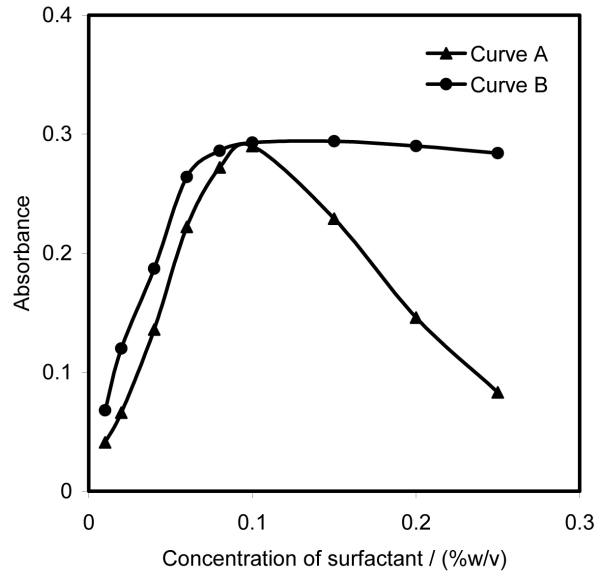


Figure 4. Cuerve A: Effect of SDS concentration on the CPE preconcentration efficiency of the system, **Curve B:** Effect of Triton X-100 concentration on the CPE preconcentration performance of the system, Conditions: furfural, 100 ng mL^{-1}; phenylhydrazine, 2.5 × 10^{-2} mol L^{-1}; HCl, 0.02 mol L^{-1}.

The effect of SDS concentration on the reaction of furfural with phenylhydrazine and also on the CPE preconcentration efficiency of the system was studied in the range 0.01–0.25% (w/v). As Fig. 4 (Curve A) shows, sensitivity of method increased by increasing SDS concentration up to 0.1% (w/v) and decrease at higher concentrations. Therefore, a concentration of 0.1% (w/v) SDS was selected as optimum.

It was observed that Triton X-100 concentration as a non-ionic surfactant can be affecting the extraction of phenylhydrazone derivative product. The effect of Triton X-100 concentration on the absorbance of the extracted phase was investigated. As Fig. 4 (Curve B) shows, the absorbance of the surfactant-rich phase increased by increasing Triton X-100 concentration between 0.01 and 0.25% (w/v) and remained nearly constant at higher concentrations. Therefore, a concentration of 0.1% (w/v) Triton X-100 was used as optimum concentration.

In general, centrifugation time hardly ever affects micelle formation but accelerates phase separation in the same sense as in conventional separations of a precipitate from its original aqueous environment.[44] Therefore, a centrifugation time of 5 min at 3500 rpm was selected as optimum, since complete separation occurred for this time and no appreciable improvements were observed for long time.

Because the surfactant-rich phase was very viscous, ethanol was added to the surfactant-rich phase after CPE to facilitate its transfer into spectrophotometric cell.

3. 2. Analytical Characteristics

Table 1 summarize the analytical characteristics of the optimized method, including regression equation, li-

Table 1: Analytical characteristics of the proposed method

Regression equation (n = 15)	A = 0.0028C + 0.0055, r = 0.9993
Regression equation (n = 15) before preconcentration	A = 0.0001C + 0.0077, r = 0.9995
Linear range (ng mL^{-1})	5.0–450 (500–12000)[a]
Limit of detection (ng mL^{-1})[b]	1.0 (70)[c]
Reproducibility (R.S.D., %)[d]	1.43
Preconcentration factor[e]	10
Enhancement factor[f]	28

[a] Linear range before preconcentration [b] For seven replicate measurements of blank (n = 7) [c]Limit of detection before preconcentration [d] For seven replicate measurements of 100 ng mL^{-1} furfural [e] Ratio of furfural concentration before and after the CPE method. [f] defined as the ratio of the slope of the calibration graph for the CPE method to that of the slope of the calibration graph in micellar media without preconcentration.

near range, limit of detection, reproducibility, and preconcentration and improvement factors. The limit of detection, defined as $C_L = 3S_b/m$ (where C_L, S_b, and m are the limit of detection, standard deviation of the blank, and slope of the calibration equation, respectively [45]), was 1.0 ng mL^{-1}. Because the amount of furfural in 10 mL of sample solution is measured after preconcentration by CPE in a final volume of nearly 1 mL, the solution is concentrated by a factor of 10. The enhancement factor, defined as the ratio of the slope of the calibration graph for the CPE method to that of the slope of the calibration graph in micellar media without preconcentration, was 28.

3. 3. Selectivity

To study the selectivity of the proposed methods, the effect of various species on the determination of 100 ng mL^{-1} furfural by the proposed method was tested under the optimum conditions. The tolerance limit was defined as the concentration of added ion causing less than ±3% relative error. The results showed that 1000 μg mL^{-1} Na^+, K^+, NH_4^+, Ba^{2+}, As^{3+}, Co^{2+}, Sn^{4+}, Mn^{2+}, Zn^{2+}, Cd^{2+}, Ni^{2+}, Fe^{2+}, Ca^{2+}, CO_3^{2-}, PO_4^{3-}, SO_4^{2-}, NO_3^-, Cl^-, Br^-, F^-, acetate, tartrate, citrate, methanol, ethanol, acetone, phenol, urea, thiourea, and ammonia did not interfere on the determination of furfural. The interfering effect of some of aliphatic and aromatic aldehydes on the determination of furfural was investigated. As Fig. 5 shows, 50 μg mL^{-1} acetaldehyde, benzaldehyde, p-dimethylaminobenzaldehyde, 2- bromobenzaldehyde, 3- bromobenzaldehyde and 2- chlorobenzaldehyde did not interfere on the determination of furfural. The main interferences in this case were formaldehyde, 2- nitrobenzaldehyde and 4- nitrobenzaldehyde that the interfering effect of formaldehyde was completely removed by the addition of 0.02 M of sodium sulfite to the solution.

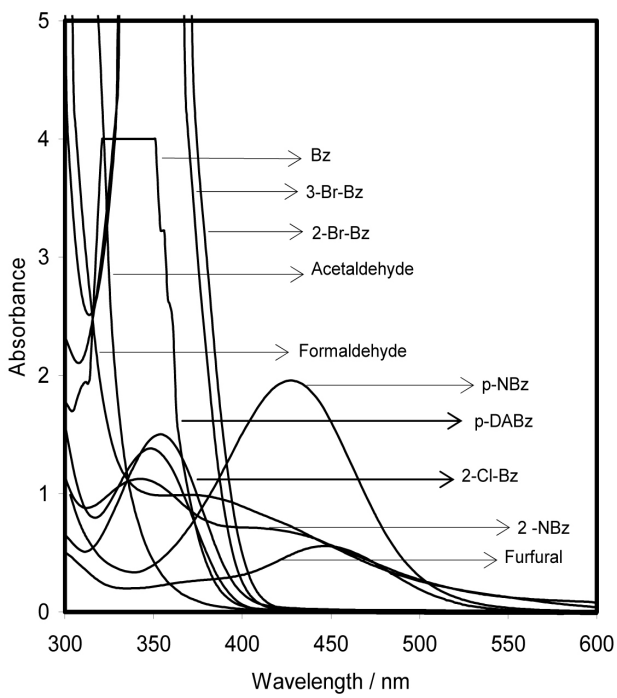


Figure 5. Interfering effect of some of aliphatic and aromatic aldehydes on the determination of furfural, Conditions: 50 μg mL^{-1} acetaldehyde, benzaldehyde (Bz), p-dimethylaminobenzaldehyde (p-DABz), 2- bromobenzaldehyde (2-Br-Bz), 3- bromobenzaldehyde (3-Br-Bz), 2- chlorobenzaldehyde (2-Cl-Bz), and 10 μg mL^{-1} formaldehyde, 2- nitrobenzaldehyde (2-NBz), 4- nitrobenzaldehyde (4-NBz) and 5.0 μg mL^{-1} of furfural.

3. 4. Application

In order to evaluate the analytical applicability of the proposed method, it was applied to the determination of furfural in water samples. To identify potential matrix effects of the water samples, they were spiked with furfural at variable concentrations. The results are given in Table 2. The recoveries for the addition of different concentrations of furfural to samples were in the range of 96–105%. The results show that the proposed method is suitable for determination of trace amounts of furfural in water samples.

Table 2: Determination of furfural in water samples by proposed method[a]

Samples	Furfural (ng mL^{-1})		
	Spiked	Found	Recovery (%)
Drinking water	10	10.3±0.35	103.0
	40	38.7±0.28	96.8
	100	102±0.64	102.0
	400	396±0.80	99.0
River water	20	19.3±0.36	96.5
	50	52.4±0.28	105.0
	150	154±0.77	103.0
	300	296±0.92	98.7
Waste water	40	42.2±0.35	105.0
	80	83.9±0.54	105.0
	200	195±1.03	97.4
	400	396±1.14	99.0

[a] Average of determinations of three separate extract.

4. Conclusion

The proposed method gives a simple, very sensitive, and low-cost spectrophotometric procedure for determination of furfural that can be applied to water samples. The surfactant has been used for separation and preconcentration of furfural in samples, and thus toxic solvent extraction, has been avoided. A comparison of the proposed method with the previously reported methods for determination of furfural (Table 3) indicates that the proposed method is faster and simpler than the existing methods and that it provides a wider dynamic range and a lower limit of detection. The results of this study clearly show the

Table 3: Comparison of the performance of the proposed method with that of other reported methods for the determination of furfural

Analytical method	Sample matrix	Linear range ($\mu g\ mL^{-1}$)	Detection limit ($\mu g\ mL^{-1}$)	Reference
Gas chromatography	Air	0.30–5.5	0.10	[7]
Gas chromatography-mass spectrometry	Vinegar	0.12–16	0.015	[8]
High performance liquid chromatography	Petroleum oil	1–1000	0.5	[10]
High performance liquid chromatography	Water and apple juice	0.05–5.0	0.005	[11]
High performance liquid chromatography	Infant formula	0.01–2.0	0.01	[16]
Differential kinetic spectrophotometry	Water and synthetic mixtures	0.50–6.0	–	[19]
Stoped flow injection- Spectrophotometry	Food and pharmaceutical and water	1.1–14.4	0.47	[21]
Flow injection- Spectrophotometry	Water	20–100	5.0	[23]
Derivative spectrophotometry	Cork	910–7820	–	[25]
Cloud point extraction-Spectrophotometry	Water	0.005–0.45	0.001	Proposed method

potential and versatility of this method, which could be applied to monitoring of furfural spectrophotometrically in various samples. To the best of my knowledge, this is the first report on the preconcentration and spectrophotometric determination of furfural using cloud point extraction.

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Povzetek

Razvili smo novo metodo določanja furfurala z mešano micelarno ekstrakcijo z uporabo natrijevega dodecil sulfata (SDS) in neionskega površinsko aktivnega sredstva Triton X-100. Dosegli smo predkoncentracijo zelo nizkih vsebnosti furfurala do te mere, da je bilo omogočeno spektrofotometrično določanje s pomočjo reakcije s fenilhidrazinom. Fenilhidrazon smo ekstrahiraliv fazi, bogati s površinsko aktivno snovjo, določitev je potekla pri 446 nm. Optimizirali smo pogoje ekstrakcije (koncentracija površinsko aktivnih snovi, koncentracije reagentov, temperatura), ter karakteristike analizne metode (meja določitve, linearnost, predkoncentracijskih količnik, obogatitev). Preiskovali smo tudi moteče katione, anione ter nekatere organske spojine. Postopek smo uporabili za določitev furfurala v vodnih raztopinah.