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Ultrasound assisted-deep eutectic solvent extraction of iron from sheep, bovine and chicken liver samples[1]

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ABSTRACT

A green, novel and effective ultrasound assisted-deep eutectic solvent (DES) extraction (UA-DES-E) procedure was developed for extraction of iron from sheep, bovine and chicken liver samples. The analytical parameters including type and composition DES, volume of DES, ultrasonication time and ratio of sample to DES were optimized by using 50 mg of the NIST SRM 1577b bovine liver certified reference material. The limit of detection (LOD) and the limit of quantification (LOQ) were found as 0.026 μg mL^{-1} and 0.085 μg mL^{-1}, respectively. The relative standard deviation (RSD) as a result of 7 replicates of 50 mg of certified reference material was 1.4%. The accuracy of proposed method was checked by the addition/recovery tests to NIST SRM 1577b bovine liver and a sheep liver. The extraction method was applied to extraction of iron from bovine, sheep and chicken liver samples retail from markets at Kayseri, Turkey with satisfactory results (recoveries higher than 95%).

1. Introduction

Iron is a vital element for normal physiological processes of living organisms. It has important roles in cellular processes and proteins like oxygen transport, the synthesis of DNA and RNA, enzyme activator, and regulation of gene expression, stabilizer and functional component of proteins [1–4]. Iron could be toxic for human body at high levels. It leads to tissue damage because of formation of reactive radicals [5–8]. Under the normal level, iron leads to some problems like limitation of oxygen delivery to cells which cause leading to poor work performance, decreased immunity and death [9,10]. The preservation of efficiency homeostasis for iron is secured either by an increased consumption of iron-containing foodstuffs like pork and calf liver, broad and kidney beans or by taking suitable anti-anaemic medicinal formulations [11]. The determination of iron in food samples has an important role in the environmental protection, food and agricultural science [12–14].

The determination of trace elements in real samples or complex matrices, like food, often requires extensive sample preparation and/or extraction techniques prior to instrumental analysis [5,15–18]. Up to now, many classical sample preparation and/or extraction technique, including wet ashing digestion [16], dry-ashing digestion [19,20], microwave-assisted acid digestion [21,22], ultrasound-assisted extraction [23,24] and slurry preparation [25] have been developed for that purpose. In many of these techniques, mineral acids and oxidizing reagents which are very problematic for environment were used.

Deep eutectic solvents (DESs), which some of them are drinkable, has emerged [26–29]. The synthesis, properties and analytical application of deep eutectic solvents are well documented in literature [28,29]. A deep eutectic solvent is simply prepared by simply mixing two safe components together to obtain a eutectic mixture. DESs are generally obtained by using of combination of ammonium salts and hydrogen bond donors (HBD) and do not react with water. As ammonium salt, the most used component is choline chloride (ChCl) (Vitamin B4) which can be either obtained from biomass or readily synthesized from fossil reserves (million metric tons) with a simple process. Urea, carboxylic acids or polyols are used as hydrogen bond donors (HBD). DESs have been used in various areas like synthesis of nanoparticles, extraction of organic compounds, digestion of inorganic compounds, dissolution of metal oxides, CO_2 absorption, drug dissolution, and purification of biodiesel and electrodeposition of metals [26–30].

Chlonide chloride/lactic acid mixture is selected as deep eutectic solvent for extraction solvent to extract directly iron from liver samples with the help of ultrasonic waves. This solvent is green solvent and easily prepared by accessible, renewable, cheap, non-flammable, nonvolatile and biodegradable components. According to our literature scanning, this deep eutectic solvent was not used for this purpose, until now.

[1] This study is a part of PhD thesis of Erkan Yilmaz.

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In this study, a simple, effective, and green ultrasound assisted-deep eutectic solvent extraction (UA-DES-E) procedure was developed for the extraction of iron from various liver samples. A suitable extraction conditions for the developed method were investigated.

2. Experimental

2.1. Instruments

The instrumental detection system used was a PerkinElmer Model 3110 flame atomic absorption spectrometer (Norwalk, CT, USA) equipped with a 10 cm a long slot-burner and hollow cathode lamp of iron (wavelength: 248.3 nm; slit width: 0.7 nm; lamp current: 23 mA;). An acetylene flow rate of 2.0 L min^{-1} was used with an air flow of 17.0 L min^{-1}. Flame atomic absorption spectrometric (FAAS) analyses were carried out with a classical external calibration approach.

An Ultrasonic bath (Norwalk, CT, USA) was used for extraction of iron from solid samples phase to DES phase. A centrifuge (ALC PK 120 Model, Buckinghamshire, England) was used for phase separation. The liver samples were dried stored at 80 °C for 24 h in the incubator (Zirbus VaCo 2-E, Bad Grund, Germany).

2.2. Reagents and solutions

All solutions were prepared with reverse osmosis purified water ((Millipore Milli-Q system 18 MΩ cm^{-1}, resistivity). Except if otherwise stated, analytical-grade lactic acid, oxalic acid, ethyl glycol, glycerol were obtained from Merck (Darmstadt, Germany). Choline chloride was taken from Alfa Aesar (Karlsruhe Germany). Standard solutions of iron(III) (1000 mg L^{-1}) were prepared by dissolving in water from nitrate salt (Merck). The NIST SRM 1577b bovine liver certified reference material was obtained from U.S. Department of Commerce National Institute of Standards and Technology (Gaithersburg, USA).

Table 1
Abbreviation of the used deep eutectic solvents.

Abbreviation	Salt	HBD	Salt/HBD Mol ratio
DES-1	Choline chloride	Oxalic acid	1/1
DES-2		Ethyl glycol	
DES-3		Glycerol	
DES-4		Lactic acid	
DES-5		Lactic acid	1/2
DES-6		Lactic acid	1/1.5
DES-7		Lactic acid	2/1

2.3. Preparation of deep eutectic solvents

DESs are prepared from available salts and hydrogen bond donors in laboratory conditions. They were listed in Table 1. The deep eutectic solvents were prepared in water by heating choline chloride-hydrogen bond donors to 80 °C with constant stirring until a homogeneous liquid was formed.

2.4. Deep eutectic solvent-based ultrasound assisted extraction procedure

The deep eutectic solvent-based ultrasound assisted extraction procedure is described in Fig. 1. An amount of 50 mg NIST SRM 1577b bovine liver certified reference material was accurately weighted into beakers. Then 8.0 mL of DES-4 (1 CHCl:1 LA) was added in the samples and the beaker covered with a watch glass. The suspensions were processed by ultrasonic extraction at a frequency of 20 kHz and an output power of 200 W max for 45 min. in the ultrasonic bath. Iron was extracted to DES phase, and a relatively uniform solution was obtained. The solution was centrifuged at 4000 rpm for 3 min to complete separation of solid and solution phase. The supernatant solution was completely separated. The resulting mixture was filtered through a blue band filter paper and diluted to 15 mL in a volumetric flask with deionized water. The concentration of iron in final solution were determined with flame AAS by using calibration curve. The same procedure was applied for blank studies. The percentage recovery of iron was calculated by using ratio of obtained value to certified value of iron in NIST SRM 1577b bovine liver certified reference material. The certificated value of NIST SRM 1577b bovine liver certified reference material for iron was 184 ± 15 μg g^{-1}.

2.5. Applications

The bovine liver, sheep liver and chicken liver samples were purchased from different local store in Kayseri, Turkey. The liver samples were cut and dried at temperature 80 °C for 24. The samples dried were homogenized with an agate homogenizer.

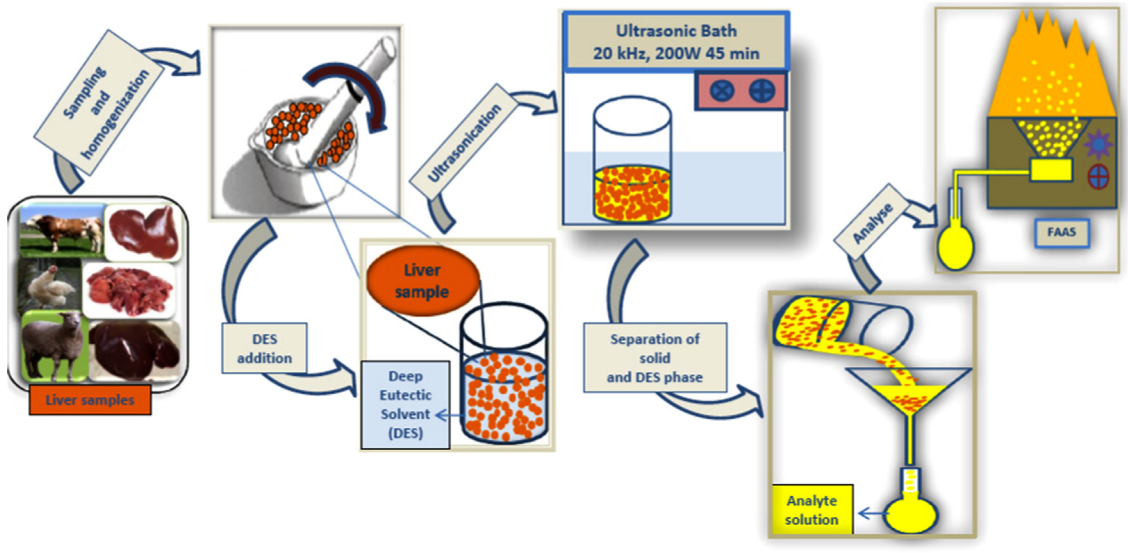

Fig. 1. The procedure of deep eutectic solvent-based ultrasound assisted extraction procedure.