



# The choice of ultrasound assisted extraction coupled with spectrophotometric for rapid determination of gallic acid in water samples: Central composite design for optimization of process variables



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## ABSTRACT

A sensitive procedure namely ultrasound-assisted (UA) coupled dispersive nano solid-phase microextraction spectrophotometry (DNSPME-UV-Vis) was designed for preconcentration and subsequent determination of gallic acid (GA) from water samples, while the detailed of composition and morphology and also purity and structure of this new sorbent was identified by techniques like field emission scanning electron microscopy (FE-SEM), X-ray diffraction (XRD) and Energy-dispersive X-ray spectroscopy (EDX) techniques. Among conventional parameters viz. pH, amount of sorbent, sonication time and volume of elution solvent based on Response Surface Methodology (RSM) and central composite design according to statistics based contour the best operational conditions was set at pH of 2.0; 1.5 mg sorbent, 4.0 min sonication and 150  $\mu\text{L}$  ethanol. Under these pre-qualified conditions the method has linear response over wide concentration range of 15–6000  $\text{ng mL}^{-1}$  with a correlation coefficient of 0.9996. The good figure of merits like acceptable LOD ( $S/N = 3$ ) and LOQ ( $S/N = 10$ ) with numerical value of 2.923 and 9.744  $\text{ng mL}^{-1}$ , respectively and relative recovery between 95.54 and 100.02% show the applicability and efficiency of this method for real samples analysis with RSDs below 6.0%. Finally the method with good performance were used for monitoring under study analyte in various real samples like tap, river and mineral waters.

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## 1. Introduction

Recently, phenolic compounds more attention due to their influence on health and nutrition fields [1,2] which emerge from presence of high content antioxidant, which cause more activity and free radical scavenging capacities which hinder from creation and progress of cancer, cardiovascular diseases, chronic diseases, and so forth [3]. flavonoids [4], polysaccharides [5] and phenolic acids [6], especially gallic acid (inset in Fig. 1) [7,8] as a member of such family applied in beverages, cosmetics, food and medicine as functional products. High importance of such compounds in different fields led to requirement for development of simple and high performance techniques which may be possible for separation

and determination of GA from aqueous environment of natural matrices.

Traditional extraction and/or preconcentration procedure namely as solid phase extraction (SPE), gel permeation chromatography (GPC), liquid-liquid extraction (LLE), cloud point extraction (CPE) and dispersive solid phase extraction (DSPE) has good ability to simplify the matrix and also enhance the analyte concentration, which led to improvement in the figures of merit correspond to under study procedure [9–13].

Although SPE as one of good candidate for supplying high preconcentration factors via simple and low cost procedure over a short time [14], while more attempts is devoted to design and suggest efficient, rapid, economical and miniaturized sample preparation methods [15–17]. A dispersive solid phase micro-extraction (DSPME) based on a Quick, Easy, Cheap, Effective, Rugged and Safe (QEChERS) efficiently and selectively is able to preconcentrated and/or extract the analyte from the most complicated matrix [18–21].

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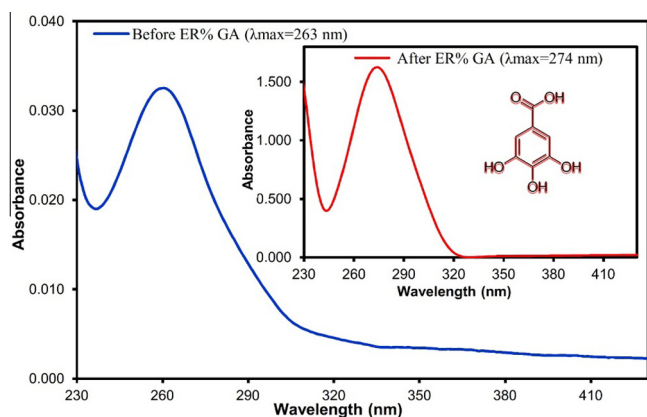


Fig. 1. Absorption spectra before (water) and after (ethanol) UA-DNSPME-UV-Vis for ER% of GA.

The metal nanoparticles are good candidate to be used as sorbent for the preconcentration of different analytes following the loading on the surface of each sorbents or maybe used solely [22–24], while application of most reactive and lower toxicity compound like zinc and copper based nanoparticles led to improvement in characteristic performance of the method with lower cost and higher efficiency [25]. On the other hand, the sorbent based on such atoms is associated with generation of more reactive centers and the border line nature led to increase in their ability to bind the various functional groups of analytes [26–28] which encourage us to apply this new material for enrichment and preconcentration of analytes like gallic acid through the interaction with hydroxyl, carboxylic and/or non-localized  $\pi$  electron, which seems the loading on to the surface of supports like activated carbon make to design environmentally friendly extraction techniques in short time and lower consumption of solvents and energy [15,29,30]. Ultrasound-assisted extraction (UAE) is good choice for reducing extraction time via best and efficient mixing of sorbent with sample solution, while may led to improvement in concentration gradient and the diffusion coefficient of the analyte [31,32]. Sonication following generation of very fine droplet of samples led to very large interfacial contact areas between the liquids and a corresponding dramatic increase in the mass transfer between two immiscible phases [33,34]. Ultrasound assisted dispersive nano-solid phase micro-extraction (UA-DNSPME) known as simple operation, fast, high sensitivity and high linear range procedure is suitable for monitoring different analyte at various level specially phenolic compounds [35].

HPLC methods are widely combined with a pre- or post-column derivatization which takes too much time and using toxic reagent is a common practice in these methods, while its expensive and its maintenance also is tedious [7,36,37]. Combination of spectrophotometry (UV-Vis) advantages with under study preconcentration technique able the researcher to quantify this analyte at trace level in complicated matrix with a simple operation and low cost instrument and the combination with ultrasound-assisted dispersive nano solid-phase micro-extraction coupled with spectrophotometry (UA-DNSPME-UV-Vis) led to suggestion of good operational conditions and figures of merit.

In this study, UA-DNSPME-UV-Vis method based on a nanomaterial efficiency and the influence of parameters such as the pH, amount of sorbent, sonication time, type and volume of eluent solvents were studied and optimized using response surface methodology. Then, the validation parameters tested include the linearity, recovery, precision and sensitivity. Finally, the method was successfully applied to determination of trace amounts of GA in natural water samples.

## 2. Experimental

### 2.1. Reagent and solutions

Zinc acetate ( $\text{Zn}(\text{CH}_3\text{COO})_2$ ), thioacetamide ( $\text{CH}_3\text{CSNH}_2$ ) were purchased from Scharlau Company. EDTA salt, NaCl, HCl, gallic acid, copper acetate ( $\text{Cu}(\text{CH}_3\text{COO})_2$ ) and activated carbon were provided from Merck (Darmstadt, Germany). All chemicals were used as received without further purification. HPLC-grade methanol, ethanol, acetonitrile, DMSO, DMF and acetone were purchased from Merck (Darmstadt, Germany). The stock solution ( $100 \text{ mg L}^{-1}$ ) of analyte are prepared by dissolving 10 mg of solid analyte in 100 mL double distilled water and the working concentrations was prepared daily by their suitable dilution.

### 2.2. Sample preparation

To evaluate the accuracy and applicability of proposed method, the extraction and determination of gallic acid was performed in different water sample matrices, including tap, mineral and river. River water samples were taken from Beshar river, Yasouj, whereas the Tap water was taken from Yasouj city, and mineral water (Zam zam mineral water, Iran) samples were purchased in a local supermarket. Prior to determination of gallic acid in samples, 500 mL of each sample were filtered through Whatman filter paper (Whatman, Grade 1 and 11 mm, cellulose filters Maidstone, England) and the samples were stored at  $4^\circ\text{C}$  until analysis and processed within 1 week of collection.

### 2.3. Apparatus

The pH measurements were carried out using pH/Ion meter model-686 (Metrohm, Switzerland, Swiss) and the GA concentration was determined using UV-Vis spectrophotometer (model V-530, JASCO, Japan) at their respective wavelength. EDX analysis were conducted using energy dispersive X-ray microanalysis system (Oxford INCA II energy solid-state detector) attached to SEM. The morphology of the nanoparticles were observed by field emission scanning electron microscopy (FE-SEM: Hitachi S-4160) under an acceleration voltage of 15 kV. X-ray diffraction (XRD, Philips PW 1800) patterns are recorded from  $20$  to  $70^\circ$  using  $\text{Cu}_{K\alpha}$  as the X-ray source ( $k = 1.54 \text{ \AA}$ ). An ultrasonic processor (Tecno-GAZ SPA Ultra Sonic System, Parma, Italy) operated at 40 kHz with a power of 130 W was used as the source of ultrasound for the enhanced recovery of GA.

### 2.4. Synthesis of sorbent

The Zn: CuS nanoparticles loaded on activated carbon was prepared as follows: 6.0 mL of  $1 \text{ mol L}^{-1}$  zinc acetate ( $\text{Zn}(\text{CH}_3\text{COO})_2$ ) solution was mixed completely with 15.0 mL of  $0.2 \text{ mol L}^{-1}$  EDTA solution and 15.0 mL of  $0.4 \text{ mol L}^{-1}$  thioacetamide solution and the solution pH adjusted on 6.0 and diluted to 100 mL. In the later stage 100 mL of above solution was transferred to Microwave oven (180 W) for 30 min and subsequently the produce nanoparticles in bottom of the baker was removed and filtered, washed several times by de-ionized water and then dried at room temperature. In the next step, 0.3 g of the prepared nanoparticles was dispersed into 50 mL de-ionized water to form an insoluble suspension. Then, 5.0 mL of  $0.2 \text{ mol L}^{-1}$  copper (II) acetate solution was added to the suspension to form Zn: CuS-NPs, which efficiently was deposited on to the activated carbon (AC) along strong stirring for 4 h at room temperature.

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