

Electrochemical Behavior and the Determination of Furan in Beverage Samples Using Glassy Carbon Electrode

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ABSTRACT: Furan is a possible human carcinogen in many types of foods. A new and sensitive electroanalytical method for determination of furan has been developed and validated. The best condition for electrochemical response was obtained with 0.1 M Britton-Rabinson buffer solution (pH=5) a glassy carbon electrode (GCE) was used as the working electrode, a Ag/AgCl/ KCl(sat.) electrode served as the reference electrode, and a platinum wire as the auxiliary electrode under the Differential Pulse Voltammetry (DPV) mode. The peak current obtained from DPV was linearly dependent on the Furan concentration in the range 12–360 μM (0.81–24.5 ppm) with correlation coefficients of 0.999 and a limit of detection (LOD) of 3 μM (0.2 ppm) and limit of quantification (LOQ) of 10 μM (0.68 ppm) were calculated, respectively. The values of the electron-transfer coefficient (α) involved in the rate determining step calculated from the linear plots of E_p against $\ln(v)$ in the pH range investigated were 0.8 confirming the irreversible nature of the oxidation peak. The reproducibility of the method was tested by analyzing 10 samples containing 30 μM of Furan. The RSD % of the method thus obtained was 3.0 % which showed excellent reproducibility for this developed method.

Key words: Furan, Electrochemical studies, Differential pulse voltammetry, Beverage Samples

INTRODUCTION

As a result of industrialization, synthetic chemicals have imposed adverse effects on human beings and other organisms (Vinodhini and Narayanan, 2009; Agbozu and Opuene, 2009; Abdullahi *et al.*, 2009; Aktar *et al.*, 2009; Chibunda, 2009; Hasan *et al.*, 2010; Sadashiva Murthy *et al.*, 2009; Dutta and Dalal, 2008; Srivastava *et al.*, 2008; Banaee *et al.*, 2008; Asi *et al.*, 2008; Wasim Aktar *et al.*, 2008; Priju and Narayana, 2007; Jafari and Ebrahimi, 2007; Nasrollahzadeh *et al.*, 2007; Shahidi Bonjar, 2007; Shegefti *et al.*, 2010; Adjei-Boateng *et al.*, 2010; Belarbi and Al-Malack, 2010; Hassani *et al.*, 2010; Ghaderi *et al.*, 2012). Furan is a heterocyclic organic compound, consisting of a five-membered aromatic ring with four carbon atoms and one oxygen. The class of compounds containing such rings is also referred to as furans. Furan is a colorless, flammable, highly volatile liquid with a boiling point

close to room temperature. It is soluble in common organic solvents, including alcohol, ether and acetone, but is insoluble in water (Jakubke and Jeschkeit 1994). It is toxic and may be carcinogenic. Furan is used as a starting point to other specialty chemicals (Hoydonckx *et al.*, 2005). Furan is used as an intermediate in the synthesis of many chemicals and pharmaceutical agents such as solvents, resins, and lacquers (Carfagna *et al.*, 1993). Furan yields a tumor incidence, where more than 90% of the tumors are adenocarcinomas, and the remainders are squamous cell tumors. In rats, furan induces cholangiocarcinomas, which are tumors that arise from the intrahepatic epithelium. Cholangiocarcinomas tend to grow slowly and to infiltrate the surrounding hepatic parenchyma (NTP, 1993). Furan is formed in many common foods during the thermal treatment of either ascorbate, carbohydrates (with or without amino

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acids), unsaturated fatty acids, carotenes, or organic acids (Becalski and Seaman, 2005). During recent decades, a variety of pioneer methods for detection of xenobiotics have been introduced (Ashraf *et al.*, 2012; Clemente *et al.*, 2012; Shwetha *et al.*, 2012; Maheswari and Ramesh, 20112; Garcia-Flores *et al.*, 2013; Etale and Drake, 2013; Ashan and Del Valls, 2011; Annabi *et al.*, 2011; Kapdan *et al.*, 2011; Chen *et al.*, 2011; Akhtar and Tufail, 2011; Naim *et al.*, 2011; Heidari *et al.*, 2011; Saeedi *et al.*, 2013; Jovic *et al.*, 2013; Bhuvaneshwari *et al.*, 2013; Olivalla *et al.*, 2013; Blagojevic *et al.*, 2012; Lopez-Pineiro *et al.*, 2012; Sotelo *et al.*, 2012). There are currently a large number of analytical methods for the determination of furan in foods, the analytical problems first reported (Senyuva, and Gökmen, 2005; Goldmann *et al.*, 2005; Bianchi *et al.*, 2006; Hasnip *et al.*, 2006; Wenzl, *et al.*, 2007; Sarafraz-Yazdi *et al.*, 2012; Sid kalal *et al.*, 2012; Byrns *et al.*, 2006; Fan., 2005; Kallio *et al.*, 1989; Locas *et al.*, 2004; Maga., 1979; Mark *et al.*, 2006; Merritt *et al.*, 1963; Persson *et al.*, 1973; Stoffelsma *et al.*, 1968; Tatum *et al.*, 1969; Zoller *et al.*, 2007). In recent years, the electrochemical techniques have led to the advancement in the field of analysis because of their sensitivity, low cost and relatively short analysis time, as compared with other techniques. Electrochemical have proven to be useful for development of very sensitive and selective methods for the determination of organic molecules. In this work, differential pulse voltammetry (DPV) was method with good precision and accuracy for the determination of furan in beverage samples developed.

MATERIALS & METHODS

Furan (>99.0% purity), was purchased from Merck and Co. Inc. (Darmstadt, Germany).

A stock solution of furan (1mM) was prepared in methanol-water (10:90, v/v), britton-rabinson buffers from pH 2.0-10 (0.1 M) which used for pH experiment and also supporting electrolyte, were prepared in doubly distilled water. All Other reagents were of analytical grade and obtained from Merck, and all solutions were prepared with doubly distilled water. Voltammetric experiments were performed using a μ Autolab Type III electrochemical system. A conventional three-electrode cell consisting of a glassy carbon working electrode (2 mm in diameter), a platinum wire counter electrode and a saturated Ag/AgCl reference electrode were used for voltammetric experiments. A digital pH-meter (Ion Analyzer 827, Metrohm) with precision of ± 0.001 was used for pH measurements. All the experiments carried out at room temperature. Before each experiment and transferring the glassy carbon electrode to the solution, it was cleaned by polishing with 0.05 μ m alumina slurry on a

polishing cloth and rinsed thoroughly with doubly distilled water. After each polishing step to get a stable and reproducible background current, Cyclic voltammetry were performed at scan rate of 100 mV/s between 0.0 and 1.6 V for 10 times in 0.1 M H₂SO₄ solution (Bagheri and Hosseini 2012). To study the accuracy and repeatability of the developed method, carried out using the standard addition method by adding the known amounts of Furan to the pre-analyzed samples then mixtures were analyzed by the developed method.

RESULTS & DISCUSSION

In order to understand the electrochemical process occurring at the GCE (glassy carbon electrode), cyclic voltammetry were applied. Fig.1 shows a cyclic voltammogram of 1mM Furan in 0.1 M britton-rabinson buffer solution (pH=5) at a glassy carbon electrode at 50 mv/sscan rate. As shown in Fig. 1, Furan gives one well-defined irreversible oxidation peak at +1.44 V. The effect of supporting electrolytes on current–potential curves was investigated by a 0.1 M solution of various supporting electrolytes (Fig.2). The results showed that the britton-rabinson buffers can give the best background and signal response. For studying the effect of pH of britton-rabinson buffer solution on voltammetric response of Furan, the cyclic voltammograms of 1.0 mM Furan were recorded from pH 2.0 to 10.0 at a scan rate of 50.0 mV/s. The anodic peak current was affected by the pH of the solution (Figs.3-4). As Fig. 4 shows, the maximum peak current was obtained at pH 5.0. So pH 5.0 was chosen for the subsequent analytical experiments. Useful data involving electrochemical mechanism mostly can be acquired from the scan rate and its relationship with peak current and potential. Therefore, the influence of potential scan rate on the electrochemical behavior of Furan at GCE was studied with the change of scan rate (Fig. 5A). As shown Fig. 5A, when the scan rate was increased, a linear relationship between the peak current and the square root of scan rate v (Fig. 5B) in the range from 25 to 250 mV/s was obtained, suggesting a diffusion-controlled process for oxidation of furan at GCE.

A plot of logarithm of anodic peak current vs. logarithm of scan rate gave a straight line with a slope of 0.612 (Fig. 6A) closes to the theoretical value of 0.5, which is expected for an ideal reaction for the diffusion-controlled electrode process (Laviron *et al.*, 1980; Hegde, *et al.*, 2009). In addition, the E_p of the oxidation peak was dependent on scan rate. The anodic peak potential shifted to positive values when the scan rate was increased, which these results also confirmed that the oxidation reaction was irreversible (Fig. 6B).

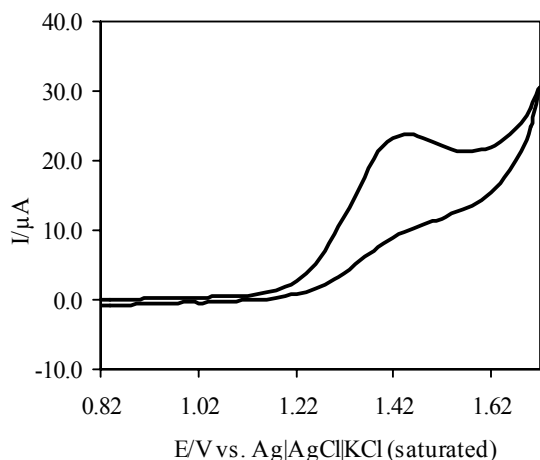


Fig. 1. a cyclic voltammogram of 1mM Furan solutions in methanol-water (10:90) and 0.1 M Britton-Rabinson buffer solution (pH=5) at a GCE and 50 mV/s scan rate to the Ag/AgCl reference electrode

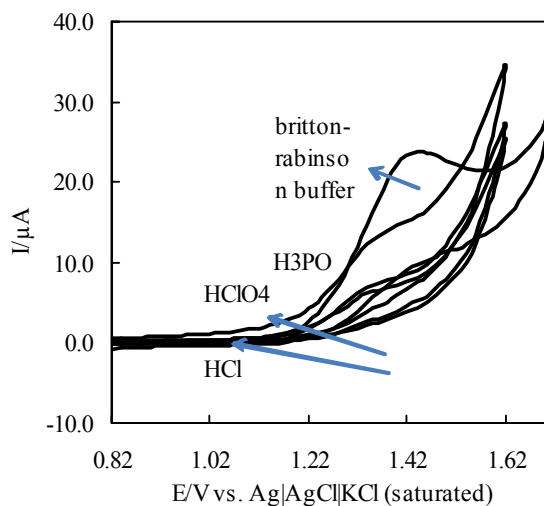


Fig. 2. Cyclic voltammograms of furan with 0.1 M solution of various supporting electrolytes

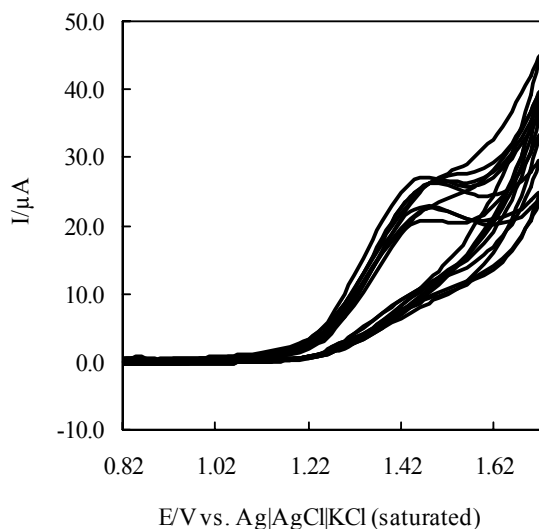


Fig. 3. DPVs of 1mM furan solutions in methanol-water (10:90) at a GCE in BR buffers at pH 2,3,4,5,6,7,8,9,10. The scan rate was 50 mv s⁻¹. The reference electrode was Ag/AgCl

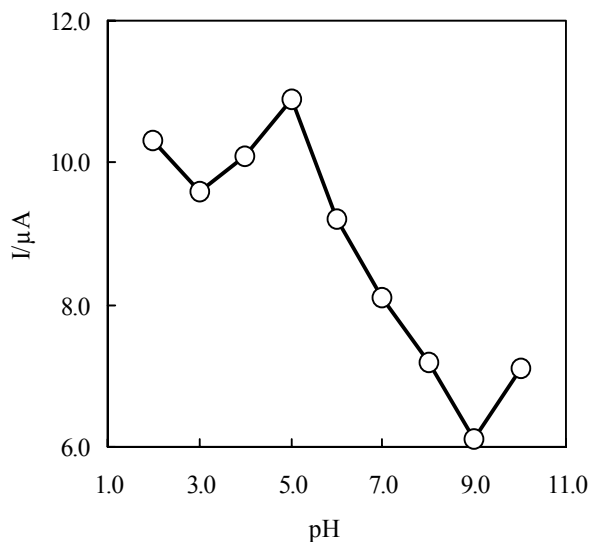


Fig. 4. Plot of I of the oxidation peak potential of 1mM Furan solutions in methanol-water (10:90) against pH for DPV at a GCE. The reference electrode was Ag/AgCl

According to Nicholson (Bard, and Faulkner, 2001), for an irreversible anodic reaction, the relationship between E_p and v is described as follows:

$$E_p = E^0 + RT/(1-\alpha)n_\alpha F [0.780 + \ln(D^{1/2}/k_s) + \ln((1-\alpha)n_\alpha F v / RT)^{1/2}] \quad (1)$$

where E is formal standard potential, α is the charge transfer coefficient, n_α is the number of the electrons

transferred in the rate determining step, D is the diffusion coefficient of Furan and k_s is the standard heterogeneous reaction rate constant. F , R and T have their usual meaning. Thus the value of $(1-\alpha)n_\alpha$ can be easily calculated from the slope of E_p vs. $\log v$. In this system, the slope is 0.149, taking $T = 298$ K, $R = 8.314$ J K⁻¹ mol⁻¹ and $F = 96,480$ C, $(1-\alpha)n_\alpha$ was calculated to be 0.2. Generally, the results suggested that one-electron transfer process are the rate-limiting by step

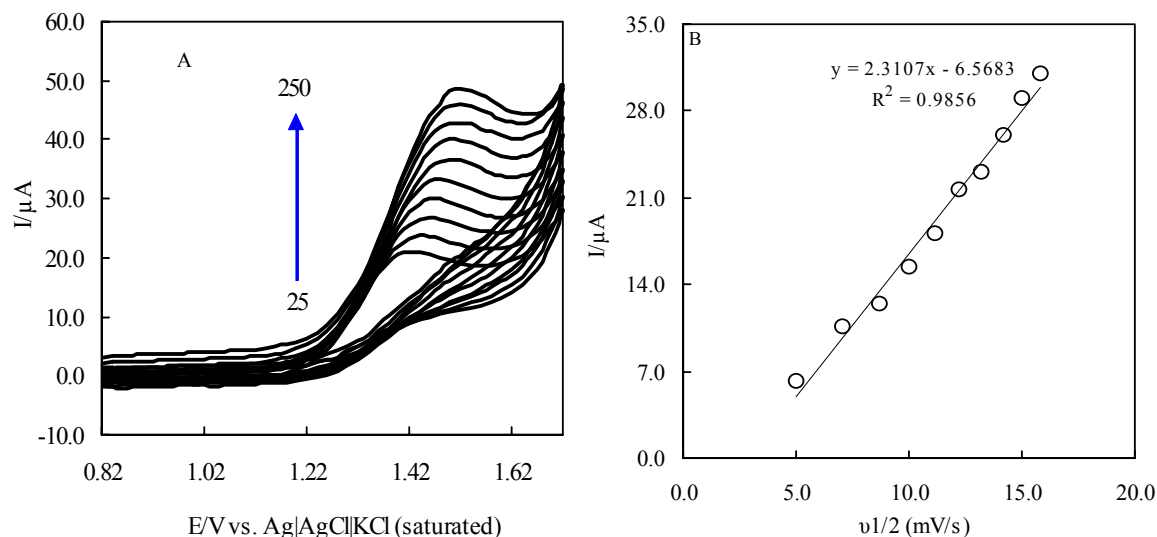


Fig. 5. A: Cyclic voltammograms of 1mM furan solutions in methanol-water (10:90) in 0.1 M britton-rabinson buffer solution (pH=5) , at various scan rates, from 25 to 250 mV/s.

B: Variation of $v^{1/2}$ with anodic peak current I, at a GCE with Ag/AgCl as reference electrode

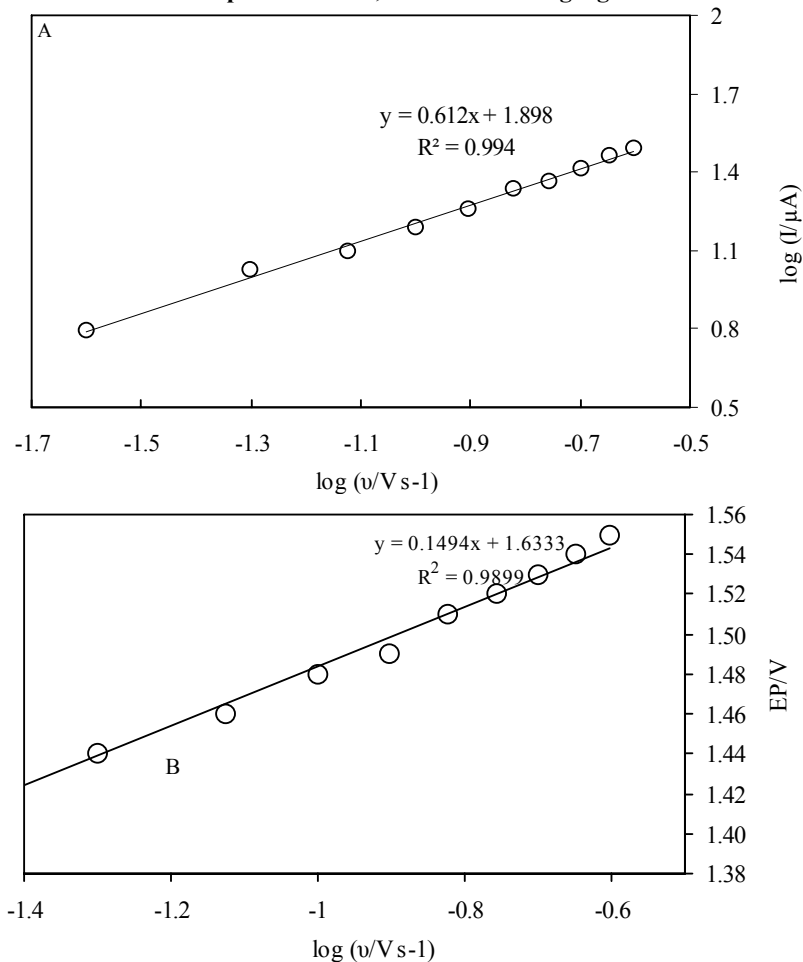


Fig. 6. A:Variation of the logarithm of peak current I with the logarithm of the scan rate **B:**Variation of E_p versus the logarithm of the scan rate for 1mM Furan

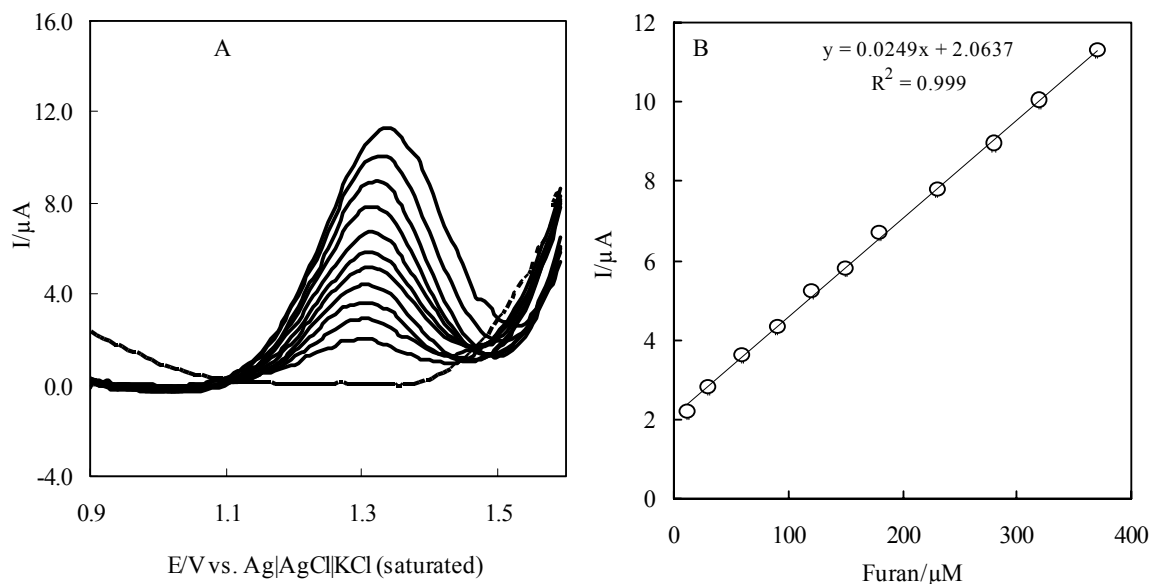


Fig.7. (A): differential pulse voltammograms of Furan at GCE in 0.1 M Britton-Robinson buffer solution (pH 5.0). Furan concentrations (from bottom to up): 12, 30, 60, 90, 120, 150, 180, 230, 280, 320 and 370 μM. (B): corresponding linear calibration curves of the anodic peak currents vs. Furan concentration

Table 1. Determination of furan in beverage samples

Beverage	Spiked (10 ⁻⁶ M) ^a	Found (10 ⁻⁶ M) ^a	Recovery(%) + RSD%
Sample 1	30	30.3	101 ± 3.6
	50	49.7	99.4 ± 3.90
	70	69.1	98.7 ± 4.2
Sample 2	30	30.9	103 ± 4.1
	50	50.8	101.6 ± 3.2
	70	68.7	98.14 ± 3.6
Sample 3	30	29.7	99 ± 4.6
	50	48.3	96.6 ± 3.2
	70	68.2	97.4 ± 4.7

a: four determination

assuming charge transfer coefficient $\alpha=0.8$ for Furan. Differential-pulse voltammetry (DPV) is one of the most sensitive electrochemical detection methods. Therefore, DPV was used for determination of Furan. Under the optimized experimental conditions, the DPV curves were obtained using different concentrations of Furan (Fig. 7A). As it can be seen, the height of the DPV peaks (I_{pa}) increases with increase in concentration of Furan. The calibration curve was obtained using data from these measurements (Fig. 7B). According to the obtained results, linear calibration graphs were obtained for Furan with the linear dynamic range of 12–360 μM (0.81–24.5 ppm). The linear regression equations were $I_{pa} (\mu A) = 2.063 + 0.024 C_{Furan} (\mu M)$, with a correlation coefficient of 0.999 and a sensitivity of 0.024 μA/μM. A limit of detection (LOD) of 3 μM (0.2 ppm) and limit of quantification (LOQ) of

10 μM (0.68 ppm) were calculated according to the $3 s_b/m$ and $10 s_b/m$ criterions, respectively, where m is the slope value of the calibration curve and s_b is the calculated standard deviation for the peak currents of the blank (Four runs).

The reproducibility of the method was tested by analyzing 10 samples containing 30 μM of Furan. The RSD % of the method thus obtained was 3.0 % which showed excellent reproducibility for this developed methods. To demonstrate the efficiency of the method, the developed method was applied for determination of Furan in three different beverage samples. The recovery experiments were carried out using the standard addition method by adding the known amounts of Furan to the beverage samples, and the calibration plot was used for determination of spiked Furan in beverage samples. The results of recovery

experiments were also shown in Table 1. The results illustrated that the proposed method is accurate enough for practical applications.

CONCLUSION

A glassy carbon electrode was used as the working electrode for determination of Furan. The electro-catalytic activity of the GCE electrodes showed a good electro-catalytic effect toward Furan electro-oxidation. The results of oxidation kinetics of Furan indicated that the oxidation process is diffusion controlled. Finally, the oxidation current of Furan on GCE electrodes was used for determination of Furan in aqueous solution and a linear calibration was found in the range of 12–360 µM. Therefore this method can be used for quantitative determination of Furan in real samples.

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