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# Voltammetric Studies of Aspirin in Pharmaceutical Sample Using Ctab - Pencil Graphite Electrode

**P. Manjunatha[1], C. C. Vidyasagar[2]* and Y. Arthoba Nayaka[1]**

[1]*Department of Chemistry, Kuvempu University, India*

[2]*Department of Chemistry, Rani Channamma University, India*

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***Corresponding author:** C. C. Vidya sagar, Department of Chemistry, Rani Channamma University, Belgaum-591 156, India

**Abstract**

Aspirin is a salicylate drug, often used as analgesics to relieve minor aches and pains. In order to determine the concentration of Aspirin in pharmaceutical formulation, here a voltammetric investigation has been carried out to get the electrochemical response. It is necessary to modify a bare PGE with suitable modifier for good selectivity, sensitivity and responsibility. Therefore to determine the Aspirin in pharmaceutical samples have large scope today. So, in order to determine the concentration of Aspirin in samples, here a voltammetric investigation has been carried out to get a good electro chemical response. It is necessary to modify a bare PGE/CPE with suitable modifier to perform the studies. Today, voltammetric techniques are available for the mechanistic study of redox systems. It enables the electrode potential to be scanned is search of redox couples. A couple can be characterized through the potential of peaks of voltammogram and changes by variation of scan rate. The capability together with its variable time scale and good sensitivity made voltammetry the more versatile electro-analytical technique. It must, however be emphasized that its merits are largely in the realm of qualitative or diagnostic experiments. Voltammetry has its ability to generate a species during one scan and then probe its fate with subsequent scans. Main objective of this work is to study the versatility of modified PGE electrode in term of application to investigate electrochemical behaviour of aspirin and elucidation of sequence of electron transfer that occur at the electrode surface by voltammetric techniques.

**Keywords:** Aspirin; CTAB; Penicillin Graphite Electrode (PEG); Redox; Scan rate and Voltammeter

## Introduction

Acetyl salicylic acid (ASA) is commonly known as aspirin. It is one of the most important anti-inflammatory drugs in the world [1-3]. Aspirin is a white powder stable in a dry environment but that is hydrolyzed to Salicylic acid and acetic acid under humid or moist conditions. Hydrolysis also can occur when aspirin is combined with alkaline salts or with salts containing water of hydration [4]. In 1960's Felix Hofmann of the Bayer Company in Germany prepared aspirin [5]. This was found to good medicinal properties, low membrane irritation and a reasonable taste. He called the new medicine as aspirin ('a' for acetyl- the systematic name for the compound at the time of was acetyl salicylic acid, 'spir' for spirea, the meadow sweat plant). The active ingredient of aspirin was first discovered from the bark of the willow tree in 1763 by Edward stone. Aspirin is parts of a group of medications called as non steroidal-inflammatory drugs (NSAIDs), but differ from most of other NSAIDs in the mechanism of action [3-5].

Aspirin is rapidly absorbed largely interact from the stomach and upper small intestine on oral administration but is largely hydrolyzed by plasma esterase's. It is available in large dosage forms and strengths as tablets, suppositories, capsules, enteric coated tablets and buffered tablets [6]. Today aspirin was detected through various chemical and instrumental methods, because aspirin is an electrochemically active substance. The various methods that are used for detection of aspirin are HPLC, ultraviolet spectrophotometer, fluorescence detection, Molecular imprinted technique (MIP), pensive method, Flow injection analysis and Batch injection analysis. The electrochemical behaviour of the aspirin molecule was investigated by employing Cyclic Voltammetry (CV), Electrochemical Impedance Spectroscopy (EIS), Chrono Coulometry (CC), and Adsorptive stripping differential pulse voltammetry (Ad SDPV) [7-9].

Aspirin is used in the treatment of a number of conditions, including fever, pain, rheumatic fever, and inflammatory diseases, such as rheumatoid arthritis, pericarditis, and Kawasaki disease. Lower doses of aspirin have also shown to reduce the risk of death from a heart attack or the risk of stroke in some

circumstances. There is some evidence that aspirin is effective at preventing colorectal cancer, though the mechanism of this effect is unclear [10]. Aspirin is a well established anti platelet drug in the treatment of atherothrombotic vascular disease. These effects include, dose dependent inhibition of platelet function, the enhancement of fibrinolysis, and the suppression of blood coagulation. Aspirin is also used long term, at low doses, to help prevent heart attacks, strokes, and blood clot formation, in people at high risk of developing blood clots. Aspirin clearly decreases mortality and reinfarction when given as short-term therapy for AMI, when given to patients with unstable angina, and when given as long-term secondary preventive therapy in a wide range of patients with established cardiovascular disease. The dose of aspirin should always be the lowest dose that is known to be effective (i.e. 160 to 325 mg for acute treatment of cardiovascular events and 75 to 160 mg/d for primary and secondary prevention) because higher doses result in higher rates of complications [11,12]. Higher dose of aspirin only contribute to its side effects, especially internal bleeding upper gastrointestinal irritations. The inhibition of prostaglandin synthesis is responsible for the anti-inflammatory effects of aspirin but also results in the alteration of normally protective prostaglandin functions with potentially serious consequences, including gastric ulcers, renal failure, and impaired platelet function with resultant hemorrhagic complications. In case of high dosage it also causes indigestion, bleeding, allergy like reactions and effect at the time of pregnancy [13].

The present work is aimed to Quantitative investigation and electrochemical studies of biological important electro active molecule Aspirin in pharmaceutical samples. Modifier to perform the studies. Today, voltammetric techniques are available for the mechanistic study of redox systems. It enables the electrode potential to be scanned is search of redox couples. A couple can be characterized through the potential of peaks of voltammogram and changes by variation of scan rate. The capability together with its variable time scale and good sensitivity made voltammetry the more versatile electro-analytical technique. It must, however be emphasized that its merits are largely in the realm of qualitative or diagnostic experiments. Voltammetry has its ability to generate a species during one scan and then probe its fate with subsequent scans. Main objective of this work is to study the versatility of modified PGE electrode in term of application to investigate electrochemical behaviour of aspirin and elucidation of sequence of electron transfer that occur at the electrode surface by voltammetric techniques.

## Methods and Materials

All the voltammetric measurements were performed using electrochemical workstation (model CHI660D, USA). All electrochemical experiments were performed in a standard three-electrode assembly Incorporating the pencil graphite electrode (PGE) or CTAB modified pencil graphite electrode (CTAB-PGE), platinum wire , and standard calomel electrode as working, auxiliary and reference electrode. All the chemicals used were of analytical grade. Aspirin was produced from Sigma Aldrich (China) and potassium dihydrogen phosphate, di-potassium hydrogen phosphate were produced from Merck (Mumbai, India). The doubled distilled water was used for the preparation of all reagents.

### Preparation of pencil graphite electrode (PGE) and CTAB modified pencil graphite (CTAB-PGE)

A working electrode was constructed with 2B graphite leads (0.5 mm diameter) produced from Camlin Ltd and posterior end of the pencil lead was connected to a copper wire of electrical contact. Then the pencil lead was inserted in the plastic tube and filled with epoxy resin. After 24 hours (time required for the setting of epoxy resin) interior end of electrode was scrapped using a sharp knife and mirror polished using emery paper followed by butter sheet. The mirror polished PGE was sonicated and finely washed with double distilled water. The prepared PGE was soaking in CTAB-solution for 15-20 min, for well modification. The CTAB solution was prepared by dissolving 10mg of CTAB in 10mL of doubly distilled water, after 20 minutes the electrode was taken out from the solution, it was washed with doubly distilled water and it was directly used for voltammetric investigation of Aspirin [14].

### Preparation of real sample

Take one complete tablet of aspirin (Ecosprin-150) and finely grinded using pestle mortar. From this take 30mg and it is dissolved in 25mL of phosphate buffer. The solution was filtered off to get clear solution. The filtrate was directly used for the electrochemical studies.

## Results and Discussions

### Electrochemical behaviour of $K_3[Fe(CN)_6]$at PGE and (CTAB-PGE) electrode

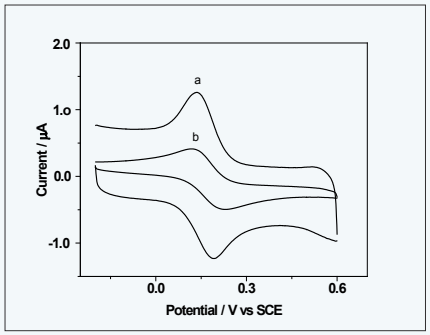


**Figure 1:** Shows Cyclic voltammogram of 5mM $K_3[Fe(CN)_6]$ in 0.1M KCl solution at CTAB-PGE and PGE with scan rate 100 $mVs^{-1}$.

The behaviour of CTAB-PGE can be monitored via the redox behaviour of the $[Fe(CN)_6]^{3/4-}$ couple. (Figure 1) shows cyclic voltammogram obtained at PGE and CTAB-PGE in 0.1M KCl containing 5mM $K_3[Fe(CN)_6]$. The PGE shows a reversible


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voltammogram with a peak of the $[Fe(CN)_6]^{3-/4-}$ couple characteristic of a diffusion controlled redox process (curve-b). The redox response of $[Fe(CN)_6]^{3-/4-}$ couple at CTAB-PGE was observed (curve a). The reduction in peak to peak separation of potential at CTAB-PGE clearly indicated the better performance than that of PGE. From the (Figure 1), it is also confirmed that the redox peak current of $[Fe(CN)_6]^{3-/4-}$ couple was increased at CTAB-PGE in contrast to be unmodified PGE.

## Electrochemical response of phosphate buffer solution and aspirin at PGE and CTAB-PGE

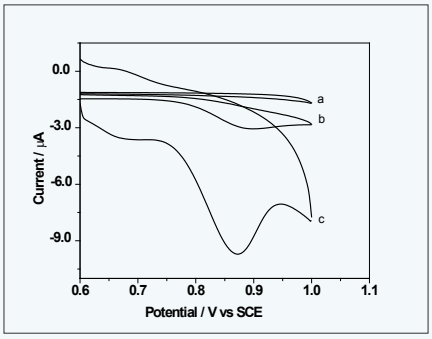


**Figure 2:** Shows the cyclic voltammogram of phosphate buffer at PH-7 and aspirin in PGE and CTAB-PGE.

In order to compare the PGE with CTAB the method of cyclic voltammetry was used to follow electro catalytic behaviour of electrode (Figure 2). It shows that, the electro chemical response of phosphate buffer solution of pH-7 (curve-a). The baseline is only due to the charging and discharging of the electrode double layer. In the applied potential range there is no redox peaks were observed. But the Electrochemical oxidation of aspirin at PGE and CTAB-PGE is shown in the figure. At PGE (curve b) electrode, a poor electrochemical response and hence, weak oxidation peak is observed at a potential 0.8848 V with lower peak current. By comparison, with the oxidation of aspirin at CTAB-PGE (curve c) occurred at potential of 0.8714 V with highest peak current. The increase in the peak current at CTAB-PGE is due to the good electro catalytic activity of CTAB-PGE. These results suggest that an efficient catalytic oxidation reaction could be achieved at CTAB-PGE.

## Effect of scan rate

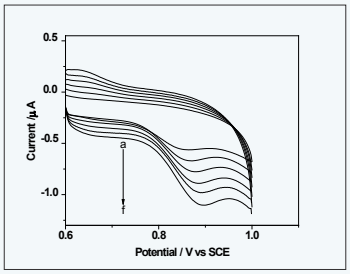


**Figure 3:** Cyclic voltammogram of 5mM Aspirin in phosphate buffer of $P^H$-7 at different scan rates (a→f, 50, 100, 150, 200, 250, 300 $mVs^{-1}$).

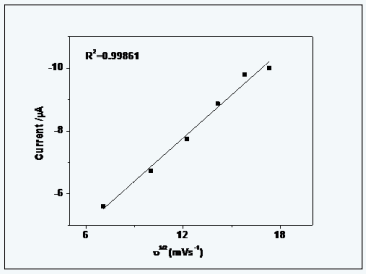


**Figure 4:** Effect of square root of scan rate variation for Aspirin at CTAB-PGE with 0.1M phosphate solution of $p^H$- 7.

The effect of the potential scan rate on the electro catalytic property of CTAB-PGE towards aspirin was studied by cyclic voltammetry. (Figures 3 & 4) shows that cyclic voltammogram of CTAB-PGE at varies scan rate (50-300 mVs-1). The effect of varying the potential scan rate on oxidation peak of 5mM aspirin was studied in phosphate buffer of pH-7. The increase in the scan rate increases the peak current. However, the oxidation current of aspirin increased linearly with the square root of scan rate with correlation coefficient (R2) of 0.99778 (Figure 3 & Table 1). This indicates the process is diffusion controlled in this region and the potential shifts towards positive. This is a typical behavior of an irreversible electron transfer process.

**Table 1:** Electrochemical parameters of Aspirin at different scan rate.

| Curves | Scan rate | $v^{1/2}(mVs^{-1})$ | $i_{pa}$ (µA) |
|---|---|---|---|
| a | 50 | 7.07 | -5.6090 |
| b | 100 | 10.00 | -6.7255 |
| c | 150 | 12.24 | -7.7460 |
| d | 200 | 14.14 | -8.8625 |
| e | 250 | 15.81 | -9.7870 |
| f | 300 | 17.32 | -11.0045 |

## Effect of concentration

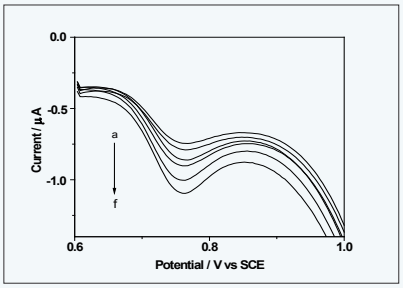


**Figure 5:** Differential pulse voltammogram of aspirin in 0.1M phosphate buffer solution in at CTAB-PGE at different concentration (a→f, 50-300µM).

The peak current of aspirin oxidation at the surface of CTAB-PGE was used for the determination of aspirin in solution. Therefore, (Figure 5) shows Differential Pulse Voltammetry (DPV) performed using 50 to 300µM aspirin in phosphate


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buffer of pH.7 at CTAB-PGE (Table 2). The DPV showed successive enhancement of peak current on increasing the aspirin concentration. The dependence of peak current on the concentration of aspirin is a linear relationship in the rate of 50 to 300μM with a correlation coefficient (R2=0.99327). This linearity helps us to detect the unknown concentration of aspirin in real samples.

**Table 2:** Electrochemical parameters of aspirin at different concentrations.

| Curves | Concentration of aspirin in μM | $I_{pa}$ in μA | $E_{pa}$ in volts |
|---|---|---|---|
| a | 50 | -0.7485 | 0.7676 |
| b | 100 | -0.7852 | 0.7659 |
| c | 150 | -0.8590 | 0.7625 |
| d | 200 | -0.8995 | 0.7608 |
| e | 250 | -1.0024 | 0.7600 |
| f | 300 | -1.0945 | 0.7508 |

## Real sample analysis

**Table 3:** Electrochemical parameters of aspirin with spike.

| Curves | Spike added in μL | $i_{pa}$ in μA | $E_{pa}$ in V |
|---|---|---|---|
| a | Tablet | -0.4550 | 0.7766 |
| b | Tablet+200 | -0.5308 | 0.7721 |
| c | Tablet+400 | -0.5791 | 0.7683 |

The practical utilization of CTAB-PGE for the detection of aspirin in the real sample was tested by determining the concentration of aspirin in pharmaceutical sample and the solution was used for CV studies as such (Table 3). An anodic peak was observed at 0.7766V and is attributed to the oxidation of aspirin (Figure 6) (curve 6a). To confirm anodic peak was due to aspirin, the sample was spiked with 200 a L and 400 a L of commercial aspirin and the resulting differential pulse voltammogram (shown in the fig curve 6b and curve 6c) an increase in the peak observed at 0.7766V was due to oxidation of aspirin.

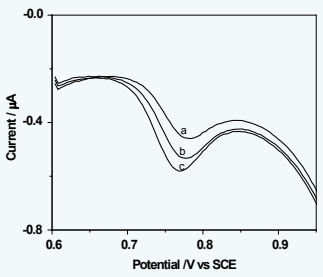


**Figure 6:** Differential pulse voltammogram of aspirin at CTAB-PGE (curve a ) and 1μM of tablet spiked with 0.1μM, 0.2μM of synthetic aspirin (curve b →c)

## Conclusion

In the present study voltammetric determination of aspirin in the aqueous solution of tablet using CTAB-PGE has been demonstrated. The CTAB-PGE exhibits potent and potential electro analytical behaviour towards the oxidation of aspirin. The result of this investigation shows that it is possible to perform electrochemical analysis of aspirin. The voltammetric technique was employed to study the electrochemical behaviour of the analyses. The voltammetric profiles demonstrate that the oxidation mechanism of aspirin is an irreversible electrochemical process followed by a slow chemical reaction of the electro oxidation products of the analysts. Voltammetric studies were utilized for this application, reveling its usefulness for quantification of the catecholamine in pharmaceutical products with high sensitivity and good repeatability. This study has also demonstrated the analytical utility of CTAB-PGE for qualitative and quantitative estimation of aspirin present in the real samples.

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