

*Full Paper*

## **Electrochemical Studies of Mesalazine at Sodium Dodecyl Sulfate Modified Carbon Paste Electrode: A Cyclic Voltammetric Study**

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**Abstract-** The electrochemical studies of mesalazine was performed at sodium dodecyl sulfate modified carbon paste electrode by cyclic voltammetric technique in 0.2 M phosphate buffer solution at pH 7.2 with a scan rate 100 mV/s. Sodium dodecyl sulfate modified carbon paste electrode showed an enhanced current signal as compared to the bare carbon paste electrode. Electrochemical parameters like effect of pH, scan rate and different concentration of mesalazine were investigated at sodium dodecyl sulfate modified carbon paste electrode. The effects of scan rate study revealed that the overall electrode process was found to be adsorption controlled processes at sodium dodecyl sulfate modified carbon paste electrode. The detection limit of mesalazine was found to be 0.238  $\mu$ M.

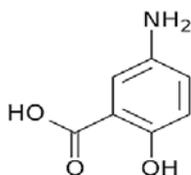
**Keywords-** Mesalazine, Sodium dodecyl sulfate (SDS), Cyclic voltammetry (CV)

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### **1. INTRODUCTION**

Mesalazine (5-aminosalicylic acid, 5-ASA) is the most commonly prescribed non-steroidal anti-inflammatory drug, used for the therapy of inflammatory bowel diseases (IBD) such as ulcerative colitis and Crohn's disease. Mesalazine can reduce leukotriene production and inhibit the cellular release of interleukin-1 [1]. Mesalazine inhibits cell injury in the

inflamed mucosa by scavenging reactive oxygen metabolites, thus suppressing their toxicity. Mesalazine acts by blocking the production of prostaglandins and leukotrienes, inhibiting bacterial peptide-induced neutrophil chemotaxis and adenosine-induced secretion, scavenging reactive oxygen metabolites [2-4]. Mesalazine is structurally correlated to the salicylates; the chemical structure of mesalazine as shown in scheme 1.



**Scheme 1.** Structure of mesalazine

The surfactants are linear, surface active agents containing hydrophilic head on one side and hydrophobic tail on other. Sodium dodecyl sulfate is a class of anionic surfactant. Currently surfactant modified electrodes have magnetize the researchers in electroanalysis due to their novel physical and chemical properties [5-8]. Surfactants were introduced in to electrochemistry to improve the detection limits of some biomolecules by Hu's group; they proposed an adsorption mechanism to interpret the enhancement effects of surfactants. Surfactants might combine with the substrate in certain forms and strengthened their accumulation process on the electrode surface, which facilitated the electron transfer between the electrode and solution [9,10]. Zheng and Zhau reported that sodium dodecyl sulfate formed a mono-layer on the surface of carbon paste electrode with a negative charged end directed outside the electrode [11]. Chengguo Hu et al. Hu and Bard have discussed, the adsorption of sodium dodecyl sulfate on both charge-regulated and hydrophobic substrates using atomic force microscopy measurement [12]. The results showed that the electrochemical responses of these compounds were greatly enhanced in presence of trace amount of surfactants [9,13,14]. Due to their unique molecular structure, surfactants have several applications in electrochemistry like electrocatalysis [15], electroanalysis [16], electroplating [17], corrosion [18] and fuel cells [19] have been widely reported. Amphiphilic or amphipathic nature of surfactant can alter the properties of the electrode-solution interactions; the interactions at the interface influence the electrochemical processes of electroactive analytes [20,21]. The immobilization of surfactant on the surface of carbon paste electrode significantly facilitates electron transfer from electroactive species to the electrode by enhancing the redox peak current of analyte and it improves the sensitivity of the electrode, related work have been carried out by Swamy *et al* [22-27] and surfactant modified carbon paste electrode can successfully used for the electroanalysis of some biomolecules and drugs [28-31].

Presently number of various analytical methods were depicted in the literature for the detection of mesalazine in pharmaceuticals and biological matrices, such as

spectrophotometry [32], High-performance liquid chromatographic methods with UV [33], fluorescence spectroscopy [34], colorimetry [35], automated chemiluminescence [36] and ultra-performance liquid chromatography [37], mass spectrometry detection [38, 39], electrochemical methods [40, 41].

Electrochemical methods traditionally gaining more attention due to their simplicity and fast response, the carbon paste electrodes are very popular due to their interesting properties such as chemical inertness, low cost; ease fabrication, wide potential window. In order to enhance the sensitivity of the carbon paste electrode, modification of electrode was attempted. The modified carbon paste electrodes possess many advantages like high sensitivity, low cost, ease of fabrication and rapid renewal, inexpensive [42-50].

In the present work, a carbon paste electrode was prepared by mixing graphite powder and silicone oil, to enhance the sensitivity of electrode sodium dodecyl sulfate was immobilized on the surface of carbon paste electrode. The fabricated sodium dodecyl sulfate modified carbon paste electrode was used for electrochemical studies of mesalazine and the fabricated electrode produced enhanced voltammetric response as compared to the unmodified electrode.

## **2. EXPERIMENTAL SECTION**

### **2.1. Apparatus**

Electrochemical measurements were performed on a model CHI-660c (CH Instrument-660 electrochemical workstation). All electrochemical experiments were carried out in a conventional three electrode cell. Platinum wire was employed as a counter electrode, a saturated calomel electrode served as a reference electrode, bare carbon paste electrode and modified carbon paste electrode employed as a working electrode.

### **2.2. Reagents and Chemicals**

Mesalazine was received from Himedia, sodium dodecyl sulfate, silicone oil were received from Merck. Graphite powder was purchased from Loba chemicals, sodium dodecyl sulfate (SDS) was dissolved in double distilled water. In all the measurements, phosphate buffer solution was used as supporting electrolyte; it was prepared by mixing standard stock solutions of 0.2 M disodium-hydrogen phosphate and sodium dihydrogen phosphate by adjusting the pH. All chemicals were of analytical grade quality and were used as received.

### **2.3. Preparation of bare carbon paste electrode**

The bare carbon paste electrode (BCPE) was prepared by manual grinding graphite powder and silicone oil at a ratio of 70:30 (w/w) in an agate mortar for about 30 minutes to

get a homogeneous paste. The obtained small quantity of paste was carefully incorporated into the cavity of a homemade electrode and then the electrode was polished on a weighing paper to get smooth surface.

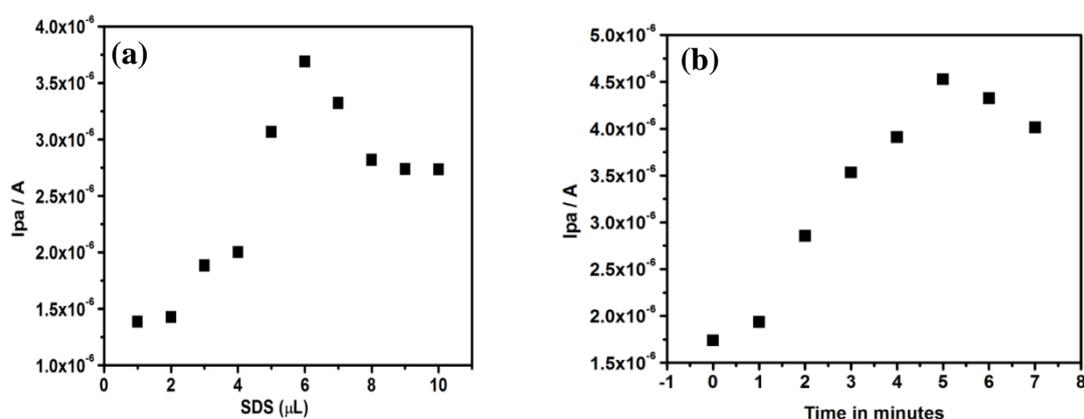
#### 2.4. Preparation of sodium dodecyl sulfate modified carbon paste electrode

A sodium dodecyl sulfate modified carbon paste electrode was prepared by immobilizing sodium dodecyl sulfate on the surface of the carbon paste electrode for 5 minutes, later the electrode was gently rinsed with distilled water to remove the unabsorbed sodium dodecyl sulfate and dried over air at room temperature.

### 3. RESULTS AND DISCUSSION

#### 3.1. Effect of concentration of sodium dodecyl sulfate and immobilization time on voltammetric response of mesalazine

In order to optimize the experimental conditions, influence of concentration of surfactant and immobilization time variation studies were performed, even the trace amount of surfactant enhances the current signal of analyte. The different concentration of sodium dodecyl sulfate solution was placed on the surface of carbon paste electrode, which is known as immobilization method, the surfactant diffuses in to the carbon paste electrode along with analyte, it results enhanced current signal.



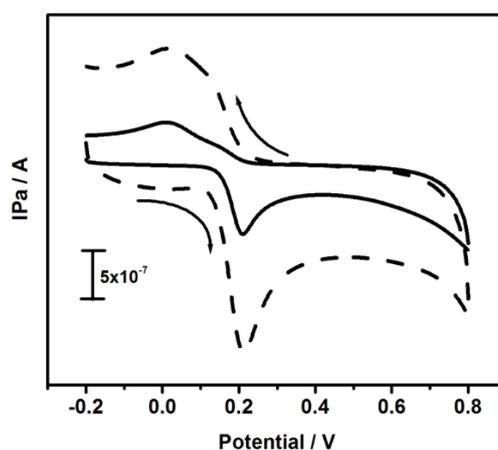
**Fig. 1.** (a) Graph of anodic current versus different concentration of sodium dodecyl sulfate at carbon paste electrode; (b) Graph of anodic peak current versus immobilization time

The Figure 1a shows that the graph of anodic peak current of mesalazine versus different concentration of sodium dodecyl sulfate, and it demonstrates the dependency of current signal on the concentration of sodium dodecyl sulfate. The current signal of mesalazine was increased gradually from 1 to 6  $\mu\text{L}$  and then decreased from 7 to 10  $\mu\text{L}$ . The maximum peak current obtained at 6  $\mu\text{L}$  SDS therefore 6  $\mu\text{L}$  surfactant was chosen for further analysis.

The effect immobilization time variation was studied to get better sensitivity of mesalazine. The 6  $\mu\text{L}$  sodium dodecyl sulfate was immobilized on the surface of carbon paste electrode then the immobilization time was varied from 0 to 7 minutes. The Figure 1b shows the anodic peak current of mesalazine at different immobilization time. The current response of mesalazine at modified electrode increased gradually with increase in immobilization time and reaching a maximum current response at 5 minutes later decreases with further increase of immobilization time. Therefore immobilization time 5 minutes was selected for further analysis.

### 3.2. Electrochemical performance of mesalazine at sodium dodecyl sulfate modified carbon paste electrode

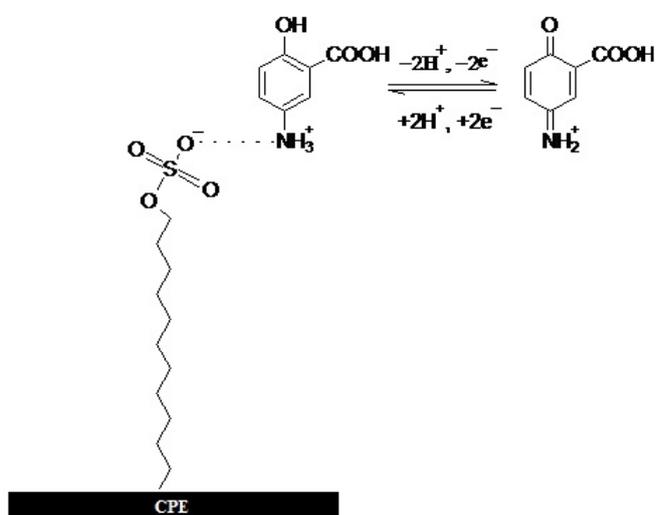
The cyclic voltammograms of mesalazine was recorded at bare carbon paste electrode and sodium dodecyl sulfate modified carbon paste electrode. The figure 2 shows the electrochemical response of 20  $\mu\text{M}$  mesalazine in 0.2 M phosphate buffer solution as a supporting electrolyte of pH 7.2 with the scan rate of 100 mV/s at bare carbon paste electrode (solid line) and sodium dodecyl sulfate modified carbon paste electrode (dashed line). The mesalazine is electroactive compound, easily undergoes redox reaction and results voltammetric signal.



**Fig. 2.** Cyclic voltammograms of 20  $\mu\text{M}$  mesalazine in 0.2 M phosphate buffer solution of pH 7.2 with scan rate of 100 mV/s at bare carbon paste electrode (solid line), sodium dodecyl sulfate modified carbon paste electrode (dashed line)

The sensitivity of voltammetric response at bare carbon paste electrode was low as compared to sodium dodecyl sulfate modified carbon paste electrode. The voltammetric response was apparently enhanced at sodium dodecyl sulfate modified carbon paste electrode

reflected by the increment of anodic peak current and cathodic peak current of mesalazine. The trace amount of surfactant on the surface of carbon paste electrode enhanced the electrochemical response of analyte [51,52]. The enhanced peak current is due to the interaction between mesalazine and sodium dodecyl sulfate surfactant. Surfactant may be adsorbed on the electrode surface to form surfactant film, which intern influence the rate of electron transfer, sodium dodecyl sulfate mono-layer on the surface of carbon paste electrode with a negative charged end directed outside the electrode and react with the positively charged mesalazine results is enhanced the rate of electron transfer [11.27]. The schematic representation of electrochemical interaction of sodium dodecyl sulfate and mesalazine are shown in scheme 2.

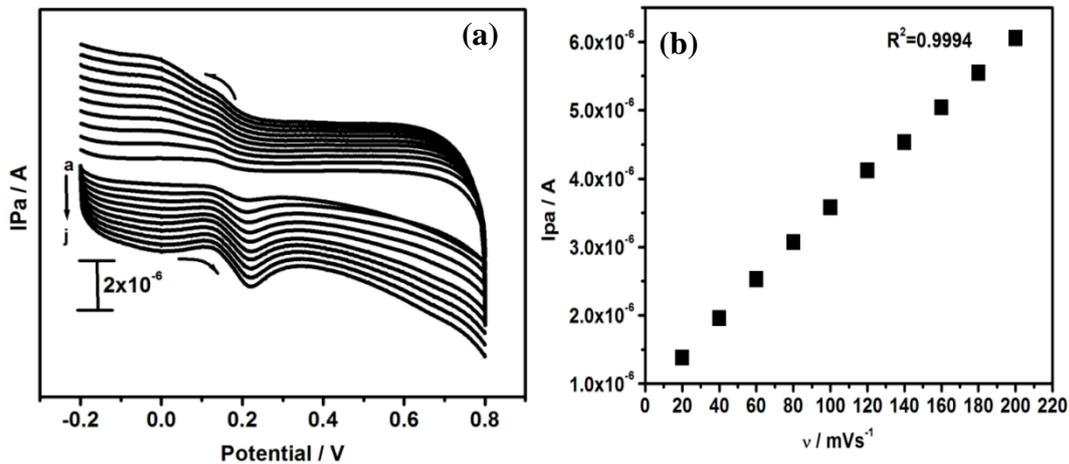


**Scheme 2.** Electrochemical reaction of mesalazine at sodium dodecyl sulfate modified carbon paste electrode

### 3.3. Effect of scan rate on the oxidation of mesalazine

The dependence of anodic and cathodic peak current response of mesalazine on scan rate was scrutinized by varying the scan rate at sodium dodecyl sulfate modified carbon paste electrode. The figure 3a shows the cyclic voltammograms of 20  $\mu\text{M}$  mesalazine in 0.2 M phosphate buffer solution over the sweep rate range from 20 to 200 mV/s at sodium dodecyl sulfate modified carbon paste electrode. The redox peak current of mesalazine was gradually increased with increase of scan rate from 20 to 200 mV/s. The experimental results authenticate the dependency of scan rate and redox peak current of mesalazine. The figure 3b shows the plot of anodic peak current ( $I_{pa}$ ) values versus scan rate, this plot is composed of a straight line. A linear correlation was obtained between the scan rate and anodic peak current,

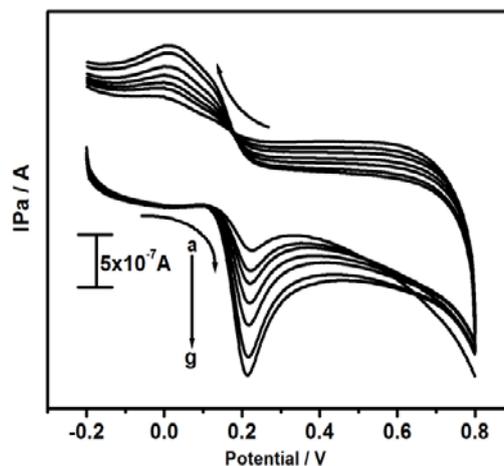
it indicates the magnitude of peak current as a function of scan rate. The anodic peak current of mesalazine was raised linearly with increase in scan rate with correlation coefficient value 0.9994 and it indicates that the reaction of mesalazine at sodium dodecyl sulfate modified carbon paste electrode was adsorption controlled process [53].



**Fig. 3.** (a) Cyclic voltammograms of mesalazine at different scan rate; (b) Graph of anodic peak current versus scan rate

### 3.4. Effect of concentration of mesalazine at sodium dodecyl sulfate modified carbon paste electrode

The sensitivity of the electrode was evaluated by preparing different concentration of mesalazine solution, the figure 4 shows the cyclic voltammograms for various concentrations of mesalazine from 10 to 70  $\mu\text{M}$  at sodium dodecyl sulfate modified carbon paste electrode in 0.2 M phosphate buffer solution at pH 7.2 with the scan rate of 50 mV/s.

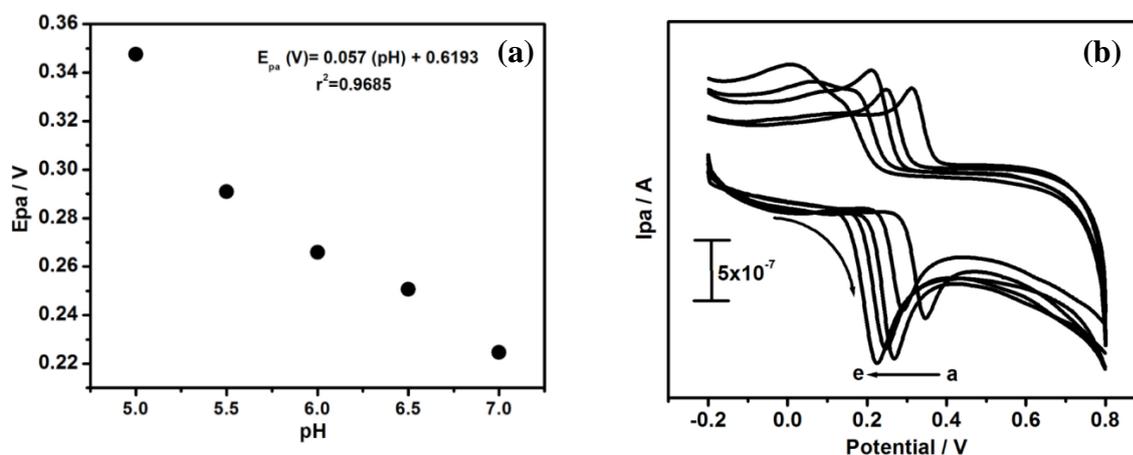


**Fig. 4.** Cyclic voltammograms of different concentration of mesalazine at sodium dodecyl sulfate modified carbon paste electrode in 0.2 M PBS of pH 7.2

The successive enhancement of redox peak current on increasing mesalazine concentration was achieved and the linearly increased anodic and cathodic peak current indicates that the magnitude of anodic and cathodic peak current of analyte was directly proportional to their concentration.

### 3.5. Effect of pH at sodium dodecyl sulfate modified carbon paste electrode

The electrochemical behavior of the analyte is influenced by pH of supporting electrolyte solution, the peak potential of analyte generally pH dependent. The potential diagram was constructed by plotting a graph of anodic peak potential versus different pH value as shown in figure 5a, the oxidation peak potential shifted towards negative direction with increase of pH value of supporting electrolyte indicates that the protons were takes place in electrochemical reaction of mesalazine. The figure 5b shows cyclic voltammograms of mesalazine for different pH (from 5 to 7) at sodium dodecyl sulfate modified carbon paste electrode. A linear relationship was found between peak potential of mesalazine and pH of the electrolyte over a range from 5 to 7 with a slope of 57 mV/pH, it indicates that an equal number of protons and electrons take part in the electrochemical reaction of mesalazine [54,55].



**Fig. 5.** (a) Graph of  $E_{pa}$  versus pH at sodium dodecyl sulfate modified carbon paste electrode; (b) Cyclic voltammograms of mesalazine for different pH (from 5 to 7) at sodium dodecyl sulfate modified carbon paste electrode

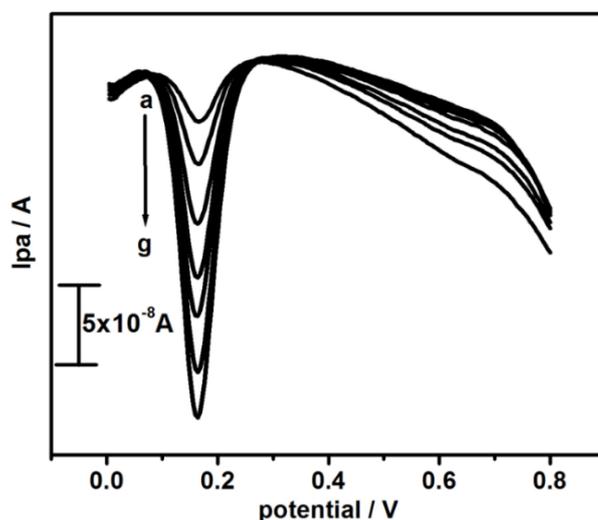
### 3.6. Differential pulse voltammetry of mesalazine

In order to investigate the sensitivity of the modified carbon paste electrode, the differential pulse voltammetric study of mesalazine in 0.2 M phosphate buffer solution at sodium dodecyl sulfate modified carbon paste electrode was conducted. The figure 6 shows the anodic peak current of mesalazine increase linearly with mesalazine concentration in the range from 1 to 7  $\mu$ M. The anodic peak current directly proportional to the concentration of

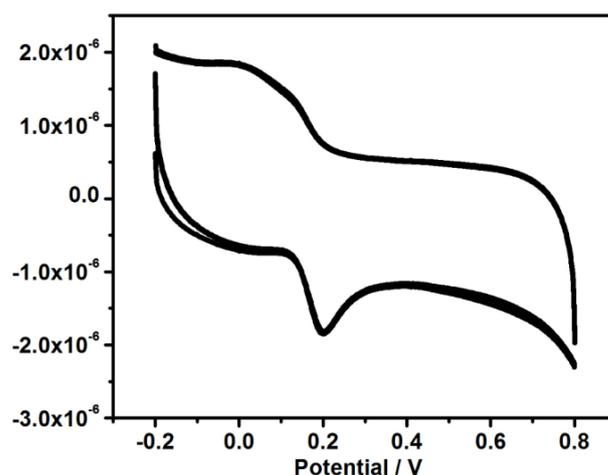
mesalazine, it indicates the peak current and concentration of analyte are dependent each other. This study reveals the sensitivity of the modified carbon paste electrode towards the detection of mesalazine. The detection limit was calculated by using equation (1) [56].

$$\text{LOD}=3S/M \quad (1)$$

Where, S is the standard deviation and M is the slope obtained from the calibration plot. The detection limit of mesalazine was found to be  $0.238 \mu\text{M}$ .



**Fig. 6.** Differential pulse voltammograms of mesalazine from 1 to 7  $\mu\text{M}$  in 0.2 M phosphate buffer solution at sodium dodecyl sulfate modified carbon paste electrode



**Fig. 7.** Cyclic voltammograms for 20 multiple cycle of mesalazine at sodium dodecyl sulfate modified carbon paste electrode

### 3.7. Electrochemical investigation of stability of sodium dodecyl sulfate modified carbon paste electrode

The stability of the fabricated electrode was tested for sensitive determination of mesalazine drug; electrochemical behavior of mesalazine at fabricated electrode for 20 cycles was recorded by cyclic voltammetry. The figure 7 shows the redox behavior of mesalazine at fabricated electrode and it seen that the anodic peak current and cathodic peak currents were fairly stable. The experimental result showed excellent stability of sodium dodecyl sulfate modified carbon paste electrode. It suggests that peak currents of mesalazine in electrochemical determination at sodium dodecyl sulfate modified carbon paste electrode were quite steady.

### 3.8. Real sample analysis

The fabricated sodium dodecyl sulfate modified carbon paste electrode was applied for the analysis of mesalazine in commercial tablet to evaluate the practical utility of the electrode. The electrochemical response at sodium dodecyl sulfate modified carbon paste electrode for the spiked standard solution of mesalazine was recorded and calculated % recovery. The results were tabulated in table 1, the acceptable recoveries were obtained in the range of 97.89 to 99.60%. This result indicates that the sodium dodecyl sulfate modified shown changed to carbon paste electrode shows high sensitivity with selectivity for detecting mesalazine in pharmaceutical sample.

**Table 1.** Detection of mesalazine in tablet (N=3)

Content	Mesalazine added (M)	Found (M)	Recovery (%)
mesalazine tablet	$0.10 \times 10^{-4}$	$0.0980 \times 10^{-4}$	98.07
	$0.20 \times 10^{-4}$	$0.1957 \times 10^{-4}$	97.89
	$0.30 \times 10^{-4}$	$0.2964 \times 10^{-4}$	98.81
	$0.40 \times 10^{-4}$	$0.3984 \times 10^{-4}$	99.60

## 4. CONCLUSION

The present work demonstrates the fabrication of electrode by incorporating a anionic surfactant sodium dodecyl sulfate as a modifying species; the sodium dodecyl sulfate modified carbon paste electrode enhanced the current signals as compared to the bare carbon paste electrode. The oxidation peak current showed a linear dependency on the concentration of mesalazine. The pH study suggests that equal number of protons and electrons are involved during the electrochemical reaction of mesalazine. The modified electrode shows the stability, sensitivity and reproducibility towards the determination of mesalazine, this

study expands the application of surfactant in electroanalytical chemistry for the detection of drug. The modified electrode can be used as an analytical tool for the determination mesalazine in pharmaceutical preparations.

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