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Electroanalytical and UV- spectroscopic study for analysis of Nimesulide in pharmaceutical samples

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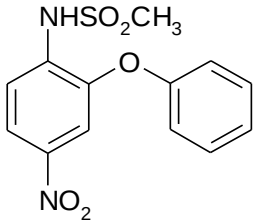
Abstract
Analytical Chemistry is very important in developing various drug compounds from their synthesis stage to marketing and it is an important process of drug development and analysis. In this study Nimesulide is analyzed using electroanalytical technique like differential pulse polarography and UV-Spectroscopy. Optimized conditions for these two techniques were determined. The methods were developed used for detection of Nimesulide. The developed DPP process can be applied for calibration and estimation of Nimesulide in tablets. It is concluded that the proposed method differential pulse polarography method is considered to be sensitive and provide shorter time for analysis than UV technique.

Keywords: polarography, Nimesulide, electroanalytical, accuracy, optimization

1. Introduction
Analytical Chemistry has very important role in developing various drug compounds from their synthesis stage to marketing since it is an important process of drug development and analysis [1]. The instrumental techniques for quantization of drugs and tablets, categorized into four fundamental types like chromatography, Spectrophotometry, electrochemical studies and radiometric analysis [2]. Electroanalytical chemistry had its basis in the last eight decades accompanying the uses of oxidation and reduction reactions.

Nimesulide is a new non-steroid anti-inflammatory pain-relieving drug [8]. Nimesulide appears to provide a convenient alternative for other nonsteroidal anti-inflammatory drugs during the treatment of peoples suffering with inflammatory conditions or ache and fever [2, 3].

Voltammetric techniques has become an accepted device for studying electrochemical reactions [4]. Pharma formulation industries and biological aids are established on the basis of HPLC, LC/MS, Spectroscopy and Microbiological assessment [5, 6]. However, there are certain problems while using these techniques. Since all above techniques needs very expensive instrumentation, delicate and have high running cost, therefore the use of simple, rapid, and cheap, sensitive, electrochemical method of working can be interesting alternative, particularly those established on the basis of electroanalytical techniques [6-8]. The structure of Nimesulide is,



Nimesulide is non-steroid anti-inflammatory analgesic drug which has been available in India since 1997. It has been given orally or rectally two times in a day basis to a number of inflammatory and pain states [9].

Many chromatographic techniques have been employed for the estimation of nimesulide such as LC, HPLC, HPLC/MS, HPTLC and reversed-phase HPLC [10-13].

The present study comprises the voltammetric estimation of pharmaceutical drugs of analgesic class [13-18]. The objectives of present study are, to investigate the electroanalytical behavior of Nimesulide by differential pulse polarography, the proposed electrochemical reduction /

oxidation mechanism of Nimesulide at DME and to develop rapid, sensitive, selective economic and accurate method and Method validation according to ICH guidelines [19-22].

2. Experimental

2.1 Equipment

Polarographic analyzer CL-362 model having PC within its RS 232C interface by using windows-based software ELICO was used for polarographic estimations as used in chapter 1. DME was used like the working electrode, SCE as reference electrode and platinum wire was used like an auxiliary electrode. Elico pH meter utilized for measuring pH. UV-VIS spectrophotometer (Perkin Elmer Lambda 25) for measuring the absorbance of nimesulide in acetate buffer solution.

2.1.1 Polarographic Cell

In polarography method the system of three electrode was used. These three electrodes are WE, RE and AE. The overall arrangement consisting of a polarographic cell having varying capacity of volume and there is a gas line to purge and cover the electrolytic solution.

2.1.2 Chemicals and Reagents

Commercially available nimesulide tablets were obtained and all other chemicals like glacial acetic acid, sodium acetate, ammonium chloride orthophosphoric acid, and sodium hydroxide, hydrochloric acid, sodium bicarbonate and sodium bicarbonate solution were used of analytical grade purchased from Merck. Standard solution of nimesulide was prepared by suitable dilution of stock solution using 0.1 M NaOH solution. All the chemicals utilized were of AR quality grade and all solutions prepared in deionized water.

2.1.3 Glassware

All glassware scrupulously washed and rinsed to reduce interference complications. Initially, it was cleaned rigorously using a washing powder and a bottle brush and cleaned with distilled water.

2.1.4 Preparation of Buffer Solution

Buffer solutions like BRB, Carbonate, Phosphate, Acetate buffers were prepared were prepared using standard methods.

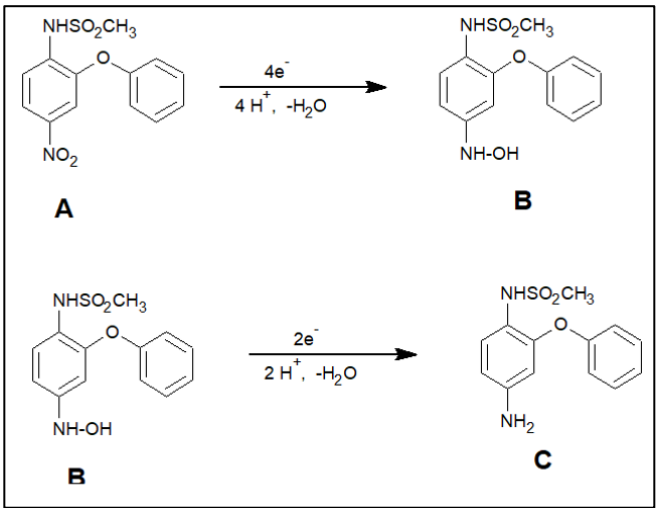
3. Results and Discussions

3.1 Optimization of conditions for DPP

3.1.1 Effect of Concentration of Supporting Electrolyte

The effect of variable concentration of NaOH which is the supporting electrolyte was studied, the result shows that NaOH having concentration 0.1 M, gives the high Ip of nimesulide on comparison with another concentrations [6, 12]. The Figure 1 shows DPP polarogram of nimesulide in NaOH as a supporting electrolyte with different concentration. From figure, it is clear that nimesulide (3.2 ppm) with 0.1 M NaOH is the most acceptable concentration for determination of nimesulide by DPP.

Proposed Reaction Mechanism of Nimesulide is



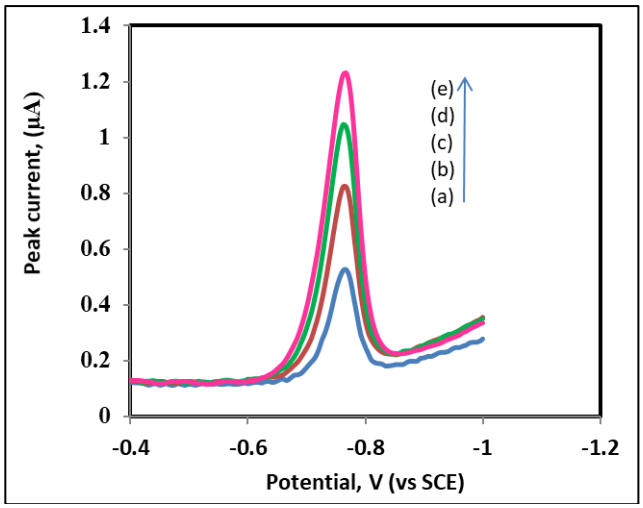


Fig 1: DPP of increase in concentration of nimesulide on peak height in 0.1 M NaOH; (a) 3.2 ppm (b) 6.4 ppm (c) 9.6 ppm and (d) 12.8 ppm, obtained at current range = 10 µA, data acquisition = fast, scan rate = 6 mV/sec, drop time = 1sec, scan type = forward, pulse amplitude = 50 mV

Table 1: Optimum parameters of 10.0 µg $ml^{-1}$of three nimesulide

Sr. No.	Parameters	Nimesulide
01	Supporting electrolyte	0.1M NaOH
02	Pulse amplitude(mV)	50
03	Scan rate (mV/sec)	6.0
04	Current range (µA)	10.0
05	Drop time (sec)	1.0
06	Scan type	Forward
07	CC Compensation (%)	0.0

3.2 Analysis of Nimesulide

3.2.1 Calibration curve of Nimesulide by DPP

The calibration curve for the estimation of nimesulide was determined by using proposed method[6]. The Ip of nimesulide with increase in concentration is given in Figure 2 This shows that when maximum value of Ip is achieved, it keeps to level off with more addition of nimesulide standard solution as predicted for method which was restricted by analyte [12, 19]. The DPP and the linear graph of Ip vs. concentration of nimesulide are as given in Figure. 2 and 3 respectively.

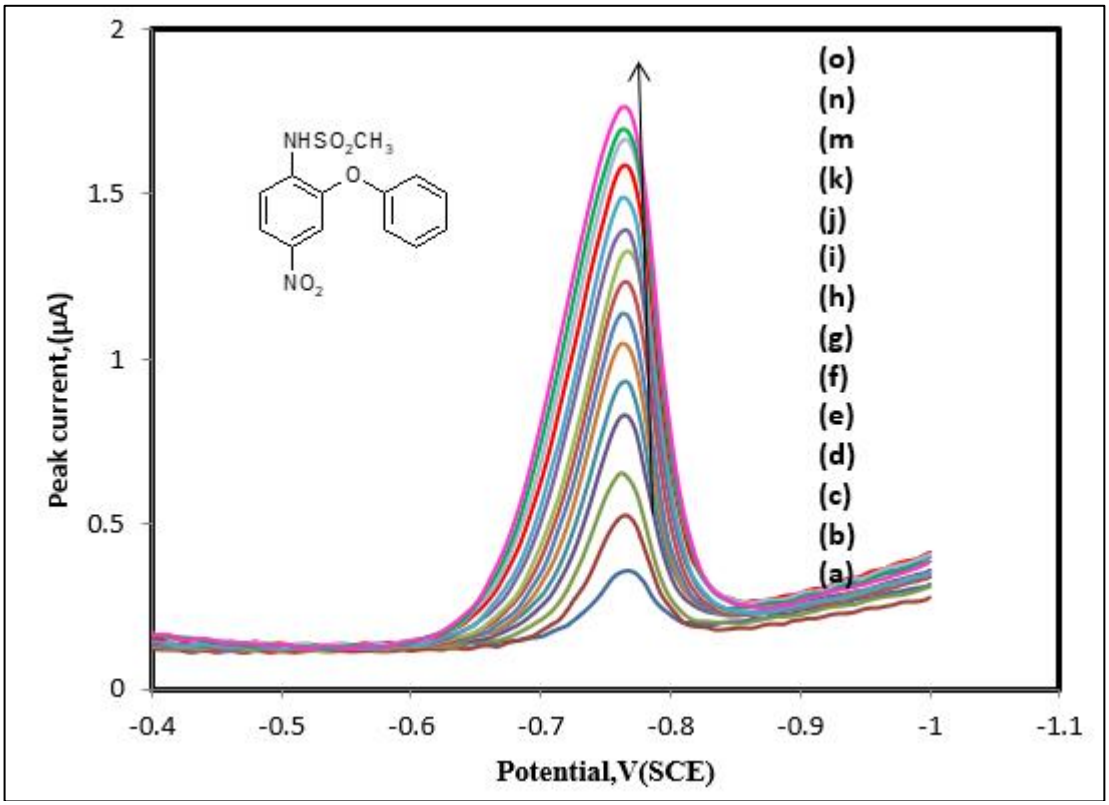


Fig 2: DPP of Nimesulide in 0.1 M NaOH solution as an electrolyte at a) 1.5 ppm b) 3 ppm c) 4.5 ppm d) 6 ppm e) 7.5 ppm f) 9 ppm g)10.5 ppm h)12 ppm i)13.5 ppm j)15 ppm k)16.5 ppm l)18 ppm m) 19.5 ppm n) 21 ppm o)22.5 ppm.

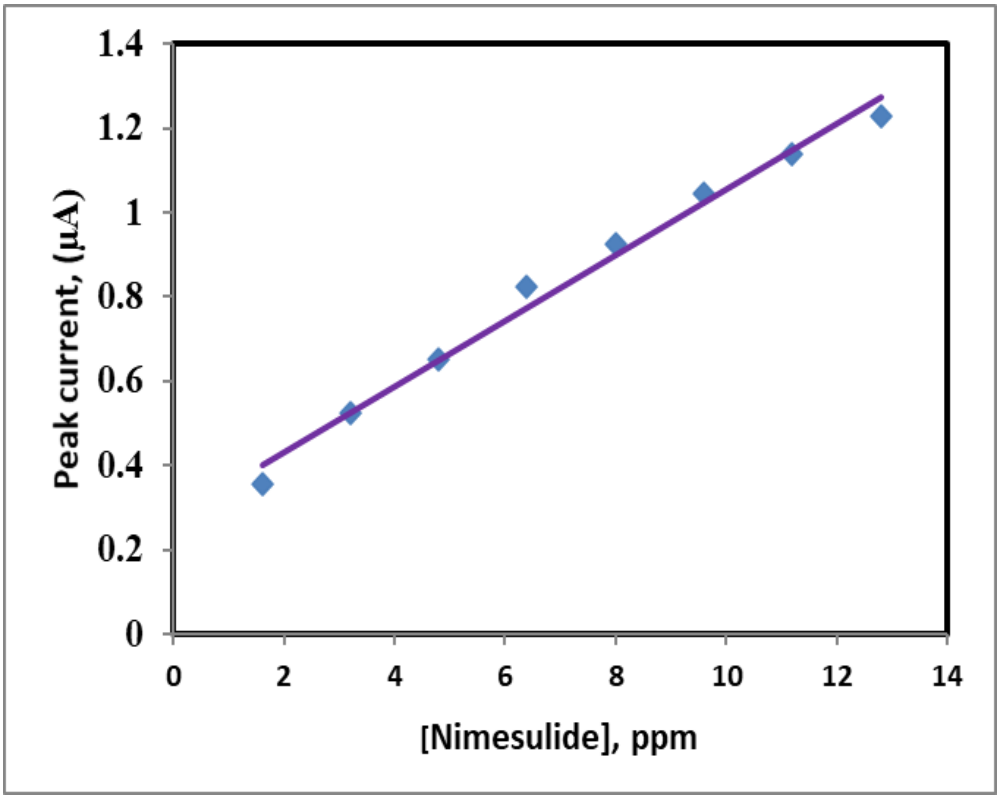


Fig 3: Linear graph of Ip vs. conc. of nimesulide in 0.1 M NaOH solution as a supporting electrolyte This indicates that range of linearity was obtained from 1.5 ppm to 14.0 ppm with LOD 1.5 µg ml^{-1} and LOQ 5.1 µg ml^{-1}. The value of R^2 was 0.996.

3.2.2 Calibration Curve of Nimesulide by UV Spectrophotometric Method

Spectroscopic studies of Nimesulide were carried out on the same abridgment. UV spectrophotometer is simple, fast and sufficiently sensitive process for analysis of nimesulide.

To study the optical characteristics of the investigated compounds, the corresponding spectra were recorded in 0.1 M NaOH solution within wavelength range 200–400 nm. Representative spectra of nimesulide are shown in Figure. 3.11. The spectra of nimesulide have two discrete absorption bands with maxima at 393.45 nm.

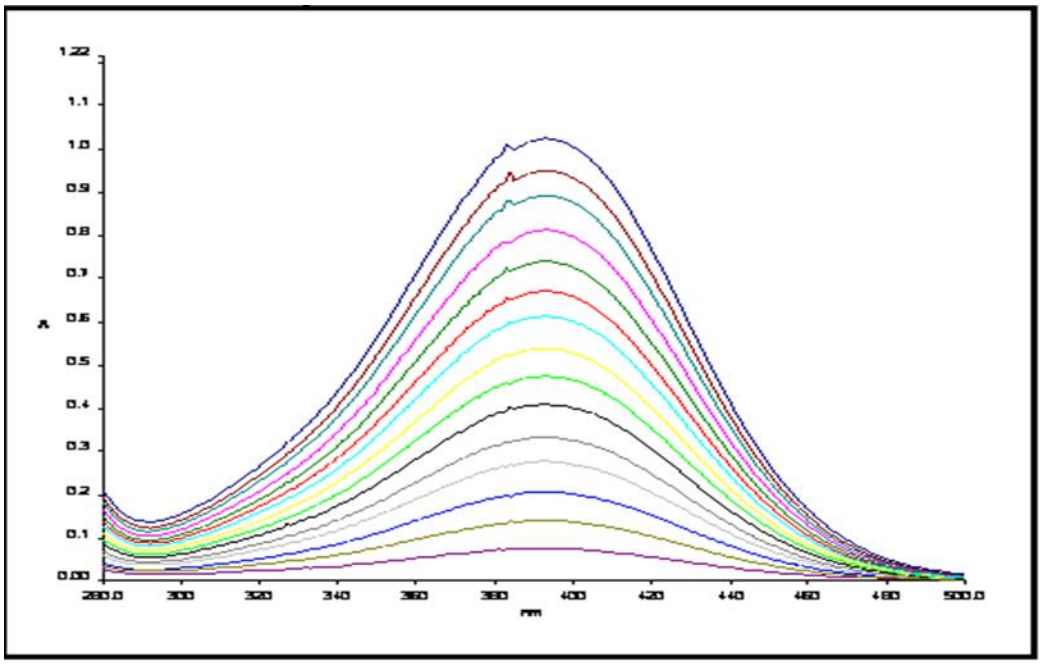


Fig 4: UV Spectrum of increasing concentration of Nimesulide in 0.1 M NaOH a) 1.5 ppm b) 3.0 ppm c) 4.5 ppm d) 6.0 ppm e) 7.5 ppm f) 9.0 ppm g) 10.5 ppm h) 12.0 ppm i) 13.5 ppm j) 15 ppm k) 16.5 ppm l) 18 ppm m) 19.5 ppm n) 21 ppm o) 22.5 ppm.

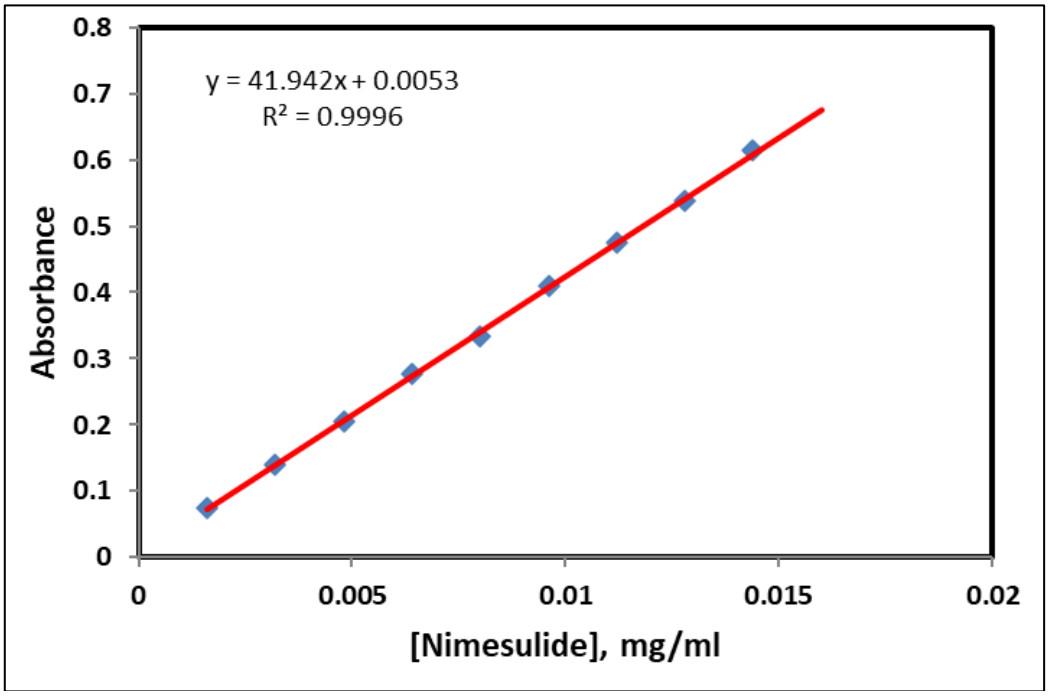


Fig 5: Linear graph of absorbance vs. conc. of nimesulide in 0.1 NaOH solution. It shows that linearity range was obtained from 1.5 ppm to 14.0 ppm with a LOD 0.28 µg ml^{-1} & LOQ 0.933 µg ml^{-}. The value of R^2 0.994.

3.2.3 Determination of precision

To determine correctness of the proposed DPP method, it was evaluated from 3 independent quantifications i. e. 3.0, 6.0 and 9.0 ppm of nimesulide solution on one and the same day, to get RSD of 0.30% 0.21% and 0.21% respectively. The precision was satisfactory because the average values do not surpass 15% of the coefficient of variance (CV). For the interday i. e. within 3 days, the accuracy was determined, RSD for first, second and third day obtained for evaluation of 3.0 ppm were 0.30%, 0.30% and 0.69% respectively and for 6.0 ppm were 0.21%, 0.41% and 0.34% and for 9.0 ppm were 0.21%, 0.33% and 0.21%. The results obtained after precision study for intra and interday are given in Table 2.

Table 2: Ip (µA) for intra and interday precision study 3.0, 6.0 and 9.0 ppm by developed DPP method. (n=3)

Nimesulide ppm	Intraday Detection Ip ± SD (%RSD)	Interday Detection Ip ± SD (%RSD)		
		Day 1	Day 2	Day 3
2	0.4223 ± 1.94 (% 1.23)	0.4223 ± 1.94 (% 1.23)	0.424±0.0005 (% 0.11)	0.423±0.0005 (% 0.11)
4	0.539 ± 0.0011 (% 0.22)	0.539 ±0.0011 (% 0.22)	0.548 ±0.001 (% 0.18)	0.543 ± 0.0015 (% 0.28)
6	0.653 ± 0.0021 (% 0.36)	0.653 ±0.0021 (% 0.36)	0.659 ± 0.0013 (% 0.28)	0.6589±0.0014 (% 0.23)

3.2.4 Determination of accuracy

Accuracy was evaluated by determining the proportion of relative error between the measured average and added concentrations. The recoveries observed were 99.34 ± 0.416%, 99.51 ± 0.721% and 99.28 ± 0.35% respectively, given in Table 3. Result conveys that recovery of nimesulide standard was considered as superior.

Table 3: Mean values of recovery of standard solution of Nimesulide (n = 3)

Expt. No.	Quantity Added Ppm	Peak Current Ip (µA)	Quantity Found ppm	% Recovery	% Recovery±*SD* (RSD)
1	3.2	0.5220 0.5190 0.5215	3.169 3.189 3.170	99.11 99.71 99.17	99.33±0.416 (0.42%)
2	6.4	0.766 0.770 0.769	6.39 6.33 6.31	99.91 99.21 98.19	99.45±0.721 (0.72%)
3	9.6	1.018 1.020 1.019	9.56 9.51 9.52	99.61 99.11 99.15	99.29±0.35 (0.36%)

4. Conclusion

In this study DPP and UV techniques were employed for study of Nimesulide. DPP curves were recorded in different electrolytes out of that NaOH was found to be best suited for detection of nimesulide. The developed DPP process can be applied for calibration and estimation of nimesulide in tablets. It is concluded from the LOD and LOQ values of both the techniques that the proposed method differential pulse polarography method is considered to be sensitive, and provide shorter time for analysis as compared with UV technique.

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