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Evaluation of Diclofenac by UV-Vis Spectrophotometer in Some Locally Produced Tablets

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Abstract

Diclofenac is a common and randomly used pharmaceutical product, was evaluated for its active ingredient by UV-Vis spectrophotometer at 282 nm. Diclofenac sodium of 50 mg dose of seven different companies of Bangladesh was extracted from the tablets, cleaned up and the active ingredient was evaluated. Evaluation was carried out with respect to calibration curve of standard diclofenac sodium. Amount of diclofenac in 50 mg tablets of seven different companies were found to be in the range of 47.91 ± 0.90 to 58.52 ± 0.41 mg. Recovery experiments were done by spiking excipient of the medicine at two different concentration levels with 5-7 replicate studies. Correlation coefficient (r^2) was found to be 0.9974 and the recovery was103.39 ± 3.93 to 107.96 ± 3.56 % for the drug.

Key words: Diclofenac, Absorption, Correlation Coefficient.

I. Introduction

Diclofenac [Fig. 1. 2-(2,6-dichloranilino) phenylacetic acid] is a nonsteroidal anti-inflammatory drug[1,2,3] (NASID) and widely prescribed for the treatment of rheumatoid arthritis[4,5], osteoarthritis[6], dysmenorrhea, musculoskeletal. injuries[3,7] and post-surgery analgesia in human and veterinary medicine[2,7].

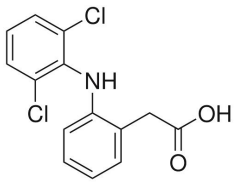

Fig. 1. Structure of diclofenac

Due to large demand, various pharmaceutical companies are now manufacturing diclofenac using their own brand name in Bangladesh. Pharmaceuticals sector is one of the fastest growing sectors in Bangladesh which is exporting medicines to the global market including European countries.

Proper dose of the drugs are very important to get relief of the complicacy for less side effects, otherwise proper control of the diseases are not possible. Diclofenac is essential medicine and is produced by local companies to make them available for all the citizens. It is a non-prescribed drug and also available in the developed countries. Quality Assurance (QA) and Quality control (QC) of all the pharmaceutical companies of our country are not at the same levels. This paper describes an easy and cheaper method for assay of raw materials and finished products of diclofenac.

II. Materials and Methods

Sample collection

Commercial tablets of diclofenac produced by different pharmaceutical companies (National and multinational) were purchased from local pharmaceutical stores of Dhaka city (D1 - D7).

Chemicals and reagents

Extra pure methanol (Merck, KGaA, 64271, Darmstadt, Germany) was used to carry out the study. Certified diclofenac sodium (99.70 % w/w) was obtained through courtesy of ACI Pharmaceutical Limited.

Instruments

A double beam Ultraviolet-visible spectrophotometer (Shimadzu, UV-1800), an analytical balance (AL 104, Mettler Toledo), a vortex machine (Kebo LabReax-2000) and a centrifuge machine (Cowbell) were used.

Preparation of standard solutions

Stock solution (500 µg/mL and working standard solutions (50, 40, 20, 10, 5, 3.5, 3.0, 2.5 and 2.0 µg/mL) of diclofenac sodium were prepared in methanol. Absorptions of working standard solutions of the medicine were measured; calibration curves were drawn by plotting absorption vs concentration graph (Fig. 2) and limit of detection and limit of quantification were found out.

Extraction and clean-up of active ingredients

Weights of ten tablets of diclofenac of each company were measured and homogenized by making powder (using mortar and pestle). For each company triplicate studies were carried out simultaneously using same homogenized powder of the medicine.

Considering the average weight of diclofenac (50 mg) tablets, the required amount of powder samples were suspended in methanol to obtain 20 µg/mL active ingredient of the medicine. The suspended material were vortexed (2 min), centrifuged (3000 rpm; 5 min) and the supernatants were collected. The active ingredients present in the supernatants were cleaned up by filtering through HPLC grade syringe filters (0.45µm). Absorptions of the cleaned extracts (three samples for each company) were measured and their concentrations were calculated using external calibration curve of the certified diclofenac sodium. Relative standard deviations were calculated and the data are presented in the (Table 2).

Recovery experiment

Recovery experiments were done by spiking excipients of the medicine at two different concentration levels (10 and 20 µg/mL). Replicate studies of the said medicine were 5-7. Relative Standard Deviations were calculated and the results are presented in (Table1).

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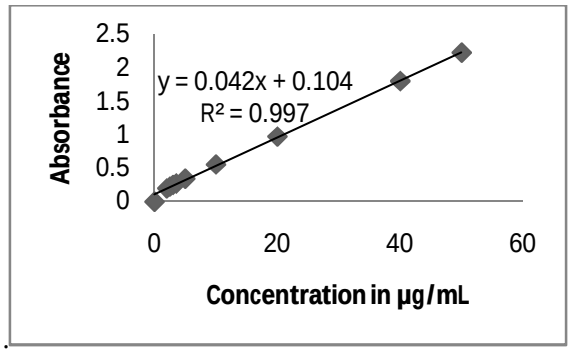
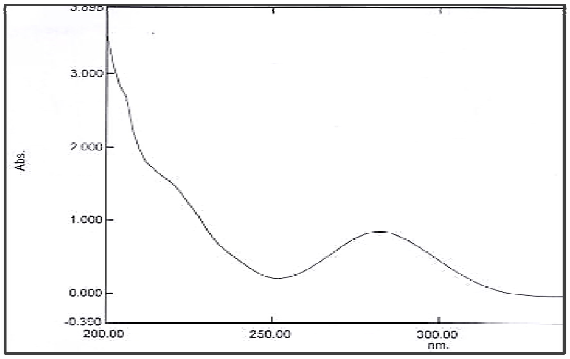
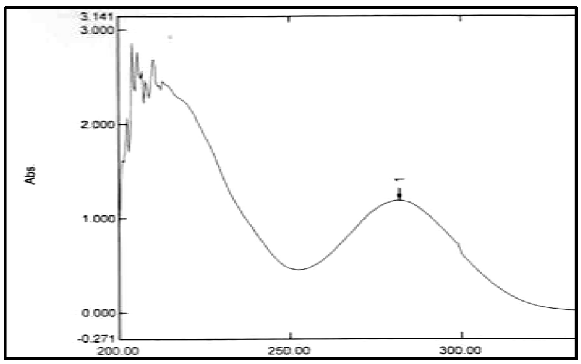


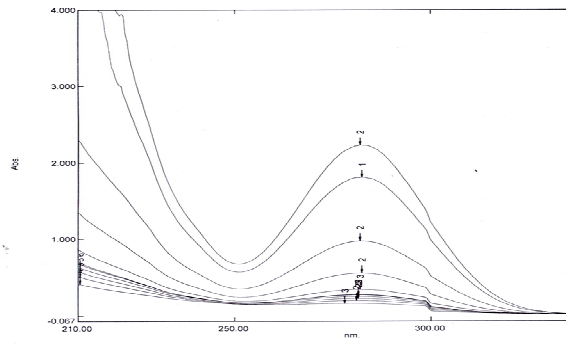
Fig. 2. Calibration curve of diclofenac.



(a)



(b)



(C)

Fig. 3. Spectra of standard diclofenac (a), cleaned extract of tablet (b) and overlain spectra (c).

Table 1. Accuracy studies in standard diclofenac

Spiking concentration (µg/mL)	% Recovery	% Mean Recovery
10 (n=5)	107.96 ± 3.56	105.68 ± 3.75
20 (n=7)	103.39 ± 3.93	

Table 2. Assay of diclofenac

Sample code	Declared amount of diclofenac in each tablet (mg)	Estimated amount of diclofenac in each tablet (mg)	Relative standard deviation (%)	Amount of active ingredient (%)
D1	50	49.79 ± 0.63	1.26	99.58
D2		48.33 ± 1.58	3.27	96.66
D3		49.28 ± 0.82	1.67	98.56
D4		58.52 ± 0.41	0.70	117.04
D5		47.91 ± 0.90	1.87	95.82
D6		49.57 ± 0.63	1.27	99.14
D7		51.61 ± 1.08	2.09	103.22

III. Results and Discussion

The wavelength of maximum absorption (λ_{max}) of the cleaned-up extracts of diclofenac tablets (Fig. 3b) and their overlain spectra (Fig. 3c) fitted very well with standard spectra (Fig. 3a) recorded at 282 nm. The correlation coefficient (r^2) value 0.9974 (Fig. 2) of the medicine showed accuracy of experiments. Low limit of detection (LOD) of diclofenac was 2 µg/mL (Fig. 3c) showed sensitivity of the method. The method was validated from recovery experiments (Table 1) and the low respective relative standard deviations indicated the method was satisfactory. The results also showed acceptable repeatability of the method.

The active ingredient was in the range of 47.91 ± 0.90 to 58.52 ± 0.41 mg (Table 2). Two of the samples (D4 and D7) were found to be contained higher amount of active ingredient (51.61 ± 1.08 and 58.52 ± 0.41 mg) and other five samples (D1, D2, D3, D5 and D6) were found to be

contained lower amount of active ingredient (47.91 ± 0.90 to 49.79 ± 0.63 mg) than the declared value (50 mg) in the formulated drugs.

The accuracy of the method was done by recovery experiment (Table 1). The % of mean recovery was 105.68 ± 3.75.

The methodology for determination of diclofenac was new from the best of our knowledge. The described method for the determination of diclofenac was satisfactory with a wide range of concentration. The method was very much easy to carry out and also cheaper than other methods such as HPLC[8], polarography[9]. Our goal was to determine diclofenac easily in dosage forms with sufficient precision and accuracy. As the relative standard deviation value (0.70-3.27%) obtained from the analysis were below 4%, so the method was satisfactory. The method was sufficiently sensitive with high accuracy and precision, analysis of diclofenac of pharmaceutical dosage forms can be done within a short period of time.

IV. Conclusion

Diclofenac is common medicine but insufficient or excessive amount can bring harmful situation. Analytical method used in the present study was found to be easy, cheap and also specific, precise, linear and accurate. The method will be very helpful for routine analysis of diclofenac for pharmaceutical companies in Bangladesh.

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