Surekha S. et al. / Vol 3 / Issue 2 / 2013 / 77-80.

International Journal of Pharmaceutical Research & Analysis

e-ISSN: 2249 – 7781
Print ISSN: 2249 – 779X

www.ijpra.com

RECENT APPLICATIONS OF FLOW INJECTION SPECTROPHOTOMETRY

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ABSTRACT

Pharmaceutical analysis is one of the most important fields in analytical chemistry. The discovery of new drugs and the on-going update of international regulations for the safety and efficacy of pharmaceutical formulations demand the continuous development of new analytical methods. Inevitably, automation plays an important role, especially when a lot of samples have to be analyzed in the minimum of time. The present study reviews the applications of flow injection (FI) spectrophotometry to the determination of active pharmaceutical ingredients (APIs) in their respective formulations. However, the topic covered in this study is important not only to pharmaceutical analytical scientists. The principles, figures of merit and "chemistry" of the presented methods can be of interest to bio-analytical and clinical chemists as well for the analysis of biological samples, to environmental analysts that study the up-to-date demand of the determination of the fate of pharmaceuticals in the environment and even to toxicologists and forensic scientists. This review covers scientific contributions published later than 2000. A variety of FI procedures based on homogeneous (direct UV measurements, colour-forming reactions, metal-drug interactions) and heterogeneous (optical sensors and solid-phase reactors) systems are discussed and also covers on-line sample pre-treatment (solid-phase extraction, liquid-liquid extraction, on-line digestion, etc).

Keywords: Flow injection spectroscopy, Active pharmaceutical ingredients, Bio-analysis.

INTRODUCTION

In recent years more and more strict regulations related to the quality control of pharmaceuticals led to increasing demands on the automation of analytical assays carried out in appropriate control laboratories [1]. At the same time, during twenty-five years of its existence, the FIA technique became a versatile instrumental tool that contributed substantially to the development of automation in pharmaceutical analysis. This can be well documented by a number of reviews on the use of FIA in the analysis of drugs. In the field of flow injection in pharmaceutical analysis several reviews were published by our group. In order to facilitate better orientation in the respective matter we decided to adopt the scheme of sorting the FIA methods according to the type of detection as the major classification feature regardless of auxiliary on-line procedures employed within the FIA scheme, such as solvent extraction, dialysis, solid-phase pre-concentration, photolysis, use of packed-bed enzyme reactors or immune sorbents, etc.

Flow Injection Analysis

Definition

The term Flow Injection Analysis (FIA) was first described in 1975 as the injection of a liquid sample into a moving, non-segmented continuous carrier stream. FIA can be used at-line, on-line and in-line for PAC.

Principle

This section is designed to provide an understanding of processes that yield FI response curve, and to offer tools for optimizing sensitivity, detection limit and sampling frequency of flow injection based assays. We begin with definition of three cornerstones on which all. We begin with definition of three cornerstones on which all flow injection techniques are based [2]

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- sample injection
- controlled dispersion
- reproducible timing

They will continue with examples how these parameters are controlled and manipulated through change of injected volumes, flow rates and manipulated through change of injected volumes, flow rates and manifold configurations. While single reagent assays can be performed using the simplest, single stream manifold, it will be shown why a majority of FI techniques use multistream manifolds, where several reagents are sequentially merged with a carrier stream that moves the injected sample zone through the manifold and a flow cell. Note that discussion in the following sections deals with a single stream system. For multistream systems D-values have to be corrected by dilution caused by additional streams [3].

Phases of flow injection

Dispersion

This can be defined as the ratio of concentrations before and after the dispersion process has taken placein the detector [4].

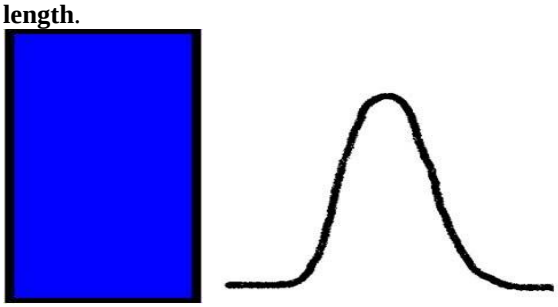
Dispersion, D, can be quantified as follows

$D = C_o / C_{max}$

C_o is the un-dispersed sample i.e. no dilution taking place between the sample injection valve and the detector, whileC_{max} is a result of sample dispersion e.g. a flow injection detector response [5].

Some detectors require an undiluted sample to be reproducibly transported to a detector e.g. pH i.e. no mixing required (D = 1 to 3). However, if analyte conversion to a compound is required, based on the mixing of reagents with the sample e.g. in spectrophotometry, then D is increased (D = 3 to 10). In extreme circumstances more mixing may be required such that the dispersion is very large (D = > 10).

Various parameters can be altered in FIA that affect dispersion including **sample volume** and **tube length**.



C_0 **C_{Max}**

Influence of Sample Loop Volume on Dispersion

Injecting increasing volumes of the same sample concentration, influences dispersion and affects method sensitivity [6].

Equipment

The apparatus required for FIA is relatively simple and consists of:

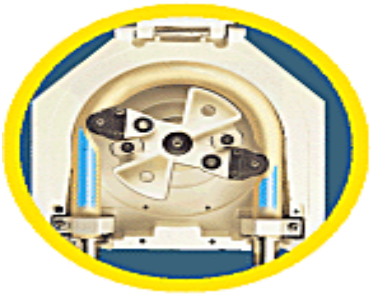
- A peristaltic pump to move liquid (carrier stream);
- An injection valve to introduce a small discrete sample or standard into the carrier stream;
- A sample processing stage (commonly called the reaction coil). This allow for:
  - mixing of reagents/samples/standards;
  - dilution of the sample;
  - Enrichment of the sample for trace analysis.
- A flow through detector to measure a response [7].

All of the components are connected to one another to produce a continuous moving and dynamic system that is capable of being automated [8]. A typicaldiagrammatic representation of these components is shown as figure 4.

Pump

The **peristaltic pump** works by rotating a series of rollers attached to the outside circumference which compress flexible pump tubing against a hard surface i.e. platen. The continual compression and relaxing (returning to normal dimensions) of the tubing, by the moving rotor, allows liquid to be moved forward. The multi-rollered nature of the pump means that more than one set of pump tubing can be in operation at the same time. In addition, by using different internal diameters of the flexible pump tubing, allows different flow rates to be achieved using a constant rotor speed [9].

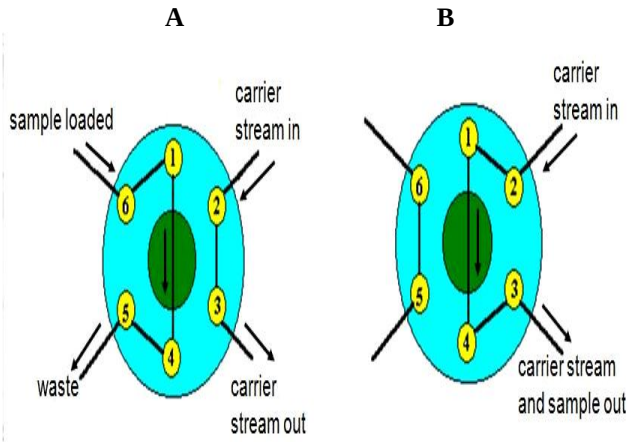
Injection Valve



The most common **injection valve** for FIA is the 6-port injection port. This device allows a discrete amount of sample or standard to be reproducibly introduced in to a moving carrier stream with minimal disruption whilst at the same time not interrupting the flow of the carrier stream. The injection valve operates in two positions.

In this position the sample is loaded into the loop whilst maintaining a constant flow of carrier stream; sample loop is between port 1 and 4.

In this position the sample is removed from the sample loop by the carrier stream allowing efficient introduction of the sample into the carrier stream [10].



A 6-port injection valve (A) load position, and (B) inject position

Detectors

A flow through detector is located downstream from the sample injector and records a chemical physical parameter [11]. Various different types of detector can be used For example:

- colorimeter
- fluorimeter
- ion-selective electrode
- biosensors

Changing injection volume has a powerful influence on dispersion. An increase in peak height and sensitivity is achieved by the increasing sample loop volume. Conversely, dilution of concentrated sample material is achieved by reducing injection volume [12].

Applications

- Molecular Spectroscopy Detection
- Atomic Spectroscopy Detection Methods
- Electrochemical Detection Methods
- Enzymatic Methods of Detection and Immunoassays
- Other Detection Methods Used in FIA
- On-Line Sample Processing in FIA Systems
- Speciation Analysis Using Flow Injection Methodology
- Applications of Flow Injection Methods in Routine Analysis
- Sequential and Batch Injection Techniques
- Commercially Available Instrumentation for FIA
- Current Trends in Developments of Flow Analysis

Fig 1. Phases of flow injection

Fig 2. Influence of injection volume on peak height

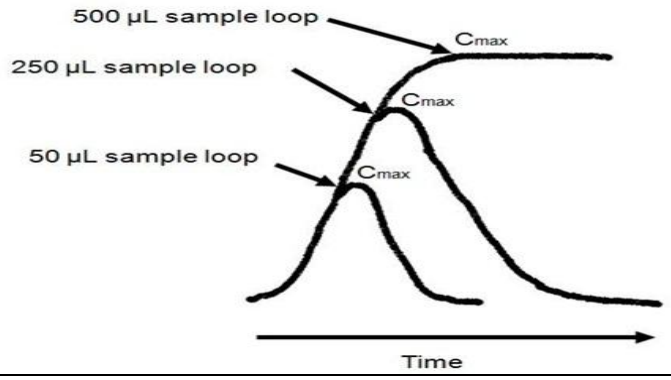


Fig 3. A plot of injection volume versus dispersion produces a graph.

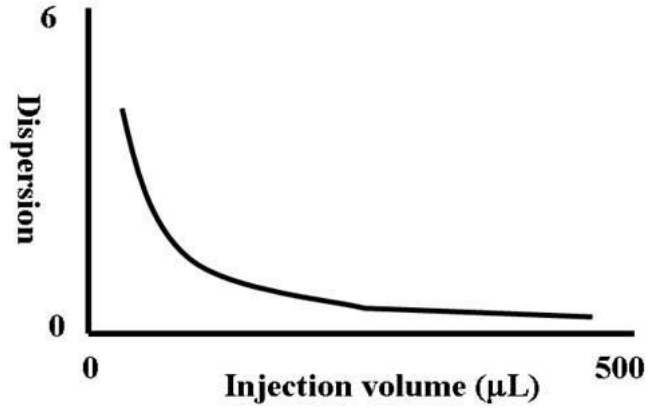


Fig 4. A typical diagrammatical representation of a FIA system

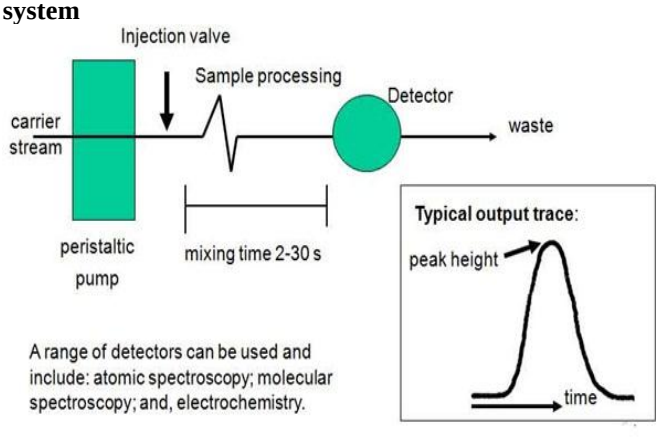


Fig 5. Photograph of an injection valve

CONCLUSION

The FIA technique became a versatile instrumental tool that contributed substantially to the development of automation in pharmaceutical analysis. This can be well documented by a number of reviews on the use of FIA in the analysis of drugs. In the field of flow injection in pharmaceutical analysis several reviews were published by our group. In order to facilitate better orientation in the respective matter we decided to adopt the scheme of sorting the FIA methods according to the type of detection as the major classification feature regardless of auxiliary on-line procedures employed within the FIA scheme, such as solvent extraction, dialysis, solid-phase pre-concentration, photolysis, use of packed-bed enzyme reactors or immune sorbents, etc.

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