Available on line www.jocpr.com

Journal of Chemical and Pharmaceutical Research

J. Chem. Pharm. Res., 2010, 2(2): 118-125

ISSN No: 0975-7384

Flow injection: A new approach in analysis

Ashish Parikh*, Kamlesh Patel, Chetan Patel and Patel BN

Shri Sarvajanik Pharmacy College, Arvind Baug, Mehsana, Gujarat, India

Abstract

The manual handling of solutions (known as "beaker chemistry") remains the Achilles Heel of modern analytical instrumentation. It is currently being replaced by flow injection analysis (FIA), which is computer compatible and allows automated handling of sample and reagent solutions with a strict control of reaction conditions. The flow injection analysis (FIA) was preceded by the success of segmented flow analysis, mainly in clinical and environmental analysis. This advance, as well as the development of continuous monitors for process control and environmental monitors, ensured the success of the FIA methodology. The fast and intensive development of the FIA methodology was due to several factors essential for routine analytical determinations, such as very limited sample consumption, the short analysis time based on a transient signal measurement in a flow-through detector and an on-line carrying out difficult operations of separation, physicochemical conversion of analytes into detectable species. On this field, Flow injection analysis offers unique features and advantages. FIA is therefore a mature technique with well-defined and explored principles of operation.

Key Words:-Flow injection analysis, description, principle, instrumentation, application.

Introduction

One of major development in the analytical chemistry during last four decades has been the appearance of the Commercial automatic analytical system, which provides analytical data with a minimum of operator intervention. Initially, these systems were designed to fulfill the needs of clinical laboratories, where perhaps 30 or more species in blood and urine samples are routinely determined for diagnostic and screening purpose. Domestically, hundreds of millions of clinical analyses are performed annually; the need to keep their cost at a reasonable level is obvious. These two considerations motivated the development of early automatic analytical system[1]. Now, Automation is a key demand in modern Analytical Chemistry. Process and Quality control require fast and reliable results in all areas of human activity. Such instrument finds

application in fields as diverse as the control of a wide spectrum of species in air, water, Soil, pharmaceuticals and agricultural products.

FIA (Flow Injection Analysis) was defined by Ruzicka and Hansen in 1975. Simultaneous patents by Ruzicka and Hansen in Denmark and Stewart in the USA launched a new technology that would quickly gain worldwide acceptance.

Three key attributes of this technology ensured its rapid acceptance:

- The fundamental principles are easy to understand and implement.
- Instrumentation can readily be assembled from simple, inexpensive, off-the-shelf components.
- It provides a simple means of automating many manual wet chemical analytical procedures.

A typical definition describes FIA as:

- "A simple and versatile analytical technology for automating wet chemical analysis, based on the physical and chemical manipulation of a dispersed sample zone formed from the injection of the sample into a flowing carrier stream and detection downstream". [1]

Description of FIA

The schematic below groups the FIA process into three stages to help visualize how the technique performs a method or analysis.

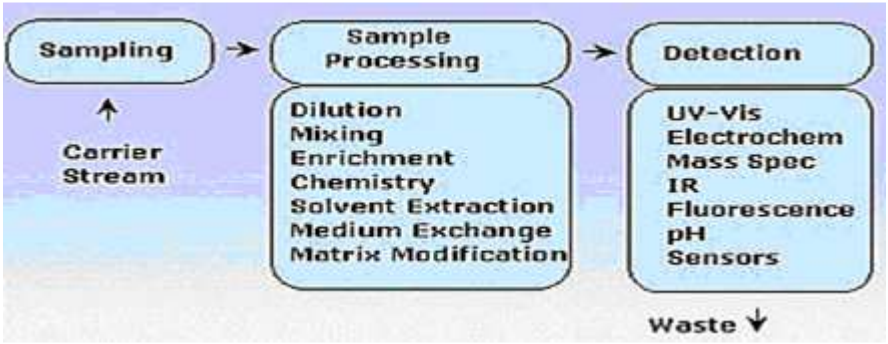

Fig 1: Description of FIA

Sampling

First is sampling, where the sample is measured out and injected into the flowing carrier stream (thus, the name Flow Injection Analysis). This step is generally performed with a sample injection valve.

Sample processing

Second stage is sample processing. The purpose of this step is to transform the analyte into a species that can be measured by the detector and manipulate its concentration into a range that is compatible with the detector, using one or more of the indicated processes.

Detection

The third stage is detection where the analyte, or a derivative of it, generates a signal peak that is used to quantify the compound being determined. As indicated, a large variety of detectors can be used in FIA. The power of FIA as an analytical tool lies in its ability to combine these analytical functions in a wide variety of different ways to create a broad range of different

methodologies, and perform these methodologies rapidly and automatically with minute (µL) amounts of sample. The first and last stages are, largely, conventional technology. [2]

Principle of FIA

A FIA peak occurs due to two processes, one involving the simultaneous physical process of zone dispersion and the second involving the chemical process resulting from reaction between sample and reagent species. A difference in the concentration gradient is thus generated.

Immediately after injection with a sampling valve, sample zone (plug) concentration profile is rectangular shown in figure (a).

For all practical purposes, flow through the bypass ceases with the valve in this position because the diameter of sample loop is significantly greater than that of the bypass tubing.

As it moves through the tubing, band broadening or dispersion takes place. The shape of the resulting zone is determined by two phenomena. The first is convection arising from laminar flow in which the center of the fluid moves more rapidly than the liquid adjacent to the walls, thus creating the parabolic shaped front and the skewes zone profile had shown in figure (b).Broadening also occurs as a consequence of diffusion.

Two types of diffusion can, in principle, occur:

1. Radial, or Perpendicular to the flow direction
2. Longitudinal or parallel to the flow.

The latter is of no significance in narrow tubing, whereas radial diffusion is always important under this circumstance. In fact, at low flow rates it may be the major source of dispersion.

Here, the radial dispersion from the walls toward the center serves the important function of essentially freeing the walls of analyte and thus eliminating cross-contamination between samples.

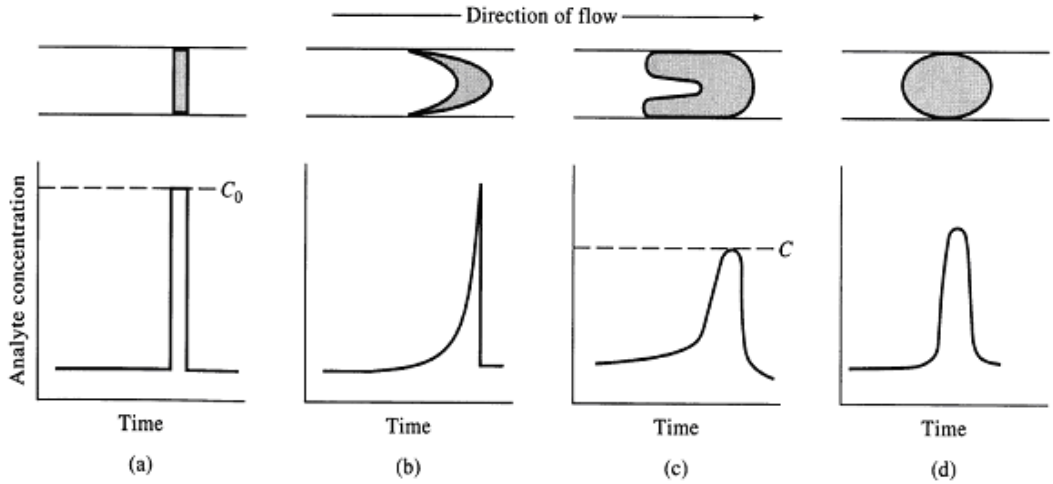

Fig 2: Effect of convection on concentration profiles of analytes at the detector, (a) no dispersion, (b) dispersion by convection, (c) dispersion by convection and radial diffusion, (d) dispersion by diffusion.

Dispersion

The extent of dispersion of dilution is measured in terms of dispersion coefficient (D). D is the ratio of the concentration of the sample before and after the dispersion process takes place. Dispersion coefficient (D) is defined by the equation

$$D = c_o/c$$

Where c_o = concentration in injection volume,
c =peak concentration at detector

When a FIA cell is to be fabricated one must know to what extent the original solution is diluted while flowing through to the detector and time between the sample injection point and read out point. Hence dispersion coefficient (D) is used to decide these dimensions.

D affected by

- Sample volume
- Tube length
- Flow rate
- Tube id

The sample dispersion is rated as

- Limited if D=1 to 3,
- Medium if D=3 to 10,
- Large if D>10

Out of three limited dispersion is preferred. [3]

Instrumentation

The following diagram can be used to describe the basic components and principles of FIA.

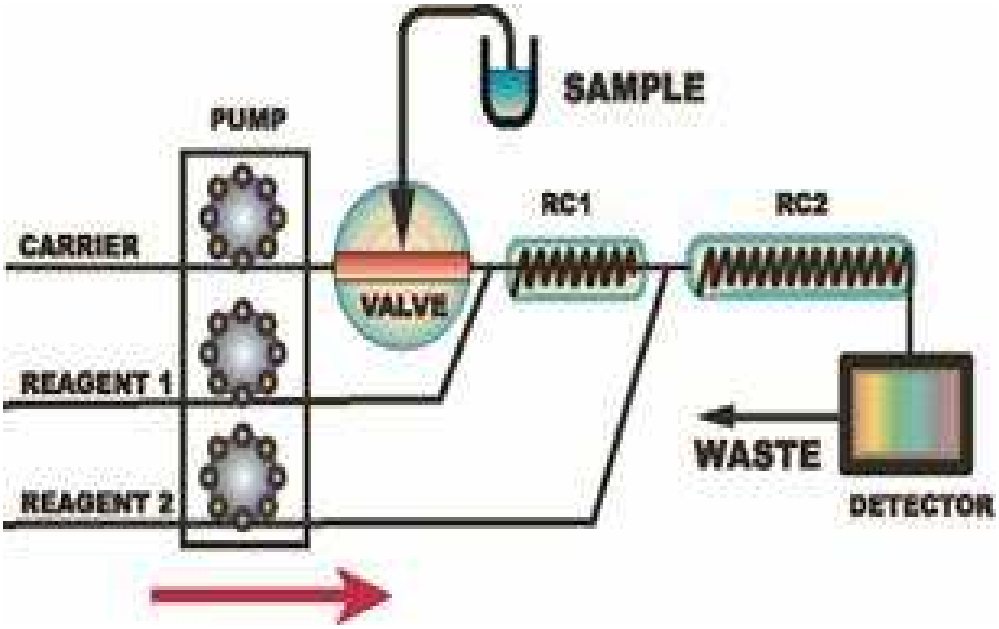

Fig 3: Flow injection analyzer

The modern Flow Injection Analysis system usually consists of:

- An injection valve,
- A high quality multi channel peristaltic pump,
- A coiled reactor,
- A tubing manifold
- A detector
- An auto sampler

Additional components may include a flow through heater to increase the speed of chemical reactions, columns for sample reduction, de bubblers, and filters for particulate removal.

Working of flow injection analyzer

In its simplest form, the sample zone is injected into a flowing carrier stream of reagent. As the injected zone moves downstream, the sample solution disperses into the reagent. As this sample is carried to the detector, the fluid dynamics of flow through narrow-bore tubing mixes sample and reagent, leading to chemical reaction to form a detectable species. This species is sensed by the detector as a transient peak. A detector records the desired physical parameter such as colorimetric absorbance or fluorescence, electrode potential, refractive index or electrical conductivity. Such variation in any physical property occurs due to the passage of the sample through the flow cell. The height and area of the peak are proportional to concentration, and are used to quantify the concentration of the compound being determined by comparison to samples of known concentration. The typical FIA flow rate is one milliliter per minute, typical sample volume consumption is 100 microliters per sample, and typical sampling frequency is two samples per minute.

Sample Introduction

Injection valve

Important features of valves suitable for FIA:

- High precision,
- Fast switching,
- Pressure limits of about 100 psi,
- Ability to inject sample volumes from a few micro liters to several hundred micro liters, and in some cases fractions of a micro liter.

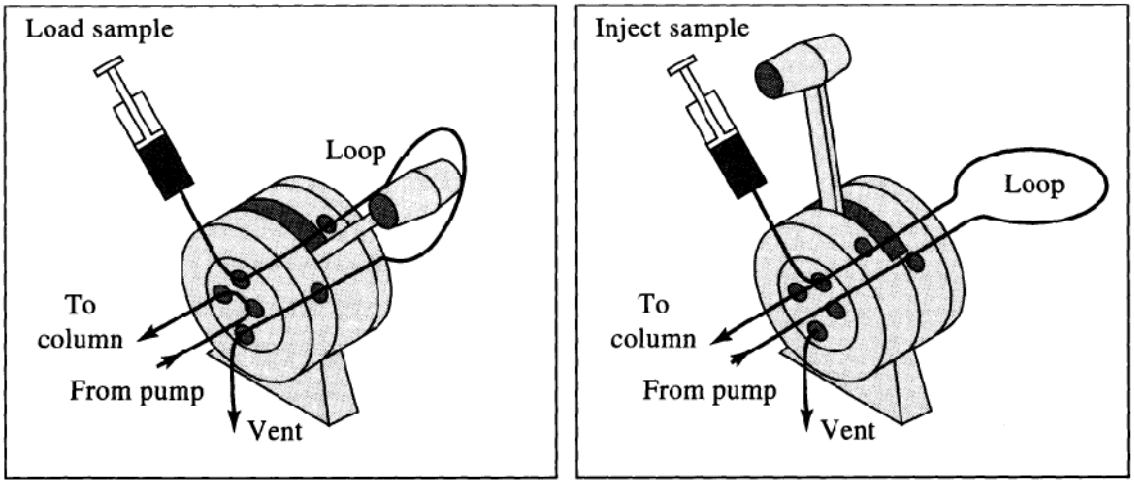

Fig 4: An injection valve

Pumps
(i) Variable speed peristaltic pump
(ii) Flow rate (0.0005 to 40 ml/min) controlled by pump speed and tube id
(iii) The pump is used to propel one or more streams through the Detector via narrow bore (0.5 - 0.8 mm ID) tubing. These streams may be reagents, solvents, or some other medium such as a buffer.

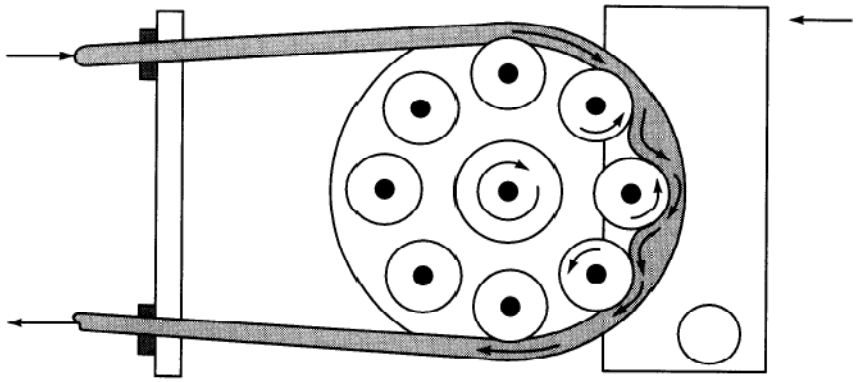

Fig 5: Peristaltic pump

This design leads to a flow that is relatively pulse free. The flow rate is controlled by speed of motor, speed is >30 rpm and the inside diameter of the tubing.

Tubes and Reactors
(i) Small tube diameters (<0.1-1 mm id)
(ii) Reactor coil (<50 cm long) tightly wound to increase mixing
A wide variety of tube sizes are available commercially and permits flow rates as small as 0.0005 ml/min and as great as 40 ml/min. A flow injection systems often contain a coiled section of tubing (diameter 1 cm or less) whose purpose it is to enhance axial dispersion and to increase radial mixing of the sample and reagent, both of which lead to more symmetric peaks.

Detection
Detection is most frequently photometric (uv/vis and more recently IR). In the field of life sciences, different luminescence techniques are gaining popularity. Electrochemical techniques such as amperometry, and potentiometer, have gained new life by coupling them to flow-based sample handling techniques such as FIA. Even AAS, ICP-MS and ICP-AES, and even GC have been coupled to FIA manifolds. [4]

Advantage of FIA
It is easy to see that compared to manual analyses; the tubing lines serve as solution containers and transfer vessels, the injection valve serves as a micropipette, and the pump replaces the lab technician using all this lab ware.
FIA has been very successful in simplifying chemical assays. The main reasons for the success are the following advantages of FIA over conventional manual techniques:

1) Reduced labour costs due to automation.
2) Great precision due to mechanical performance of the assays.
3) High sampling rate.
4) Smaller sample and reagent consumption and waste generation.
6) Simplicity and low cost instrumentation.
7) Availability of instrumentation in almost all laboratories.

8) Reduced analyses cost when a lot of samples have to be analyzed.
9) Increased precision compared to batch methodologies.
10) Automation in sample preparation and detection. [5]

Application of FIA

Typical applications of flow injection analysis include the following fields

1. Pharmaceutical application
2. Environmental analysis
    a. Sea water
    b. Waste water
    c. Sediments
3. Food analysis
    a. Fruit juice, soft drinks
    b. Wine
    c. Milk and dairy products
4. Biological material
    a. Plants, animal
5. Mineral material
    a. Soil
    b. Fertilizers
    c. Alloys
6. Clinical assay
    a. Serum
    b. Plasma
    c. Whole blood
    d. Urine
7. Bioanalytical chemistry
        a. Proteins
        b. Amino acids
        c. Ammonia
        d. Glucose
8. On line monitoring in Biotechnology
9. Monitoring waste and its treatments [6], [7]

Pharmaceutical applications

Various Pharmaceutical applications of FIA are listed below:

- Determination of Chloride ions by Automated Flow Injection Analysis.
- Determination of Total carbonate in an aqueous solution by gas diffusion technique.
- Determination of caffeine in acetylsalicylic acid preparations.
- Determination of Fluoride ions by FIA.
- Application in environmental science for continuous monitoring.
- NO2 can be detected in the atmosphere by allowing 1-naphthyl ethylene diamine HCL with sodium nitrite solution in the presence of MBT to give a coloured complex which can easily be measured continuously.
- The course of the reaction between $(NH_4)_2MoO_4$, H_3PO_4, HNO_3 and ascorbic acid can be measured with FIA at 660 nm as molybdenum blue complex.
- By using Pararosaniline method one can measure SO2 from the atmosphere on a continuous basis.
- Determination Calcium in water by formation of a coloured complex with o-cresolphthalein complexone at pH 10. [8-10]

Discussion

Automation is a key demand in modern Analytical Chemistry. Process and Quality control require fast and reliable results in all areas of human activity. On this field, Flow injection analysis offers unique features and advantages. The scope of the method grew from serial assay of samples to a tool for enhancement of performance of spectroscopic and electrochemical instruments. Most recently FI became applied in biology for study of live cells by fluorescence microscopy and flow cytometry. Other fields include monitoring of chemical processes in real time, biotechnology, immunoassays including antibody/antigen reactions.

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