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RESEARCH ARTICLE

Determination of Hydrogen Peroxide (H_2O_2) by New Design of Continues Flow Injection Unit

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Abstract

This study includes the design of continues injection system to determine the hydrogen peroxide (H_2O_2) containing valve is designed locally and this method is quick, cheap and characterized by high accuracy. The method is based on the fast reduction of the complex compound $K_3[Fe(CN)_6]$ by H_2O_2 in basic medium with maximum wavelength at 420 nm. Various parameters (physical and chemical) affecting on the determination have been investigated such as flow rate, volume of the loop, concentration of the complex compound and concentration of the base. The calibration curve was prepared and the dispersion coefficient, reproducibility and application were studied. The linear range was (0.06 - 0.8) % at sampling rate of 60 sample per hour, (R^2=0.9923) and the detection limit at S/N=3 was 0.02%. Dispersion coefficient was also measured for the method.

Keywords: *Continues Injection, Determination Hydrogen peroxide H_2O_2.*

Introduction

Flow injection analysis (FIA) arises as a consequence of the growing trend towards automation in chemical analysis, and as a natural evolution of the so-called continuous flow analysis. (CFA) which has revolutionized the conception of chemical analysis, especially in the field of clinical analysis and sample manipulation [1].

Continues flow Injection analysis (CFIA) introduced by Skeggs , relies on the fact that the sample and the reactive react while traveling in the tube that conducts them to the detector [2]. Air bubbles separate the portions of fluid (reactive) in order to diminish the longitudinal dispersion of the sample and the interaction between samples, which in turn favor mixing and increase the analysis frequency [3], this technique have a wide applications[4-6].

Hydrogen peroxide is an environmentally friendly chemical, used for oxidation reactions, decomposing to only oxygen and water, hydrogen peroxide is one of the cleanest chemicals available [7,8]. The analytical methods for H_2O_2 determination are based on different techniques such as volumetric titration, colorimetric, fluorometric, electrochemical ,luminescence, chemiluminescence and spectrophotometric methods [9-15].However, the accurate and rapid determination of small quantities of H_2O_2 by flow injection analysis [16]. Determination of hydrogen peroxide is usually based on the production of colored peroxy compounds or on its oxidizing and reducing properties. Based on this property, numerous methods have been developed for the determination of hydrogen peroxide [17].

H_2O_2 is widely used in the fields of foods, pharmaceuticals, dental products, textiles, environmental protection and it is also involved in advanced oxidation processes and various biochemical processes [18-21].Due to its mild oxidation ability, H_2O_2 is widely used in different large-scale processes such as those in textile, paper, food, beverages and pharmaceutical industries [22].

Experimental

Apparatus

Spectrophotometer Labomed in G single beam, USA, spectrophotometer Shimadzu UV-1700 spectrophotometer Japan, Recorder Pen Siemens C 1032 Germany, Analytical balance sensitive Denver Instrument Germany, peristaltic pump Germany, pH meter Germany, files Interaction with the radius of 0.5 mm, homemade valves, pipes load of Teflon, flow cell volume of 450 µL.

Preparations

Series of solutions were prepared by dissolving 0.01g of K3 [Fe (CN) 6] with 0.2% of NaOH and different concentrations of Hydrogen peroxide (0.06% - 0.8%) in water and dilute the solutions with distilled water to 50 ml in volumetric flask.

Design of Continuous Injection Units

Fig.1: Photo for the real Design of continuous injection Unit

This research included design of a new valve from cheap and obtainable materials, this valve consists of two secondary valves (1) and (2), and each secondary valve has three apertures with lever control the flow direction of the chemical reaction. The work of the unit involves several steps which are injection step, pump, detect and recording step as can be shown in Figure (2).

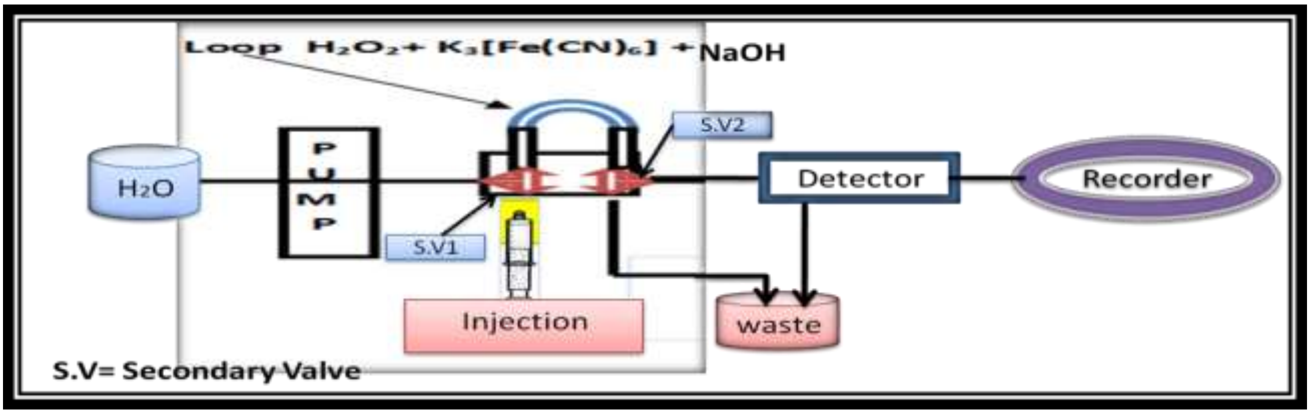

Fig. 2 Schematic diagram of the new design

Results and Discussion

Determination of the Absorption for Maximum Wavelength

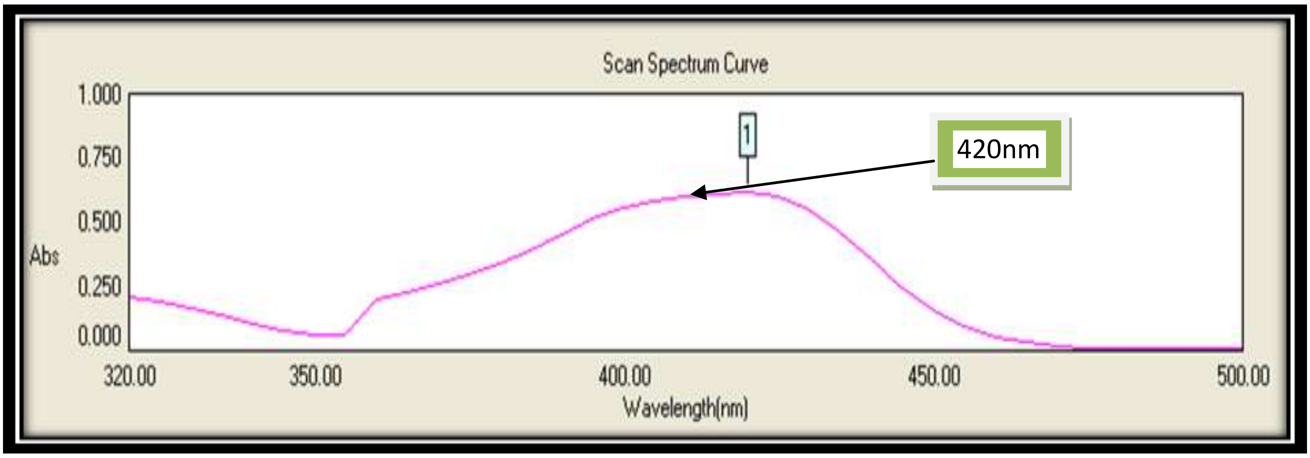

Fig. 3: UV-Vis spectrum for the complex compound K_3 [Fe (CN) $_6$]

Ultraviolet visible spectrum was used to determine the λ max of the complex formation as in figure (3), the λmax of complex was 420 nm.

Physical Parameters

Effect of the Flow Rate

The effect of the flow rate on the peak height was studied in the range of (8.67-20.01) mL min^{-1} as shown in table (1) and figure (4), it was found that lower flow rate (8.67-10.56) mL min^{-1} cause doublet, while in the flow rate (12.40-20.01) mL min^{-1} the peaks were enhance significantly toward sharp peak, therefore the optimum peak at flow rate 12.40 mL min^{-1}.

Table 1: Effect of flow rate on the peak height at; 1% of H_2O_2, 100 ppm of Fe (III), 0.02% of NaOH, volume of mixture (H_2O_2, K_3 [Fe (CN) $_6$, NaOH) = 177.75 µL

Speed of pump rpm	Flow rate mL.min^{-1}	Mean Peak height (V)	S.D	R.S.D%	The response form
100	20.01	65.00	0.000	0.000	narrow
90	18.33	56.33	0.144	0.256	narrow
80	16.99	51.58	0.520	1.009	narrow
70	15.22	47.50	0.125	0.277	(sharp and narrow)
60	12.40	42.50	0.000	0.000	(sharp and narrow)
50	10.56	38.33	0.382	0.996	Slightly distorted and broad
40	8.67	36.25	0.250	0.690	Distorted, partitioned, and broad

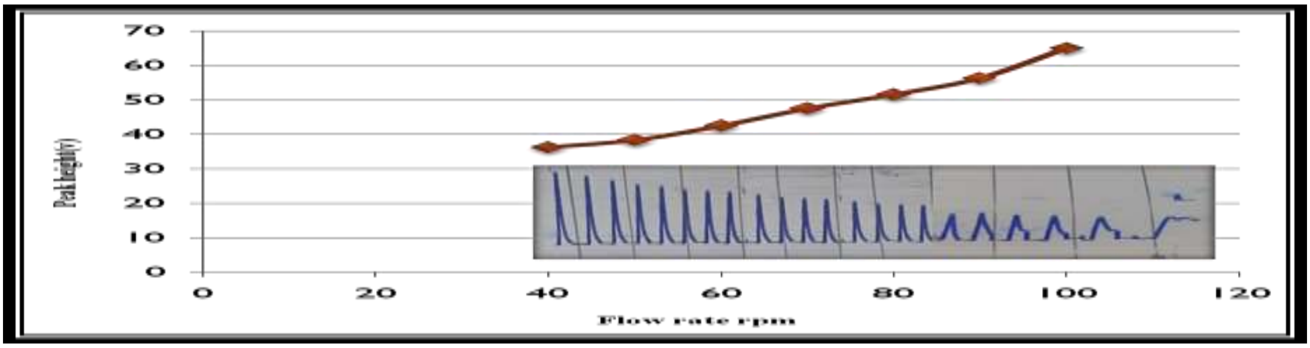

Fig.4: Effect of flow rate on the form and height of the peak

Effect of Mixture (H_2O_2, K_3 [Fe (CN) $_6$], Noah) Volume

The influence of the mixture (H_2O_2, K_3 [Fe (CN) $_6$], NaOH) volume on the peak height was investigated by using different volumes of loops (86.35- 235.50) µL. The significant peak height was (196.25) µL, after that volume the peak height increased. Therefore ((196.25) µL was chosen for further work as shown in Table (2) and Figure (5).

Table 2: Effect of the mixture volume on the peak height; 1% of H_2O_2, flow rate (12.40 mL min^{-1}), (100) ppm of Fe (III) and 0.02% of Noah

The volume of mixture(H_2O_2, $K_3[Fe(CN)_6]$,NaOH) (µL)	Mean Peak height (V)	S.D	R.S.D%
86.35	68.75	0.001	0.001
117.75	65.00	0.032	0.049
157.00	37.51	0.012	0.031
196.25	31.24	0.017	0.055
235.50	22.42	0.144	0.644

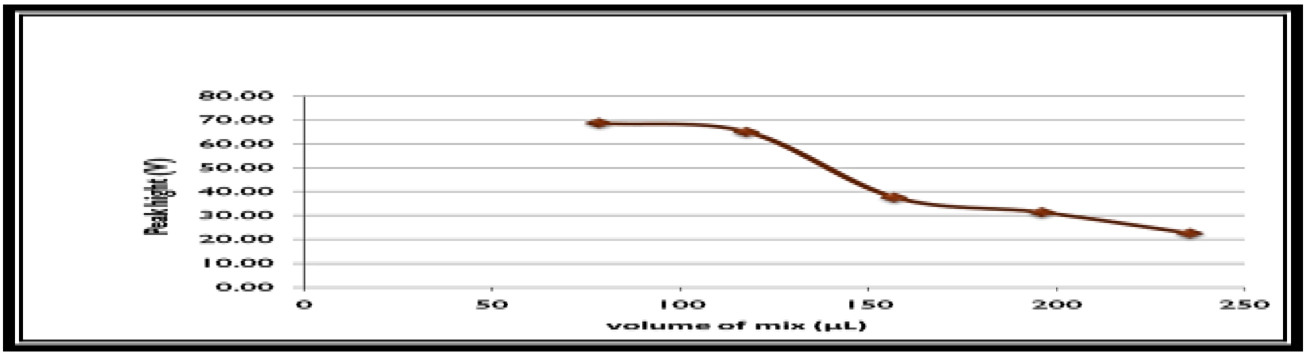

Fig. 5 Effect of mixture (H_2O_2, K_3 [Fe (CN) $_6$] and NaOH) volume on the peak height

Chemical Parameters

Effect of the Complex (K_3 [Fe (CN) $_6$]) Concentration

The complex concentration was varied in the range (50-200) ppm in order to maximize the peak height. Table (3) and figure (6) show the effect of complex concentration on the peak height. The maximum peak height was obtained with (200) ppm.

Table 3: Effect of Fe (III) concentration on the peak height at ; 1% of H_2O_2, flow rate (12.40 mL min^{-1}), 0.02% of NaOH and volume of mixture (H_2O_2, $K_3[Fe(CN)_6$, NaOH)=196.25 µL.

Conc. of $K_3[Fe(CN)_6]$ ppm	Mean Peak Height (V)	S.D	R.S.D%
50	18.74	0.012	0.062
100	25.21	0.186	0.737
150	44.67	0.577	1.293
200	68.75	0.000	0.000

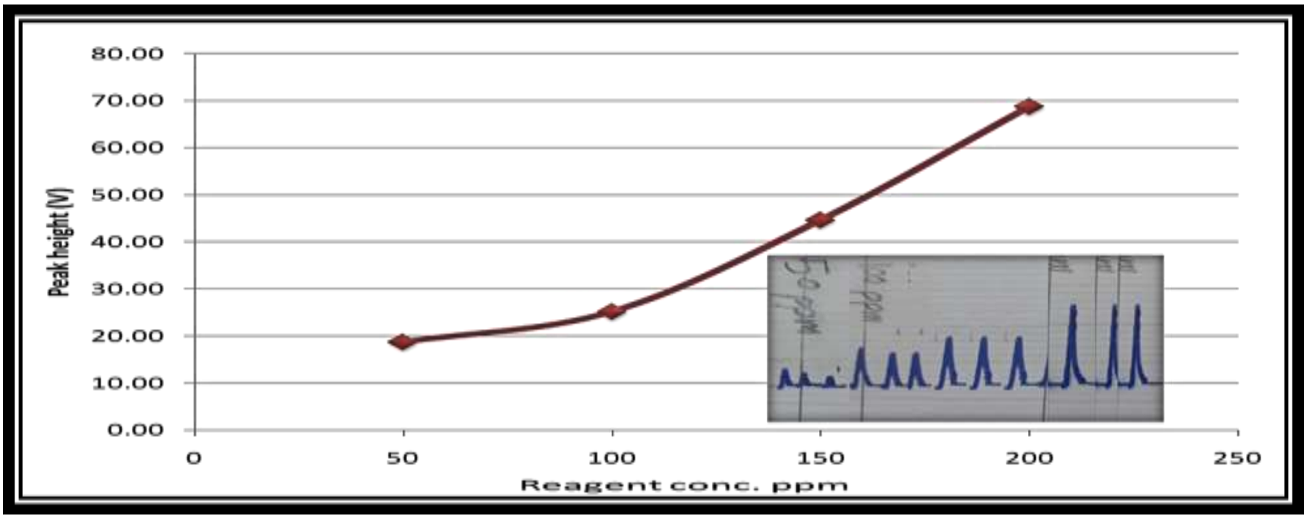

Fig. 6: Effect of complex concentration Fe (III) on the peak height

Effect of Noah Concentration

The effect of Noah concentration also studied as a catalyst for the reaction and the range of concentrations was (0.006- 0.2) %. The Noah concentration gave the significant peak height 0.2 % as shown in Table (4) and Figure (7).

Table 4 : Effect of Noah concentration on the peak height at ; 1% of H_2O_2, flow rate(12.40 mL min^{-1}), 200 ppm of Fe(III) and volume of mixture (H_2O_2, $K_3[Fe(CN)_6$, Noah)=196.25 (µL)

Conc. of Noah %	Peak Height (V)	S.D	R.S.D%
0.006	70.15	0.104	0.148
0.008	69.50	0.048	0.070
0.02	68.08	0.127	0.187
0.1	33.07	0.102	0.308
0.2	2.50	0.012	0.462

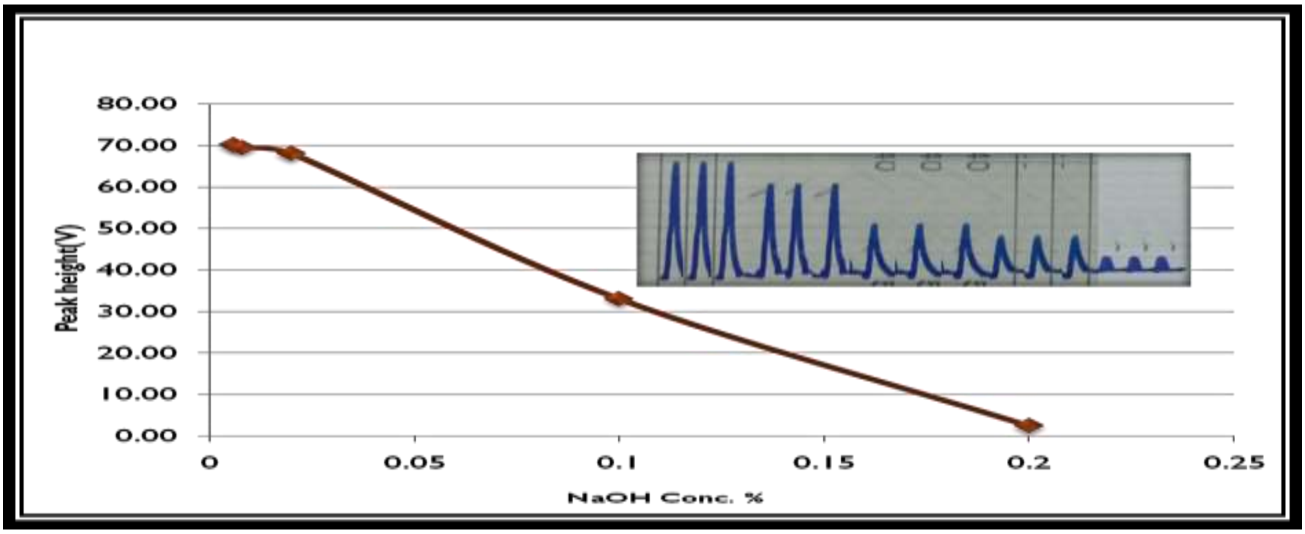

Fig. 7: Effect of base concentration NaOH on the peak height

Calibration Curve in CFIA Method

A series of H_2O_2 with different concentrations were prepared to study the calibration curve. The calibration curve showed linearity over the range of (0.06-0.8) % (R^2=0.9923), as shown in Figure (8) and Table (5). The detection limit at S/N=3 was 0.02%.

Table5: Effect of H_2O_2 concentration on the peak height at; flow rate (12.40 mL min^{-1}), 200 ppm of Fe (III), volume of mixture (sample, reagent, base) =196.25 (µL) and 0.2% of Noah

Conc. Of H_2O_2 %	Mean of Peak Height in (V)	S.D	RSD%
0.06	34.00	0.058	0.169
0.1	32.00	0.252	0.781
0.2	25.75	0.000	0.000
0.4	20.00	0.000	0.000
0.6	12.00	0.006	0.048
0.8	2.00	0.012	0.574

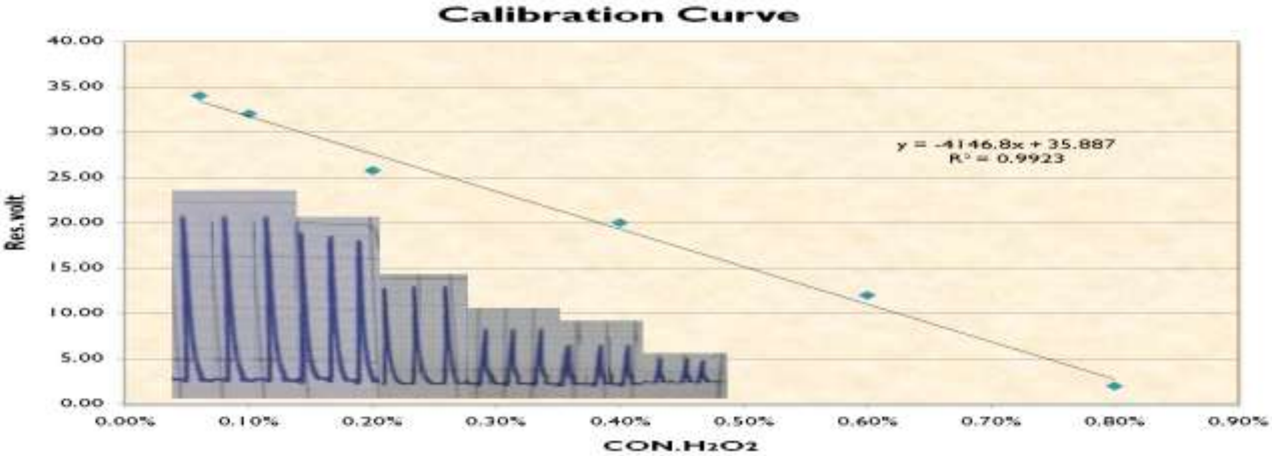

Fig. 8 Calibration curve for H_2O_2

Repeatability

Repeatability can be conducted by injection many times of the sample solution at least six injections. Two concentrations 0.1 and 0.6 % of H_2O_2 were used to study the repeatability of measurements. Six successive injections were measured under the optimum conditions. The obtained results which are tabulated in Table (6) and Figure (9).

Table 6: The repeatability of responses for the two concentrations 0.1 and 0.6% at flow rate (12.40 mL min^{-1}), 200 ppm of Fe(III), volume of mixture (H_2O_2, $K_3[Fe(CN)_6]$, Noah)= 196.75 (µL) and 0.2% of Noah

Conc. of H_2O_2 %	Peak Height (V) n = 6	S.D	RSD%
0.1	32.00	0.04	0.122
0.6	12.00	0.02	0.178

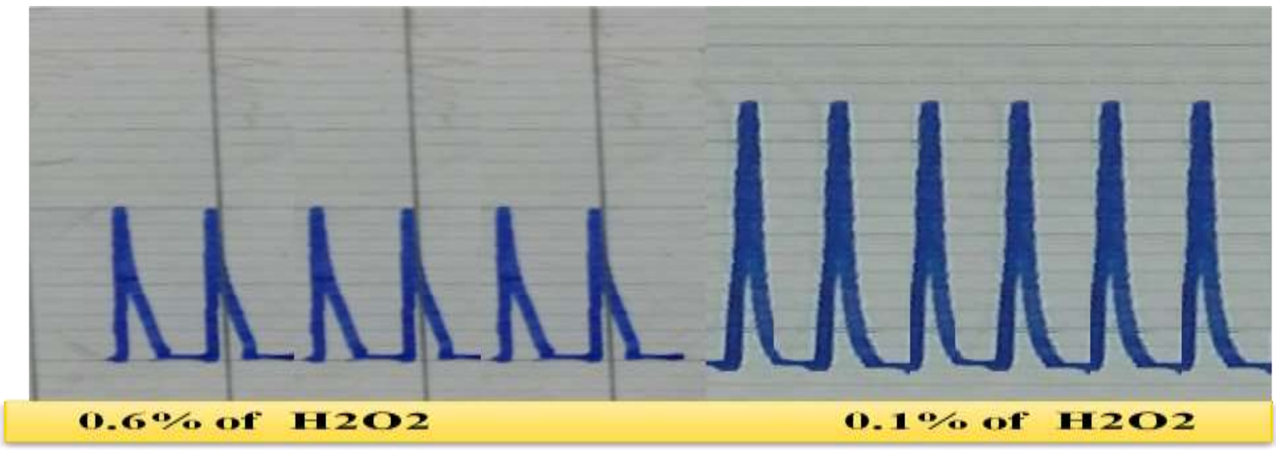

Fig. 9: Repeatability for 0.1 % and 0.6 % of H_2O_2 in CFIA unit

Study of Dead Volume

Three experiments were conducted to study the dead volume to investigate the efficiency of innovative design, however, the dead volume is zero, and it means significant results. three experiments were carried out (a) the mixture of H_2O_2 with Noah was injected in the loop ,the carrier was distilled water, it was found no response estimated and (b) H_2O_2 was injected in the loop and the carrier was distilled water ,there was no response, and (c) injected Noah in the loop and the carrier was distilled water ,there was no response, then injected the mixture of the complex compound $K_3[Fe(CN)_6]$ and H_2O_2 with Noah in order to form and measure the peak height. The result shown in Figure (10).

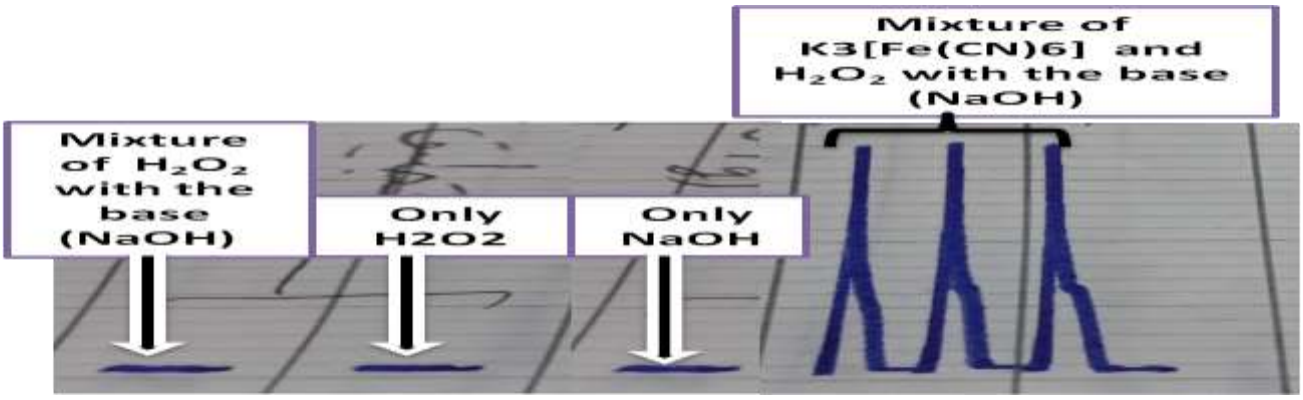

Fig. 10: Study of dead volume

Applications

The proposed CFIA method was successfully applied for the determination of H_2O_2 in disinfecting solution and water samples (Table 7). The method characterized by high precision, which were represented by a recovery.

Table 7: Applications CFIA method for determination H_2O_2

Sample	Actual concentration %	Found concentration H_2O_2%	Mean of peak height (v)	S.D	R.S.D%	E_r%	Recovery%
Tap water	0.2	0.2	41.21	0.179	0.435	0.00	100
	0.06	0.06	42.51	0.093	0.219	0.00	100
Drinking water	0.2	0.2	41.17	0.208	0.506	0.00	100
	0.06	0.06	42.20	0.100	0.237	0.00	100
Antiseptic solution	0.2	0.209	26.00	0.00	0.00	4.50	104.5
	0.6	0.58	11.55	0.00	0.00	-3.33	96.67

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