

Flow injection analysis system with smart phone for the determination of total phenolic compounds in wastewater using Folin Cioclteu reagent

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Abstract

Smart phones are playing a big role in daily life and are being employed as supporting hardware in a variety of chemical analysis applications. Flow injection analysis method for the rapid, simple, and straightforward determination of total phenols using Folin-Ciocalteu reagent. The samples were injected into a carrier stream to react with reagent and detected by spectrophotometer at 765nm. The experimental conditions were optimized such as flow rate (3mL/min) of reagent and carrier, reagent volume (0.4mL), length of reaction coil (50 cm) and concentration of reagent (4×10^{-4} mg.L⁻¹). A good linear calibration curve of gallic acid in the range of (250-2000 mg.L⁻¹) was obtained, (R= 0.9990), the limit of detection was in the amount of (0.0102 mg.L⁻¹). The method was successfully applied for the determination of the total phenols in wastewater.

Key Words : reagent , Folin-Ciocalteu , wastewater , total phenolic compounds , flow injection analysis

1. Introduction

The most practical method for processing, transferring, and sharing data and images is via a smart phone. They are also inexpensive, generally accessible, user-friendly, and portable gadgets. Additionally, Bluetooth and Internet connectivity enable analysis to be performed anywhere is convenient ⁽¹⁾. Smart phones are advanced mobile devices with operating systems capable of handling complex computing tasks, such as the downloading, mobile applications, online browsing

and email .A developing trend in business and academia is to create sensors that work with smart phones in order to profit from their portability, connection, usability, and ubiquitous connectivity ⁽²⁾.In addition to detecting enzymatic urea hydrolysis in microfluidic systems ⁽³⁾ and spectral iron detection ⁽⁴⁾ , now smart phone-based techniques offer promising methods of (F- ion) detection⁽⁵⁾, environmental sensors ⁽⁶⁾ and determination iron (II) in a pharmaceutical formulation⁽⁷⁾ .Flow injection analysis was chosen as an quantitative analysis. Because of its ability to analyze several samples, a very quick response time, tiny sample size injection, and excellent accuracy ⁽⁸⁾ .A liquid sample must be injected into a carrier solution that is moving in a continuous to the detector that records continuously the changes in absorbance ^(9,10) .Low sample and reagent use, partial and repeatable development of the related processes, and other characteristics of flow analysis are inherent⁽¹¹⁾.Phenol an aromatic organic molecule consisting of a hydroxyl functional group (-OH) .Phenolic derivative compounds widely present in the environment,are used to make dyes ,polymers,medicines,and other organic molecules.Skin absorption, inhalation, ingestion,and a number of other methods can all result in phenol poisoning.If people are exposed to high doses of phenol, they could die ⁽¹²⁾), Many methods have been reported in literature for estimating total phenolic compounds such as determination total phenolic compounds in tea samples by Flow injection analysis system ⁽¹³⁾ ,automatic method for the determination of Folin-Ciocalteu reducing capacity in food products ⁽¹⁴⁾ .Flow injection system successful and fast method after adding smart phone technology to the design .

2. Materials and Methods

2.1. Chemicals

All solutions needed in the determination of total phenols by Folin-Ciocalteu reagent method include :

2.1.1. phenol solution

A stock solution of phenol was prepared by dissolving 1.00 g of phenol in a small amount of boiled distilled water in order to increase solubility and diluted to a liter. Each milliliter of this solution contains one milligram of phenol .

2.1.2. Sodium carbonate (BHD , England)

A 75 g. L⁻¹ was prepared by dissolve in double –distilled water .

2.1.3. reagent

The 3000 mg. L⁻¹ of gallic acid (Fluka,Switzerland) stock solution was prepared by dissolve 3g of gallic acid in 1000 mL of distilled water ⁽¹³⁾ .

2.2. Equipment

UV-Visible Spectrophotometer , EMC-11UV,Emclab, Germany,(single Beam) .Electric balance ,Bp301S , Sartorius , Germany .Smart phone device (Galaxy A30s) . India

2.3. Flow injection analysis system

A schematic diagram for semi- automation design flow injection analysis system with smart phone show in fug.1.peristaltic pump was pumped reagent(RS1) and sodium carbonate(RS2) , sample injection valve ,in to carrier stream (RC1,RC2)reaction coil .The absorbance was measured by UV-VIS spectrophotometer at 765nm ,flow signals were recorded by smart phone .

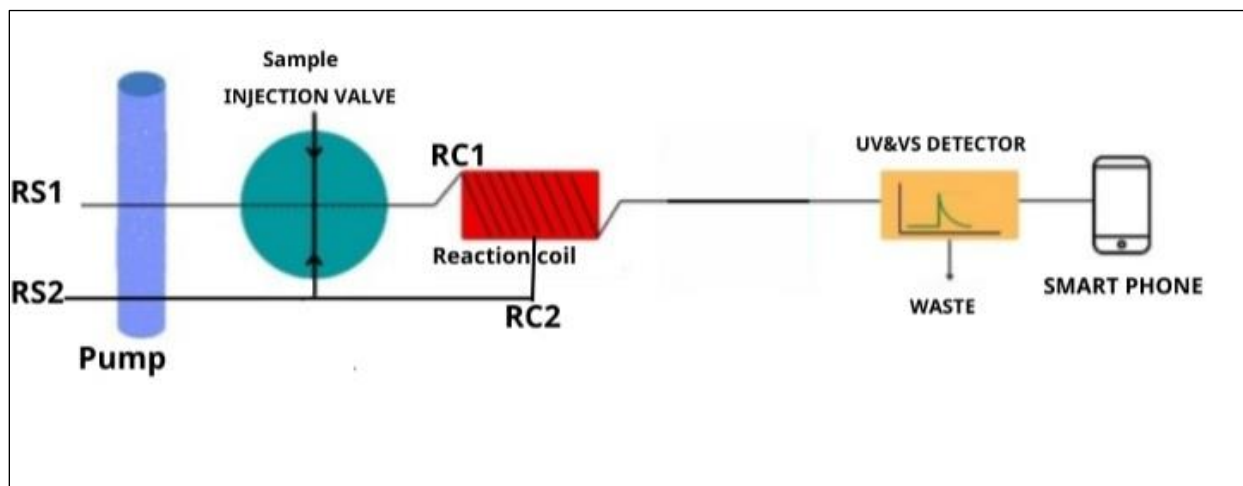


Figure 1. Semi – automation design using flow injection analysis system with smart phone

3.Results and Discussion

The effect of flow rate was studied on (Phenol and reagent) solutions by changing the flow rate with a range (1- 5) mL/min .The results showed that the optimum flow rate was (3mL/min) , then the peak height decrease because of low

measurement sensitivity in high speed due to the lack of time for the components to remain in the measurement cell and the increased mitigation which is increasing with increasing speed ,(figure 2 A) .

For the purpose of studying the effect of the reaction coil length at the peak height different lengths were used of the reaction coil length ranged from (25-150cm) , the results indicated that the optimal length was (50 cm).It was obtained at this length on the best peak height , then the height was lower as a result of an increased dilution ,accompanying increased coil length ,(figure 2 B) .

Concentration was studied on the process of reagent .It was between (1×10^4 - 5×10^4) mg. L⁻¹ .The results indicated that the optimal concentration was (4×10^4 mg.L⁻¹) ,the absorbance become maximal and it was obtained on the best peak height, then the height was lower as a result of an increased dilution, after this concentration the peak height was decreased due to low measurement sensitivity,(figure 2C) .

The effect of volume of Folin-Ciocalteu reagent in the range (0.1-0.5) mL .The volume was (0.4mL) giving the best peak height an increase in volume reduces the peak height,due to the increased dilution of converging areas when they reach the detector,(figure 2D) .

repeatability of FIA Unit was measured Using Folin-Ciocalteu reagent with (500 mg.L⁻¹) from standard solution of phenol ,Table 2,figure 5.

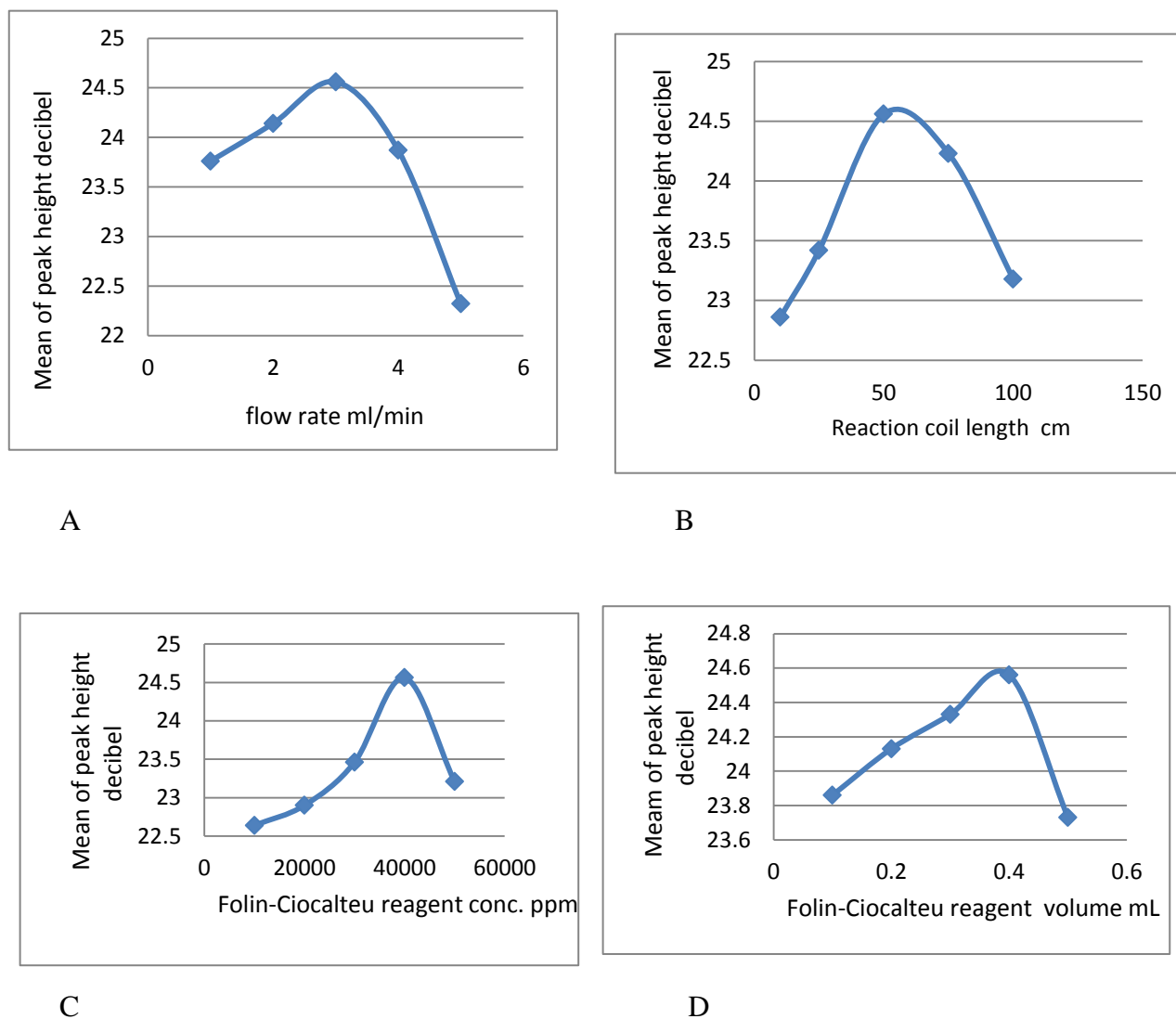


Figure 2. The Factors Affecting the Flow Injection System and choosing the Optimal Conditions

3.1. Flow injection analysis method

All solutions ,flow rate was set at 3mL/min.The sample solution standard , gallic acid solution were pumped into carrier stream and reacted with 0.4mL of 4×10^4 mg.L⁻¹ folin-Ciocalteu reagent in 50cm reaction coil (RC1) .After that,merged with the sodium carbonate (RC2).The absorbance changes were measured by UV-VIS spectrophotometer at 765nm. flow signals were recorded by smart phone show in figure 1 .

3.2. Calibration Curve in FIA Method Using Folin – Ciocalteu reagent

Series of concentrations of gallic acid was prepared .The curve was between (25-3000) mg L⁻¹ ,the study showed that the linearity of the results ranged from (250-2000) mg L⁻¹ , then there was a negative deviation, and the detection

limit was 0.0102 mg.L⁻¹(3 readings of blank) ,as in Table 1 , (figure 3).The proposed standard approach was determined to have good agreement .Relationship between decibel range and the time was measured by smart phone using (SR) application ,figure 4).

Table 1. Calibration Curve in FIA Method Using Folin-Ciocalteu

Gallic acid conc. mg L ⁻¹	Peak height dB			Mean of peak height dB	SD	RSD%
250	3.46	3.41	3.42	3.43	0.026	0.758
500	6.61	6.62	6.61	6.61	0.005	0.075
750	9.12	9.10	9.11	9.11	0.01	0.109
1000	12.31	12.36	12.32	12.33	0.026	0.210
1250	15.87	15.89	15.88	15.88	0.01	0.062
1500	18.41	18.42	18.46	18.43	0.026	0.141
1750	22.18	22.19	22.18	22.18	0.005	0.022
2000	24.56	24.57	24.56	24.56	0.005	0.020

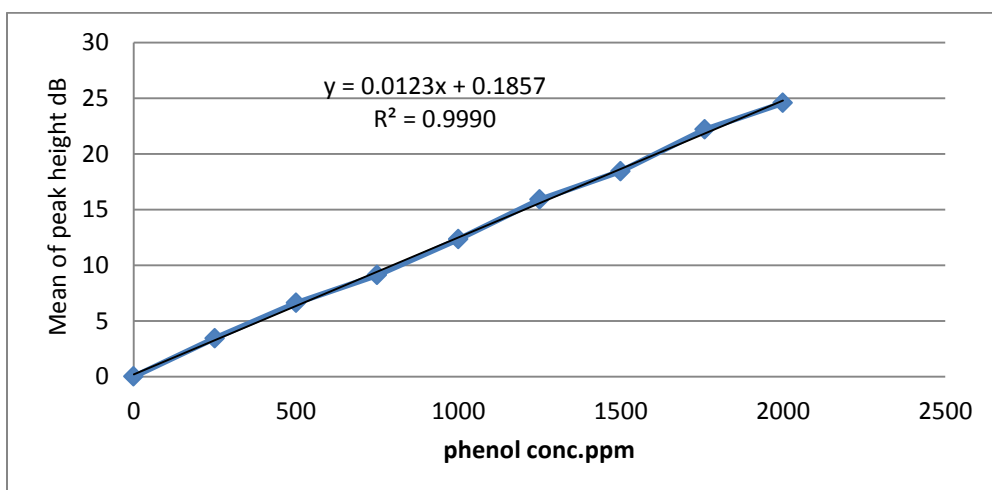


Figure 3. Calibration Curve in FIA method using Folin-Ciocalteu reagent

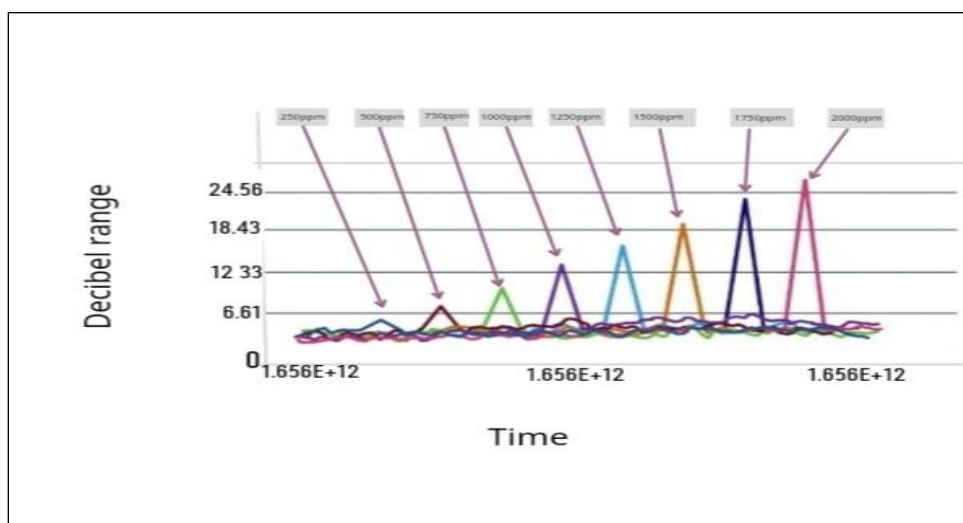


Figure 4. The change in concentration of standard solutions of gallic acid in FIA method using Folin-Ciocalteu reagent

3.3.Repeatability of FIA Unit Using Folin-Ciocalteu reagent

To study the precision and accuracy of the design system , repeatability of FIA Unit Using Folin-Ciocalteu reagent with (500 mg L⁻¹) from standard solution of phenol as in(Table 2 ,figure 5) ,the relative standard deviation for 10 time .

Table 2. Repeatability of FIA Unit Using Folin-Ciocalteu reagent

No.of peak height	1	2	3	4	5	6	7	8	9	10	Mean of peak	SD	RSD%
Peak height dB	6.61	6.61	6.60	6.61	6.60	6.61	6.60	6.61	6.61	6.61	6.607	0.0048	0.0726

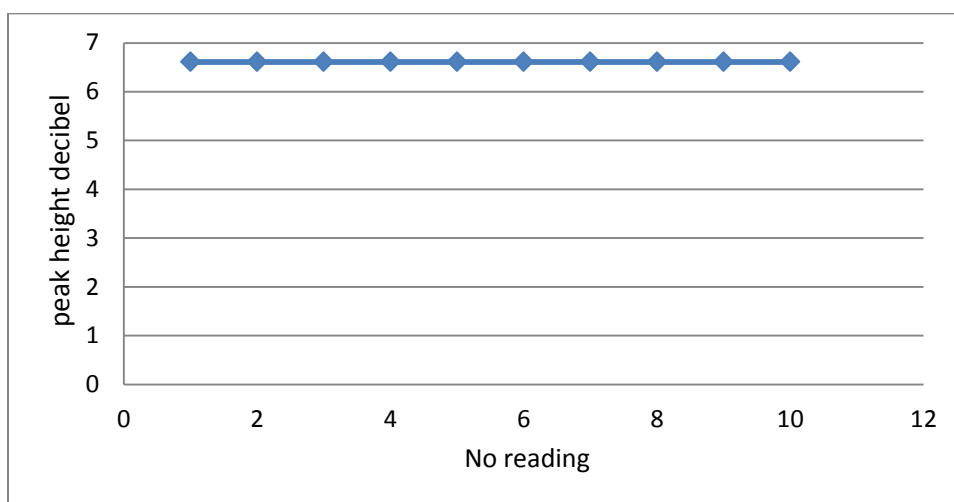


Figure 5. Repeatability of FIA Unit Using Folin – Ciocalteu reagent

3.4. Analytical of wastewater samples :

The samples were provided by an environmental monitoring station and injected into the Flow injection system without any prior preparation after being diluted with 0.05 mol L⁻¹ H₂SO₄ to fit within the calibration plot. Because more phenols were found and the response factors in the suggested technique were greater than for the traditional method spectrophotometer . show in Table 3 .The total phenolic compounds in wastewater were determined using the suggested approach. With a correlation coefficient of (0.9990),

Table 3. The results of the examination of two stations by the traditional method and the new method .

Analytical samples mg.L ⁻¹	Traditional method mg.L ⁻¹	The new method mg.L ⁻¹
Station 1	483	490
Station 2	485	494

3.5. Conclusion

For the purpose of determining the total phenolic chemicals in wastewater, a sample rapid and reliable flow injection analysis system using a smart phone was developed in the study. The proposed of method detection limit was 0.011 mg . L⁻¹ .

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