Determination of Nickel and Cobalt in Cosmetic Products Marketed in Iraq Using Spectrophotometric and Microfluidic Paper-based Analytical Device (µPADs) Platform

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Abstract:

Two quantitative, environment-friendly and easily monitored assays for Ni (II) and Co (III) ions analysis in different lipstick samples collected from 500-Iraqi dinars stores located in Baghdad were introduced. The study was based on the reaction of nickel (II) ions with dimethylglyoxime (DMG) reagent and the reaction of cobalt (III) ions with 1-nitroso-2-naphthol (NN) reagent to produce colored products. The color change was measured by spectrophotometric method at 565 nm and 430 nm for Ni and Co, respectively, with linear calibration graphs in the concentration range 0.25-100 mg L^{-1} (Ni) and 0.5-100 mg L^{-1} (Co) and LOD and LOQ of 0.11 mg L^{-1} and 0.36 mg L^{-1} (Ni), and 0.15 mg L^{-1} and 0.49 mg L^{-1} (Co). The UV/VIS data was compared to the results obtained by a novel microfluidic paper-based analytical device (µPAD) platform offering *in-situ* and cost-effect assay with a similar calibration graph with LOD and LOQ of 0.21 mg L^{-1} and 0.70 mg L^{-1} (Ni), and 0.22 mg L^{-1} and 0.75 mg L^{-1} (Co). The analysis of variance (ANOVA) indicated no significant difference between the UV/VIS, µPAD, and standard atomic absorption spectrometry (AAS) assay F_{tab}= 3.46 is much higher than F_{Stat} = 0.13 (Ni) and F_{tab}= 3.46 is much higher than F_{Stat} = 0.02 (Co). Also, a good correlation between results via the three methods was found. Thus, the µPAD platform offers a solid base for providing valuable information outside centralized laboratories.

Keywords: Cobalt, Conventional instrumentation, Lipstick, µPADs platform, Nickel.

Introduction:

One of the significant issues in the application of cosmetic products is their high contents of heavy metals[1,2]. During the cosmetic production process, heavy metals are not intentionally used but it is simply impurities that can be defined as a byproduct of the manufacturing process, formed by the breakdown of ingredients, or an environmental contaminant of raw ingredients and are not required to be listed on the product labels[3]. The accumulation of heavy metals that enter simultaneously or consecutively to the human body can cause various health problems[4,5]. Lipstick product is a multibillion-dollar industry that has sustained, with the global lipstick market expected to reach 13.4\$ billion by 2024[6,7]. The four essential ingredients of lipsticks include alcohol, wax, oil and pigments. The latter is mainly related to toxic and potentially toxic heavy elements in lipstick

ingredients coloring agents. The color of the cosmetics product may be an indicator of high content of a particular metal such as black, yellow, and red (iron oxide), ultramarine green (sodium aluminum sulfosilicate), Prussian blue (potassium ferrocyanide), carmine (for red color production) and orange containing lead chromate $PbCrO_4$ as an orange pigment [8,9].

The colorimetric method is one of the most generally used approaches for industrial and laboratory applications such as heavy metals detection in various fields such as wastewater, medical, pharmaceutical and cosmetic that has been the prominent alternative to other existing detection methods[10-14]. The microfluidic paper-based analytical device (µPADs) platform has an advantage over spectrophotometric technique due to the nearly -white background of the paper which

makes it easy for the user to visually observe the colorimetric reactions by eye. The assay results can be obtained by the comparison between the colour intensities produced by known analyte concentration to those produced by unknowns at the test zone. Simple equipment such as digital camera, cell phone or scanner and computer-based image analysis software are commonly used to obtain digital data[15-17]. In addition, paper substrate combines the advantage of ubiquity, low cost, as well as its excellent mechanical properties comprising flexibility, lightness, variable thickness, and liquid transport by capillary action and separation capabilities[18,19]. The paper microfluidic platform aims to produce simple, cheap and autonomous devices, depends on the principle of performing multiple tests or detection of several analytes simultaneously on one small piece of paper [20]. Therefore, μPADs have been developed for various applications including health diagnostics[21,22], environmental monitoring[23-27], and food quality testing[28-30].

The motivation of this work was to present a simple spectrophotometric technique alongside a user-friendly and low-cost μPADs platform that can be made from renewable substrate material. These methods were used for the quantitative analysis of nickel (II) and cobalt (III) concentration in different lipstick samples distributed in local and cheap shops via complexion reaction with dimethylglyoxime (DMG) reagent for nickel (II) and 1-nitroso-2-naphthol (NN) solution with cobalt (III) reagent. To the best of our knowledge, it is the first time to develop *in-situ*, simple, economic and with agreement with green chemistry colorimetric methods for the estimation of Ni (II) and Co(III) metals that are not listed as ingredients on commercial lipstick products with unknown brands.

Materials and Methods
Chemical and sample collection

All chemicals and reagents used were of analytical grade, and deionized water (DIW) was used regularly for all aqueous reagents preparation. Different brands of lipsticks were purchased from different 500 ID cosmetic stores in Baghdad the capital of Iraq with unknown origins. The samples were coded as Lip1 (orange), Lip 2 (brown), Lip 3 (coral), Lip 4 (green), Lip 5 (maroon), Lip 6 (golden yellow), Lip 7 (lilac), and Lip 8 (beige). The acid digestion method was used for elemental determination in lipstick following the Nnorom *et al* procedure [31].

Preparation of solutions

Dimethylglyoxime (DMG) solution (0.01M) was prepared by dissolving 0.029 g of DMG (BDH, England) in a minimum amount of ethanol and diluting to 25 mL with deionized water (DIW). **1-Nitroso-2-naphthol (NN) solution (6.25×10^{-4} M)** was prepared by dissolving 0.0027 g of NN (BDH, England) in an aliquot amount of ethanol and completed to 25 mL with DIW. This mixture was heated to achieve solubility using a water bath. **Hydrochloric acid (0.1 M)** was prepared by diluting 0.2 mL of concentrated HCl 1.18 g mL^{-1} (BDH, England) to 25 mL with DIW. **Sodium hydroxide (0.1 M)** was prepared by dissolving 0.1g of NaOH (Fluka, Germany) in 25 mL with DIW. **The stock solution of nickel ion (250 mg L^{-1})** was prepared by dissolving 0.11 g of Ni(SO$_4$). 6H$_2$O (BDH, England) in 100 mL DIW. Working standard solutions were prepared freshly by suitable dilution of stock solution with DIW. **The stock solution of cobalt ion (250 mg L^{-1})** was prepared by dissolving accurately 0.055 g of CoCl$_2$ (BDH, England) in 100 mL DIW. Working solutions were prepared freshly by suitable dilution of stock solution with DIW.

Parameters optimization using batch experiments

Batch experiments were conducted by mixing nickel ions solution with DMG reagent solution for a specific reaction time, pH, and reagent volume for both spectrophotometric and μPADs platform. This was done to achieve the maximum absorbance intensity by varying one parameter at a time and keeping the others fixed. Similar batch experiments were done for cobalt ions after mixing with NN reagent solution.

Spectrophotometric calibration curve
To construct the calibration graph for Ni; after fixing all optimum conditions, the calibration curve was constructed by the addition of an aliquot of Ni (II) 250 mg L^{-1} standard solution to cover the range of calibration graph (0.25-100) mg L^{-1} into a series of 10 mL volumetric flask. Then, 2 mL of DMG 0.01M was added, the content was left to react for five minutes, and diluted to the mark. After 5 minutes, the absorbance was measured at λ_{max} 565 nm versus reagent blank using a Shimadzu double beam recording spectrophotometer (UV-VIS 260 digital)

To construct the calibration graph for Co: After fixing all optimum conditions, the calibration curve was constructed by the addition increasing volumes of Co(III) 250 mg L^{-1} standard solution to cover the calibration graph between (0.5-100) mg L^{-1} to a series of 10 mL volumetric flask. Then 3 mL of NN 6.25×10^{-4} M was added and diluted to the mark. Absorbance was measured immediately at λ_{max} 430 nm versus the blank solution.

Microfluidic paper-based analytical device (μPAD) calibration curve

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Ni calibration curve: The calibration curve was constructed under optimum conditions to estimate Ni(II) concentration by adding the increasing concentration of Ni(II) 250 mg L^{-1} standard solution to cover the calibration range between (0.25-100) mg L^{-1} into the detection circular reaction zone and left to dry at room temperature (~ 25° C). Then, 7 μL of DMG 0.01 M was spotted into each detection circular reaction zone (each paper device contain eight separated spots) producing a coloured product after 5 minutes. The intensities were recorded against blank via photographs taken by Samsung note 9 and Image J software.

Co calibration curve: The calibration curve was constructed under optimum conditions to estimate Co(III) concentration by adding increasing volumes of Co(III) standard solution 250 mg L^{-1} to cover the calibration graph ranged between (0.5-100) mg L^{-1} into the detection circular reaction zone and left to dry at room temperature (~ 25° C). Then, 9 μL of NN 6.25×10^{-4} M was spotted into each detection circular reaction zone (each paper device contain eight separated spots) producing a coloured product immediately. The intensities were recorded against blank prepared in the same condition without the Co (III) standard solution via photographs taken by Samsung note 9 and Image J software program.

Analysis of paper device images

Digital photographs were taken for the colorimetric detection circular reaction zone by Samsung Note 9 camera held within 15 cm using a ruler from the paper device held on a white background and transferred to the computer for analysis using Image J software program (National Institutes of Health, USA). Akin to a method published by Dawson [32], the images transferred to the image J program were converted to 32-bit greyscale. Then, a circle was drawn around each detection circular reaction zone and the signal intensity (number of pixels) were measured. This process was repeated for all subsequent detection circular reaction zones (without alteration in area size). One detection circular reaction zone was left blank to subtract from each sample detection circular reaction zone.

Analysis of commercial lipstick samples

The analysis of commercial lipstick samples to determine the nickel and cobalt ions concentration was done using the standard addition method to omit the effect of interferences in both spectrophotometric and μPADs methods [33]. For the nickel spectrophotometric method, increment concentration (0, 5, 10 and 50) mg L^{-1} of Ni (II) standard solution was added to 1mL of digested lipstick sample solution. Afterwards, 2 mL of DMG 0.01M was added to three volumetric flasks (5 mL) and allowed to react for 5 minutes. The absorbance was measured at λ_{max} 565 nm. Similarly, for μPADs method, 5 μL of Ni(II) standard solution increasing concentration (0, 5, 10 and 50) mg L^{-1} was added to detection circular reaction zone, then 5 μL of digested lipstick sample solution was added using Eppendorf micropipette and left to dry at room temperature (~ 25° C). Afterwards, 7 μL of DMG 0.01 M was pipetted to the mixture and allow to react for 5 minutes, then, images were captured using Samsung Note 9 camera and analysed using the Image J software programme. The same for the cobalt spectrophotometric method; increment concentration (0, 5, 25 and 75) mg L^{-1} of Co (III) standard solution was added to 1mL of digested lipstick samples. Afterwards, 3 mL of NN 6.25×10^{-4} M was added to the three volumetric flasks. The absorbance was measured at λ_{max} 430 nm versus reagent blank immediately. Similarly, for μPADs method, increasing concentration (0, 5, 25 and 75) mg L^{-1} of Co (III) standard solution was added to detection circular reaction zone, then 5 μL of digested lipstick solution was added using Eppendorf micropipette and left to dry at room temperature (~ 25° C). Afterwards, 7 μL of NN 6.25×10^{-4} M was pipetted to the mixture and images were captured using a Samsung Note 9 camera and analysed using the Image J program.

Results and Discussion:

1. Nickel and Cobalt measurements via conventional spectrophotometric analysis

The complexion reaction between nickel(250 mg L^{-1}) and DMG (0.01 M) in an aqueous solution, and cobalt (250 mg L^{-1}) and NN (6.25×10^{-4} M) led to producing pink and orange products after five minutes and immediately for nickel and cobalt, respectively against reagent blank is shown in Fig.1 (I, II). The colored products were found to have maximum absorbance at 565 nm for nickel and 430 nm for cobalt.

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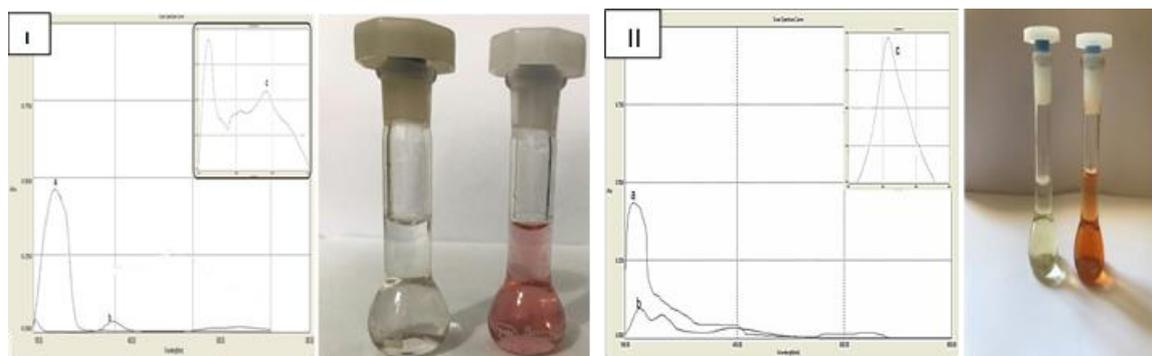
Figure 1. (I) Absorption spectra of complex formed by nickel reacting with DMG versus reagent blank. (II) Absorption spectra of complex formed by cobalt reacting with NN versus reagent blank.

Based on literature; two molecules of DMG reagent react with nickel (II) to form a colored solution[34] Scheme 1 (a), while three molecules of NN reagents tend to form complex with cobaltous as the NN ligand oxidize the Co(III) ions into its trivalent state that allows selective and quick complex formation [35,36] Scheme 1(b).
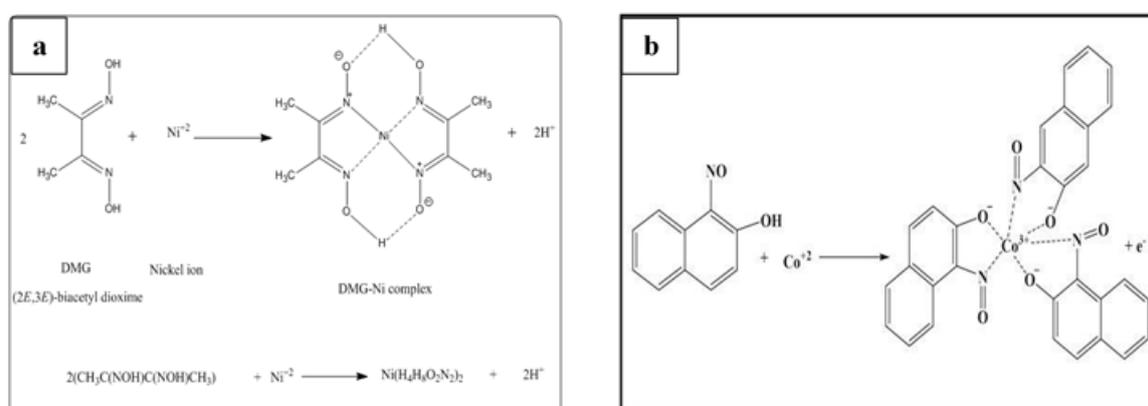
Scheme 1. (a) Illustrate the mechanism of nickel (II) metal with DMG reagent reaction to form the colored complex. (b) Illustrate the mechanism of cobalt (III) metal with NN reagent reaction to form the colored complex.

The parameters that affect color product formation were optimized by employing a 25 mg L^{-1} solution for nickel and 50 mg L^{-1} for cobalt solution. The absorbance was measured at 565 nm for nickel and 430 nm for cobalt, respectively against the reagent blank. Preliminary data for nickel complex formation showed that the addition of acid leads to solution turbidity while the base addition resulted in complex precipitation. Therefore, an aqueous medium was the best medium to achieve a clear pink solution. For the optimization of DMG reagent volume, different volumes (0.5-2.5) mL were studied. The results indicated that the absorbance increased with the increase of reagent volume up to 2 mL then there was a decrease in absorbance Fig. 2 (a). Next, the optimum reaction time was investigated. An increase in the colored product up to five minutes. Afterward, a plateau decrease in the absorbance was observed for 35 minutes Fig. 2 (b). The pink complex was found to be stable for 2 hours.

The same batch experiments for parameters optimization were done on cobalt reaction. The orange product formed immediately in an aqueous solution, while a precipitate was formed after the addition of hydrochloric acid. On the other hand, the addition of sodium hydroxide caused the complex suspension. The optimum NN reagent volume was 3 mL which gave the highest absorbance Fig. 3 (a). The complex formed immediately and a slight decrease was seen with time until 35 minutes Fig. 3 (b). The orange complex was stable for at least six hours.

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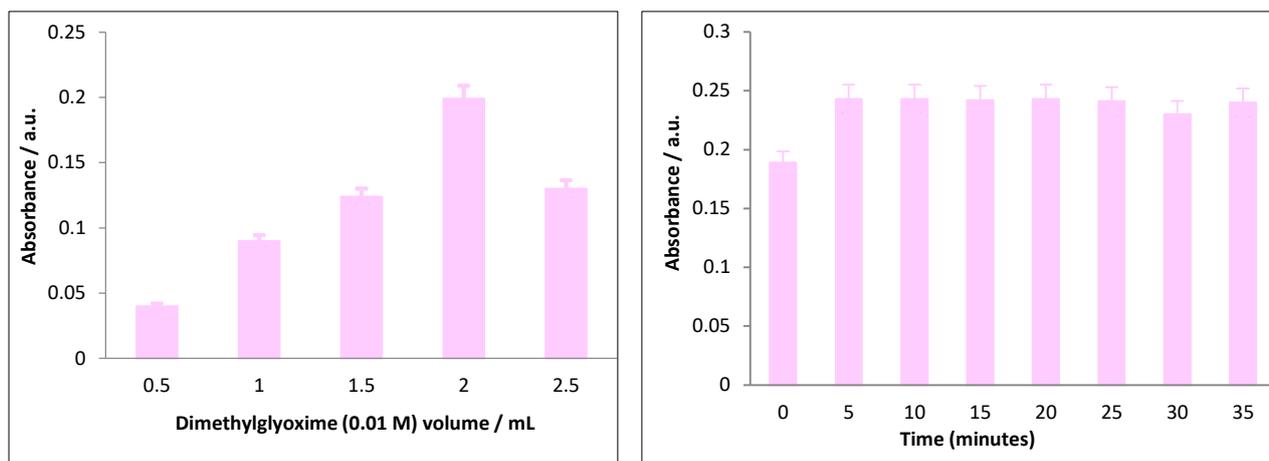
Figure 2. Influence of (a) DMG solution 0.01 M volume ranging from (0.5-2.5) mL, (b) reaction time ranging from (0-35 minutes) parameters on absorbance intensity of spectrophotometric batch experiments via the formation of the metal complex with nickel (II) 25 mg L^{-1} solution.
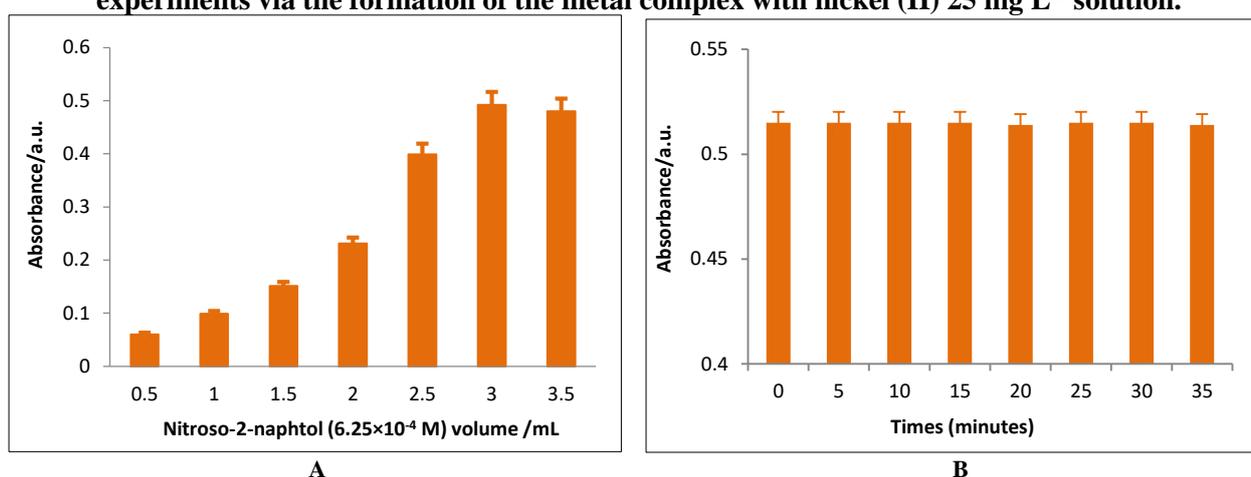
Figure 3. Influence of (a) NN solution 6.25×10^{-4} M volume ranging from (0.5-3.5) mL, (b) reaction time ranging from (0-35 minutes) parameters on absorbance intensity of spectrophotometric batch experiments via the formation of the metal complex with cobalt 50 mg L^{-1} solution.

After the optimization of different parameters, a linear calibration graph was constructed for nickel over the range (0.25 – 100) mg L^{-1} and cobalt over the range (0.5-100) mg L^{-1}, respectively Fig. 4 (a) and (b). Analytical values summarized in Table 1 show the good linearity of the proposed method, minor vales of Sb, Sa and Sy/x which indicate the low scattering of calibration curve points and the respective limits of detection and quantification which were found to be 0.11 mg L^{-1} and 0.36 mg L^{-1} for nickel, and 0.15 mg L^{-1} and 0.49 mg L^{-1} for cobalt.
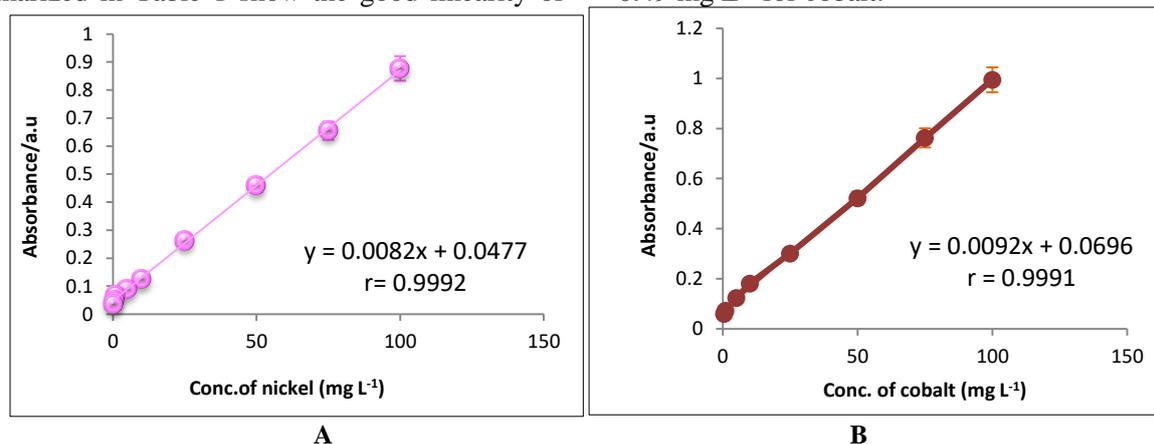
Figure 4. Linear calibration obtained for (a) nickel (0.25-100 mg L^{-1}) and (b) cobalt (0.5-100 mg L^{-1}), n=3.

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2. Nickel and cobalt measurements via µPADs

The paper microfluidic µPADs is considered as an easy-to-use and simple alternative to conventional spectrophotometer procedure towards the construction of a system that can be used for routine analysis outside centralized laboratories. The preliminary investigation showed that the patterned green hydrophobic detection circular reaction zone barriers fabricated using a solid wax ink printer can retain fluids inside a paper cellulose porous network. Pink and orange colours were developed after adding DMG reagent to nickel solution and NN reagent to cobalt solution to form nickel and cobalt complexes, respectively on the paper device as can be seen in Fig. 5 (a, b, and c).

Initial experiments were carried out using the µPADs platform to achieve the maximum colour intensity by spotting reagents on the paper device.

Various volumes of DMG reagent in the range (1-9) µL were optimized and 7 µL was chosen as the best volume that gives the maximum colour intensity Fig. 6 (a), as the addition of reagent may lead to reagent leakage outside detection circular reaction zone. In contrast, insufficient volumes of reagents may lead to a decrease in colour intensity. The required reaction time was investigated over a time interval (0-35) and showed that the reaction is accomplished quickly reaching the highest colour intensity by five minutes. Similarly, the optimization of cobalt reaction parameters was done. The best NN reagent volume that gave the higher colour intensity was 7 µL and therefore was chosen for further experiments Fig. 7 (a). The orange complex formed immediately and there was a plateau decrease until 35 minutes Fig. 7 (b).
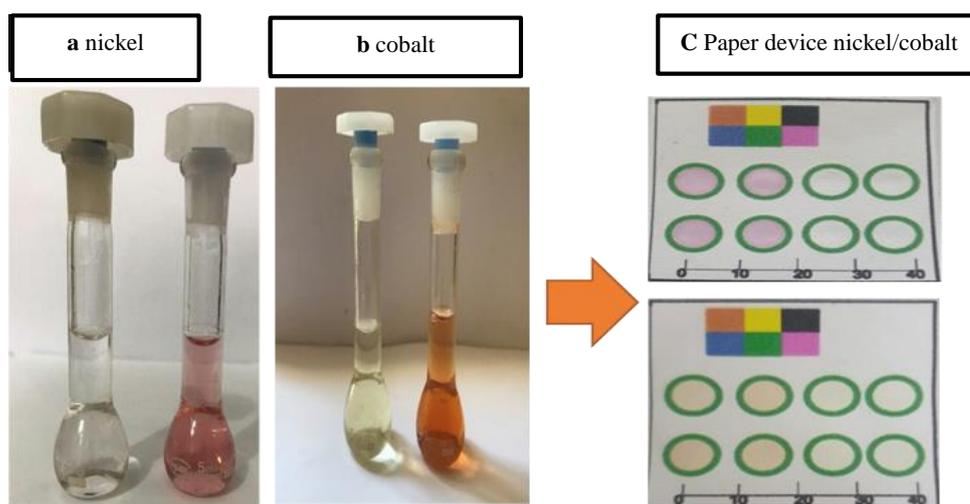
Figure 5. Adaptation of traditional spectrophotometric method to the paper-based platform. (a) The reaction of nickel with DMG in alkaline conditions to produce pink dye. (b) The reaction of cobalt with NN in alkaline conditions to produce orange dye. (c) Reactions were performed on a paper microfluidic device with reagents and analyte added to the detection circular reaction zone sequentially.
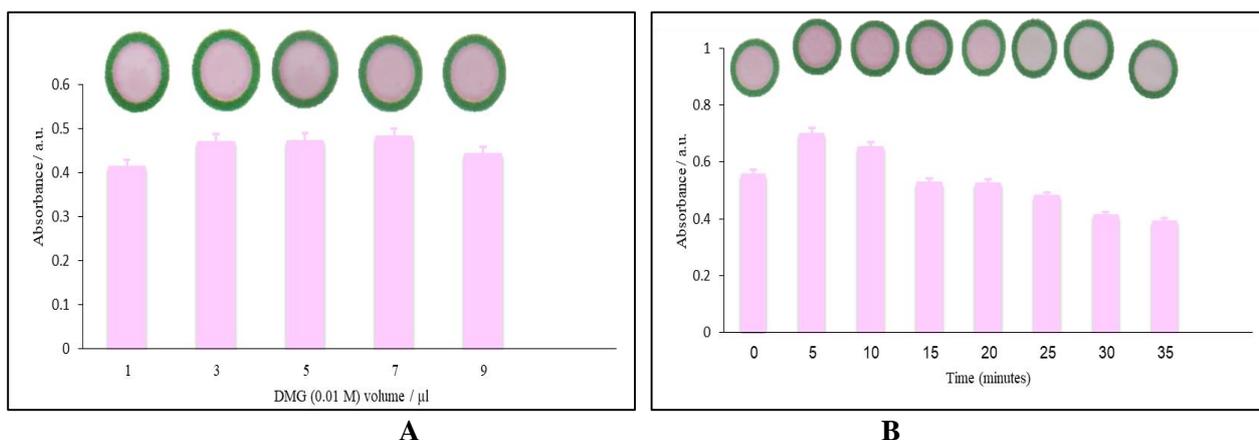
Figure 6. Influence of (a) DMG solution 0.01M volume ranging from (1-9) µL, (b) reaction time ranging from (0-35 minutes) parameters on the color intensity of µPAD platform experiments via the formation of the metal complex with nickel (II) 25 mg L^{-1} solution.
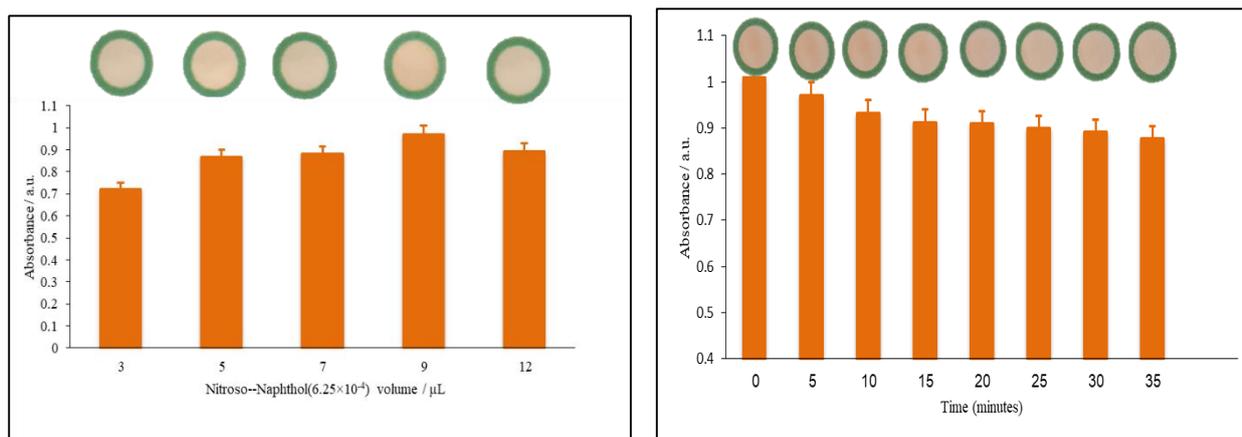
<div align="center">A</div>   <div align="center">B</div>

Figure 7. Influence of (a) NN solution 6.25×10^{-4} M volume ranging from (1-12) µL, (b) reaction time ranging from (0-35 minutes) parameters on the color intensity of µPAD platform experiments via the formation of the metal complex with cobalt (III) 50 mg L^{-1} solution.

After the optimization of different parameters, a linear calibration graph was constructed for nickel over the range (0.25 – 100) mg L^{-1} and cobalt over the range (0.5-100) mg L^{-1}, respectively Fig. 8 (a) and (b). Analytical values, summarized in Table 1, show the good linearity of the proposed method and minor vales of Sb, Sa and Sy/x which indicate the low scattering of calibration curve points. The respective limits of detection and quantification which were found to be 0.21 mg L^{-1} and 0.70 mg L^{-1} for nickel, and 0.22 mg L^{-1} and 0.75 mg L^{-1} for cobalt for spectrophometric and µPAD methods, respectively.
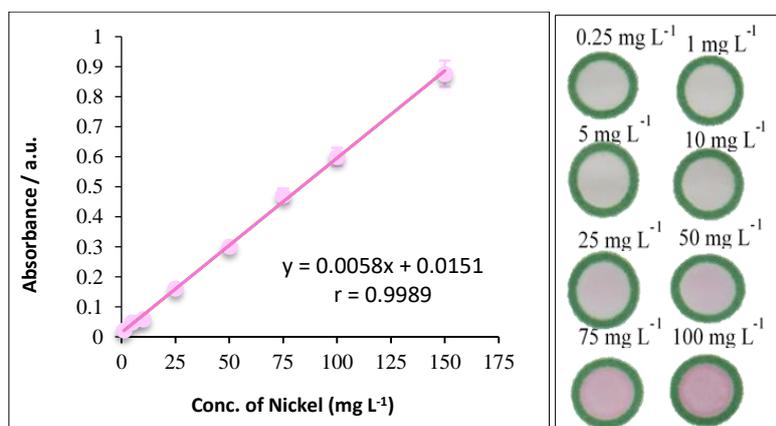
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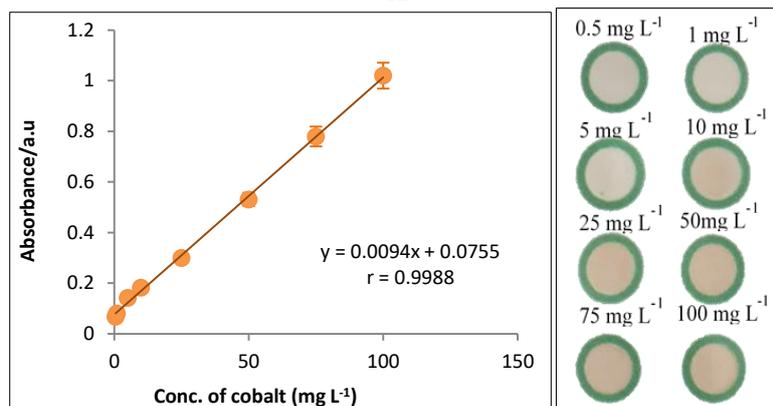
<div align="center">B</div>

Figure 8. Linear calibration was obtained for (a) nickel (0.25-100 mg L^{-1}) and (b) cobalt (0.5-100 mg L^{-1}) insert in (a and b) images of the paper microfluidic device with serial standard concentrations of nickel and cobalt spotted inside the sensing zone, n=3.

Table 1. Analytical values of statistical treatments for nickel and cobalt calibration graphs using spectrophotometric and µPADs methods, respectively.

Parameter	Value			
	Nickel		**Cobalt**	
	Spectrophotometric method	µPADs method	Spectrophotometric method	µPADs method
Correlation coefficient, r	0.9992	0.9989	0.9991	0.9988
Regression equation	y =0.0082x +0.0477	y =0.0058x +0.0151	y = 0.0092x + 0.0696	y = 0.0094x + 0.0755
Slope, b	8.2×10^{-3}	5.8×10^{-3}	9.2×10^{-3}	9.4×10^{-3}
Intercept, a	4.77×10^{-2}	1.51×10^{-2}	6.96×10^{-2}	7.55×10^{-2}
Conf. limit for slope b±tsb	$0.0082 \pm 2.16 \times 10^{-4}$	$0.0058 \pm 3.99 \times 10^{-4}$	$0.0092 \pm 2.77 \times 10^{-4}$	$0.0094 \pm 3.28 \times 10^{-4}$
Conf. limit for intercept a±tsa	$0.0477 \pm 9.886 \times 10^{-3}$	$0.0151 \pm 2.88 \times 10^{-2}$	$0.0696 \pm 1.34 \times 10^{-2}$	$0.0755 \pm 1.59 \times 10^{-2}$
Standard deviation of the residuals, $S_{y/x}$	9.58×10^{-3}	2.3×10^{-2}	1.13×10^{-2}	1.34×10^{-2}
Standard deviation of the slop, S_b	9.14×10^{-5}	1.63×10^{-4}	1.13×10^{-4}	1.34×10^{-4}
Standard deviation of the intercept, S_a	4.19×10^{-3}	1.177×10^{-2}	5.5×10^{-3}	6.5×10^{-3}
Linearity range (µg mL^{-1})	0.25-100	1-150	0.5-100	0.5-100
Molar absorptivity (L.mol^{-1}. cm^{-1})	4.812×10^{4}	3.404×10^{4}	5.421×10^{4}	5.539×10^{4}
Sandell's sensitivity (µg cm^{-2}	1.2×10^{-1}	1.7×10^{-1}	1.1×10^{-1}	1.06×10^{-1}
Limit of detection, LOD (µg mL^{-1})	0.11	0.212	0.15	0.22
Limit of quantification, LOQ (µg mL^{-1})	0.36	0.706	0.49	0.75

The precision and accuracy of nickel and cobalt were calculated and Table 2 summarizes the results for three different concentration measured in triplicate using both spectrophotometry and µPADs platform. Acceptable recoveries(Rec.) values between (99.04–101.13) for nickel and (98.506–101.333) for cobalt, low relative standard deviation (RSD) values in the range of (0.26-1.13) for nickel and (0.63-2.21), and relative error (RE) values < 2 for both metals, confirm good accuracy and precision of the proposed method for nickel(II) and cobalt(III) determination.

Table 2. Statistical data of nickel and cobalt procedures accuracy and precision using spectrophotometric and µPADs detection methods, respectively.

Method	Nickel					Cobalt				
	Taken (µg mL^{-1})	Found (µg mL^{-1})	Rec.%	*RSD%	RE%	Taken (µg mL^{-1})	Found (µg mL^{-1})	Rec.%	*RSD%	RE%
Spectrophotometry	5	4.99	99.80	0.40	-0.20	5	5.04	100.06	0.99	0.60
	10	10.07	101.13	1.13	0.70	25	25.11	100.44	0.80	0.44
	50	49.87	99.74	0.80	-0.26	75	75.28	100.40	0.70	0.40
µPADs	5	4.95	99.04	0.75	-0.96	5	5.06	101.33	2.21	1.33
	10	9.94	99.36	0.26	-0.64	25	24.63	98.51	1.85	-1.49
	50	50.04	100.08	0.91	0.08	75	75.23	100.31	0.63	0.31

*Average of three replicate.

3. Analytical application and comparison method with standard AAS

The results presented in Fig. 9 (a) showed that the concentration of nickel was the highest in the Lipstick sample coded Lip 5 (maroon) while the lowest concentration was Lip 2 (brown). On the other hand, Lip 2 (brown) revealed no cobalt concentration while Lip 4 (green) revealed the highest cobalt concentration Fig. 9 (b). The data obtained from Fig. 9(a and b) indicate also the concentration of nickel and cobalt from spectrophotometric and µPAD methods were not significantly different from conventional AAS[37]. The analysis of variance was calculated using an ANOVA programme including eight samples indicating F_{tab}= 3.46 is much higher than F_{Stat} = 0.13 for nickel and F_{tab}= 3.46 is much higher than F_{Stat} = 0.02 for cobalt Table 3. Therefore, it can be concluded that there was no significant difference between the three methods. Also, comparing the

results from spectrophotometric, paper-based device and atomic absorption spectrophotometric methods showed a good correlation between results via the three methods i.e. (r = 0.9995), (r = 0.9966) and (r = 0.9977), respectively for nickel and (r = 0.9929), (r = 0.9973) and (r = 0.9823), respectively for cobalt Table 4.

A

B
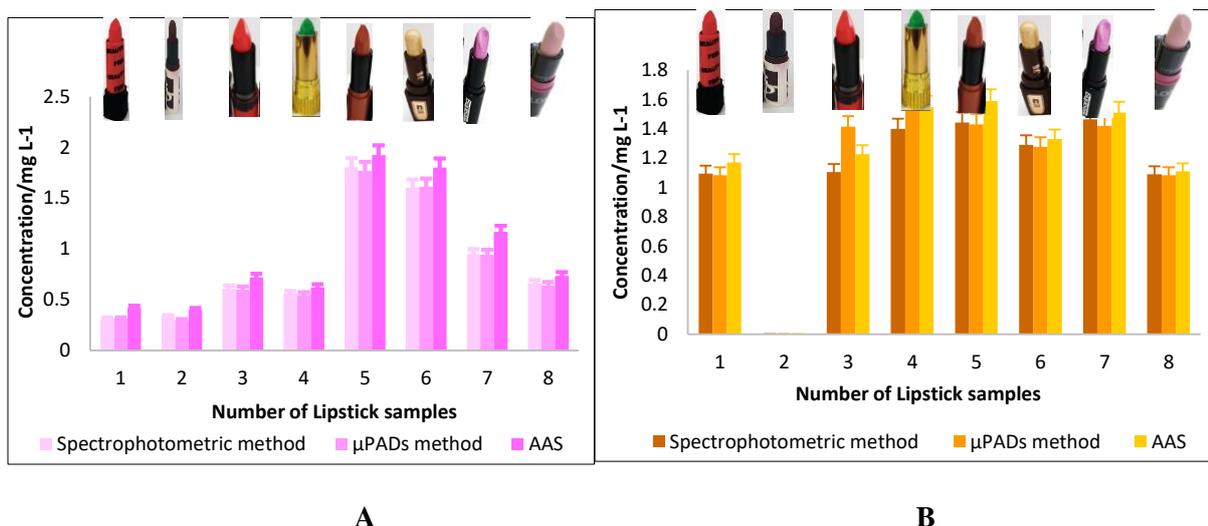
Figure 9. a) Nickel (II) concentrations b) Cobalt (III) concentrations in different lipstick samples collected from 500 I.D. stores, Baghdad, Iraq using UV/VIS, µPADs and AAS methods.

Table 3. Statistical analysis using ANOVA program

Elements	Nickel			Cobalt		
Source of Variation	Between Groups	Within groups	Total	Between Groups	Within groups	Total
ss	0.086	6.980	7.067	0.012	5.059	5.071
DF	2	21	23	2	21	23
MS	0.043	0.332		0.006	0.241	
F_{sta}	0.130			0.025		
F_{tab}	3.466			3.467		

Table 4. Comparison between spectrophotometric, paper-based device, and atomic absorption spectrophotometric methods

Elements	Nickel			Cobalt		
Method	AAS	Spectro.	µPADs	AAS	Spectro.	µPADs
AAS	1			1		
Spectro.	0.9995	1		0.9929	1	
µPADs	0.9966	0.9977	1	0.9973	0.9823	1

Conclusion:

We have employed a complexation reaction for quantitative measurement of nickel and cobalt ions in lipstick samples using spectrophotometry method and innovative paper-based analytical microfluidic device (µPADs) method. The latter device allows *in situ* analysis outside centralized laboratories and an easy-to-use alternative to conventional techniques with quantitative capability. Statistical comparison performed by using ANOVA program of the two methods results with standard AAS method results is in good agreement and with no significant difference.

Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours,

have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Baghdad.

Authors' contributions statement:

EA A. has done the experimental work. JO A. has designed the experiments, analysed the data, wrote, edited, revised, and proofreading the manuscript.

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تقدير النيكل و الكوبلت في مستحضرات التجميل المسوقة في العراق باستخدام الطريقة الطيفية و تقنية السوائل المايكروية الورقية

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الخلاصة :

تم تقديم طريقتين كميتين صديقتين للبيئة وسهلتي المراقبة لتقدير النيكل الثنائي والكوبلت الثلاثي في عينات مختلفة من احمر الشفاه والتي تم جمعها من متاجر 500 دينار عراقي في العاصمة بغداد. يعتمد التحليل على تفاعل ايونات النيكل الثنائية مع كاشف ثنائي ميثيل جليوكسيم (DMG) وتفاعل ايونات الكوبلت الثلاثية مع كاشف 1-2 نايتروزو نفثاول (NN) لتكوين مركبات ملونة يمكن قياسها عند الطولين الموجيين 565 و 430 نانوميتر للنيكل والكوبلت على التوالي. ولقد كانت خطية منحني المعايرة في مدى 100-0.25 مايكروغرام/لتر للنيكل و 100-0.5 مايكروغرام /لتر للكوبلت اما حدود الكشف وحدود التقدير فكانت 0.11 و 0.36 مايكروغرام/لترللنيكل و 0.15 و0.49 مايكروغرام /لترللكوبلت. تم مقارنه الطريقة الطيفية مع تقنية السوائل المايكروية الورقية الجديدة بنفس حدود منحني المعايرة وهي 0.25- 100 مايكروغرام/لتر للنيكل و 100-0.5 مايكروغرام /لترللكوبلت لتقديم طريقة يمكن استخدامها موقعيا وغير مكلفة مقارنة مع الطرق التقليدية بحدود الكشف وحدود التقدير 0.21 و 0.70 مايكروغرام/لترللنيكل و 0.22 و0.75 مايكروغرام/لترللكوبلت. بين تحليل التباين (ANOVA)عدم وجود فرق كبير بين تقنية المطيافية الضوئية وتقنية السوائل المايكروية الورقية وتقنية الامتصاص الذري القياسي حيث ان (Ftab=3.46) اعلى بكثير من (Fstat=0.13) للنيكل وكذلك (Ftab=3.46) اعلى بكثير من (Fstat=0.02) للكوبلت لذلك يمكن استخدام منصة السوائل المايكروية الورقية كقاعدة صلبة لتوفير معلومات قيمة خارج المختبرات .

الكلمات المفتاحية: الكوبلت، الاجهزة التقليدية، احمر الشفاه، منصة جهاز التحليل الورقي، النيكل.