

## Development of an Easy and Selective Approach for Determination of Titanium Dioxide in Commercial Cosmetics by UV-Vis Spectrophotometric Technique

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### ABSTRACT

A simple and selective method was described for the determination of amount of titanium dioxide (TiO<sub>2</sub>) in cosmetics. The method is based on the complex formation of Ti(IV) with hydrogen peroxide in acidic media and monitoring the absorbance of the colored product by spectrophotometric technique at  $\lambda_{\text{max}} = 407$  nm. The effect of experimental parameters on the reaction was investigated and optimized. Under optimal conditions, the calibration graph was linear in the range of 5.0-100  $\mu\text{g mL}^{-1}$  of Ti (IV) with the limit of detection of 3.0  $\mu\text{g mL}^{-1}$ . The validity of the method was evaluated by means of the data statistical analysis. For this purpose, the method was applied to the determination of titanium dioxide in cosmetics, and the results were statistically compared based on t- and F-tests with those obtained by the Inductively coupled plasma-atomic emission spectroscopy (ICP-AES). There was no significant difference between the mean values and the precisions of the two methods at the 95% confidence level. The results showed that the proposed method offers an accuracy and reliable approach for the determination of TiO<sub>2</sub> in commercial cosmetics, and can be suggested as a routine method in quality control laboratories.

**Keywords:** Titanium dioxide, Spectrophotometric, Cosmetics, Validation.

### INTRODUCTION

Today, cosmetics have become an inseparable part of many people's lives, as cosmetics are substances that contain a wide variety of products that are used in face and body care, to accentuate or change a person's appearance. These products including creams, powders, lotions, fragrances, lipsticks, lacquers, lenses, hair dyes, eye and face cosmetics, different types of oils and so forth.<sup>1</sup> The cosmetics industry has also grown to such an extent that anyone can access their desired products at any time. Due to the increasing growth of this field, and the importance of materials in the structure of cosmetics, quality control and their determination in cosmetic

samples is essential.

Generally, cosmetics and personal care products contain a very sophisticated matrix with various components including minerals, herbal powders, oils, waxes, fats, colors, and ultraviolet (UV) preservatives.<sup>2</sup> There are many sunscreen cosmetics that use inorganic or organic UV filters as active ingredients.<sup>3</sup> Typical inorganic compounds in cosmetic or personal care are talc and metal oxides such as titanium dioxide, zinc oxide and iron oxide. Some organic compounds in cosmetics or personal care, can be naturally derived from modified fats and oils, or synthetically produced like petrochemical derivatives. Also, there are many cosmetics that, in addition to the beauty and protective effect of the skin, incorporating the organic or inorganic UV filters as active ingredients.<sup>4</sup> Chemically stable inorganic sunscreen agents, usually metal oxides, are widely employed in high sun protection factor (SPF)

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products.<sup>5</sup> One of the most important ingredients of cosmetics is titanium dioxide.<sup>6</sup> Titanium dioxide is a known photocatalyst and is used in the production of white pigments in the cosmetics and food industries.<sup>7</sup> In addition, It has been used as a pigment for a long period of cosmetic history.<sup>8</sup>

Inorganic compounds in cosmetics may undergo retention and act directly in the skin or be absorbed through the skin into the blood,<sup>9</sup> accumulate in the body and exert toxic effects in various organs.<sup>10</sup> Some cases of topical allergic contact dermatitis and systemic effects due to the metals present in cosmetics have been reported.<sup>11</sup> Also, studies of scientific data show that commercially available cosmetics products may contain large amounts of toxic metals,<sup>12</sup> which can be hazardous for human health.<sup>13</sup> For this reason, it is important to measure the ingredients in cosmetics in small amounts with a quick and easy method in cosmetic samples.<sup>14</sup>

The determination of the content of TiO<sub>2</sub> in sunscreen samples has been attempted with different techniques,<sup>15</sup> such as volumetry,<sup>16</sup> energy-dispersive X-ray fluorescence (EDXRF),<sup>17, 18</sup> transmission electron microscopy (TEM),<sup>19</sup> portable raman spectrometry,<sup>20</sup> atomic absorption spectroscopy (AAS),<sup>21</sup> X-ray fluorescence spectrometry, inductively coupled plasma-mass spectrometry (ICP-MS),<sup>22</sup> and inductively coupled plasma-atomic emission spectrometer (ICP-AES).<sup>23</sup> Although tool-based analytical methods are best for titanium analysis,<sup>24</sup> it is difficult for small cosmetics companies because of the high cost of analysis.<sup>25</sup> Spectrophotometric methods have a special place in the determination of titanium because of their simplicity, precision, and high sensitivity.<sup>26-28</sup>

Therefore, considering the important role of titanium dioxide in cosmetics, the aim of the proposed study is to evaluate the feasibility of using UV-Vis spectrophotometry as a simple, selective and user-friendly method for direct determination determine TiO<sub>2</sub> in cosmetics.

## **Experimental**

### **Apparatus**

UV-Vis spectrophotometer Model 3310 from Hitachi Company (Japan) with 1cm quartz cells was used for all the absorption measurements. All spectral measurements were performed using a blank solution as a reference. Magnetic stirrer (Hei Tec- Hidolph) and thermostatically controlled water bath (Model Hh-S4) were applied. Inductively coupled plasma -atomic emission spectrometer (Perkin Elmer PE Optima 5300DV) was used for comparison with a validation of the method.

### **Reagents and materials**

All chemicals used in this study were analytical grade and used without any purification. Sulfuric acid, nitric acid, hydrogen peroxide (30% (w/w)), ammonium sulfate, and titanium dioxide were purchased with high purity from Merck. All cosmetic products were purchased from the cosmetics store in Tehran- Iran.

### **Experimental procedure**

To prepare a stock solution of Ti(IV), an appropriate amount of TiO<sub>2</sub> was weighed and placed in a beaker. For dissolving and digestion of TiO<sub>2</sub>, 4 g ammonium sulfate and 10 ml sulfuric acid were added. The mixture was heated to a boil until white vapors were observed. After that, the solution was cooled to room temperature and transferred to a 100 mL volumetric flask, and diluted to the mark with double distilled water. It was used as a stock solution (300 µg mL<sup>-1</sup> Ti(IV)). A 150 mM of hydrogen peroxide solution was prepared from 30% (w/w) hydrogen peroxide solution. The inorganic solvent mixture was prepared by dissolving 4 g ammonium sulfate and 10 mL sulfuric acid in 100 mL deionized water and used for dilution of standard solutions.

For determination of TiO<sub>2</sub> quantities, under optimum conditions, aliquots of solutions containing Ti(IV), so that final concentration would be in the range of 5.0 –100 µg mL<sup>-1</sup>, 1.5 mL of 150 mM of hydrogen peroxide solution were transferred into 10 mL volumetric flasks. After the formation of yellow color; the solutions were diluted to the

mark with the inorganic solvent mixture. A portion of the solution was transferred into a 1 cm quartz cell to measure the absorbance at 407 nm against a reagent as blank as the reference.

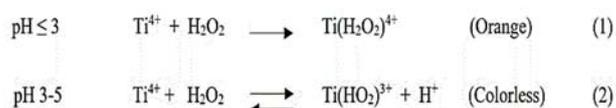
#### **Sample preparation of commercial cosmetics for TiO<sub>2</sub> determination**

In order to determine TiO<sub>2</sub> in cosmetics, the samples were placed in the furnace prior to analysis to burn the organic matter of the samples and give carbon-free ash. For this purpose, different weights of cosmetics were weighed on crucibles and placed in the furnace at 600 ° C for 2 h. After cooling of samples to room temperature, the acid digestion method was used to determine TiO<sub>2</sub>. The method involved adding a mixture of 10 ml nitric acid (65% w/w) and 5 ml concentrated sulfuric acid to carbon-free ash cosmetic samples in a beaker and placing it in a water bath until the sample was dissolved. In order to complete the dissolution of the resulting solution, the beaker was heated to 200 °C until the dense fume of nitric acid was disappeared. Finally, 5 mL concentrated sulfuric acid and 4 gr ammonium sulfate were added to dissolve the TiO<sub>2</sub>. The process was finished with the appearance of the white vapors. The resulting solution was cooled and carefully transferred into a 100 mL volumetric flask. The solution was diluted to the mark with double-distilled water.

#### **Results and discussion**

The reaction of Ti(IV) with hydrogen peroxide in an acidic media (pH ≤ 3) gives a colored complex according to scheme 1.<sup>29</sup> Thus, it can be a successful method for the spectrophotometric determination of Ti.<sup>30</sup> Fig. 1 shows the absorption spectra of the complex which display a maximum absorbance at λ<sub>max</sub> = 407 nm. Therefore, all examinations were performed at this wavelength. In order to achieve the highest performance and sensitivity, the influence of effective parameters on the reaction was investigated and optimum conditions were obtained. The optimization procedures were done by keeping all parameters to be constant and optimizing one each time. This optimization procedure may not lead to the actual

optimum, but will definitely improve the analytical method. These parameters included the effects of concentration of reagent, the concentration of sulfuric acid, time, and temperature on reaction.



**Scheme 1. Colored complex formation of Ti(IV) with hydrogen peroxide in an acidic media**

#### **Effect of the hydrogen peroxide concentration**

The effect of hydrogen peroxide concentration on the absorbance of the system and determination of Ti(IV) was considered in the range of 50-200 mM. As Fig. 2 shows, the absorbance of the system was increased by increasing the concentration of hydrogen peroxide up to 150 mM and remain nearly constant at higher concentrations that show that the concentration of hydrogen peroxide has a tremendous effect on the formation of the color product between titanium and hydrogen peroxide. So, the concentration of 150 mM hydrogen peroxide reagent was selected as the optimum concentration for further investigation in the developed method.

#### **Effect of the sulfuric acid concentration**

The reaction between Ti(IV) and hydrogen peroxide takes place in acidic media. The importance and the influence of sulfuric acid concentration of absorbance of the product were studied in the range of 0.1-3 M of sulfuric acid. The results revealed (Fig. 3) that the absorbance increases by increasing the acid concentration up to 1.8 M and does not change at higher concentrations. Therefore, a concentration of 1.8 M sulfuric acid was applied in the proposed method.

#### **Effect of temperature on the sensitivity**

The effect of temperature on the formation of color product between Ti(IV) and hydrogen peroxide was investigated in the range of 15-60 °C was investigated. The

results showed that temperature has no effect on the reaction efficiency and this is one of the special advantages of the method, which makes it a temperature-independent and simple method which can be done without the need for heating equipment.

#### **Study on stability of Ti(IV)-H<sub>2</sub>O<sub>2</sub> complex**

The influence of time on the complex formation of Ti(IV) with hydrogen peroxide was studied in a range of 1-10-min. The results showed that the reaction occurs immediately after the addition of hydrogen peroxide, and reaction of Ti(IV) with hydrogen peroxide is rapid and the reaction time is not critical. Also, it indicates that by following the absorbance at different times, the absorbance values were constant which shows that the Ti(IV)-H<sub>2</sub>O<sub>2</sub> complex is a stable complex (Fig. 4). Therefore, the method has good repeatability and there is an improvement in figures of merit.

#### **Effect of ionic strength**

In order to study ionic strength on the reaction stability and resistance of the method, different concentrations of NaCl, KCl, Na<sub>2</sub>SO<sub>4</sub>, and KNO<sub>3</sub> solutions were separately added into the solution in the range of 150- 750 mM. The results show that the increasing the electrolytes concentration did not any effects on the absorbance of the system and stability of the Ti(IV)-H<sub>2</sub>O<sub>2</sub> complex. Therefore, the proposed method to be robust, since no statistically significant differences were found when samples were subjected to these solutions.

#### **Analytical performance**

The analytical features that show the performance of the proposed method are presented in Table 1. The calibration graph was constructed by using different concentrations of standard solutions of Ti(IV) that subjected to the developed method and determined by the UV-Vis spectrophotometric method. Under optimum conditions, a calibration curve was linear in the range of 5-100 µg mL<sup>-1</sup> of Ti(IV). The regression equation for proposed the method is,  $A = 0.0151 C + 0.0066$ , with a regression coefficient (r) of 0.9994 (n=10), which A is the

absorbance at  $\lambda_{\max} = 407$  nm. The detection limit of the method, based on signal to noise ratio of 3, was 3 µg mL<sup>-1</sup>.

#### **Selectivity of method**

To study the selectivity of the proposed method, the effect of various species on the determination of Ti(IV) was tested under the optimum conditions. For this purpose, sample solutions containing 45 µg mL<sup>-1</sup> Ti(IV) and different concentrations of various ions were prepared and the developed procedure was applied. The tolerance limit was defined as the concentration of added species that caused a relative error of less than  $\pm 5\%$ . The results showed that Na<sup>+</sup>, K<sup>+</sup>, NH<sub>4</sub><sup>+</sup>, Ca<sup>2+</sup>, Pb<sup>2+</sup>, Co<sup>2+</sup>, Sn<sup>4+</sup>, Mg<sup>2+</sup>, Cu<sup>2+</sup>, Cd<sup>2+</sup>, Ni<sup>2+</sup>, Fe<sup>2+</sup>, Ca<sup>2+</sup>, CO<sub>3</sub><sup>2-</sup>, PO<sub>4</sub><sup>3-</sup>, SO<sub>4</sub><sup>2-</sup>, NO<sub>3</sub><sup>-</sup>, Cl<sup>-</sup>, Br<sup>-</sup>, F<sup>-</sup> did not interfere on the determination of Ti(IV) in a tolerance ratio ( $w_{\text{Species}}/w_{\text{Ti}^{4+}}$ ) 500:1. This shows the good selectivity of the proposed method.

#### **Statistical data analysis**

The applicability of the developed method for the determination of Ti(IV) in cosmetic samples was investigated. Sample processing was discussed in the section of sample preparation. So, all cosmetics samples were subjected to the suggested method. The results of this investigation were shown in Table 2. The results revealed that the suggested method can be successfully applied for the determination of Ti(IV) in cosmetic samples. Validation and statistical comparison of the method were done with the determination of Ti(IV) in cosmetic samples by ICP-AES according to condition listed in Table 3. Also, the values of F-test and t-test were calculated, the values obtained from this study are less than the values stated in the Table. So, these results show that no significant differences between the results of the spectrophotometric method and the ICP-AES method at a 95% confidence level, and confirming the good accuracy and precision of the developed method.

#### **Application of method in analysis of cosmetic samples**

The reliability and matrix effects in the proposed method were investigated by the spiking-recovery method.

So different portions of cosmetic samples that prepared in the section of sample preparation were subjected with the spiking-recovery method and Ti(IV) was determined in both spiked and unspiked portions, and the relative recoveries (*R*) were calculated according to Eq. 3. :

$$Recovery (\%) = (C_1 - C_2)/C_3 \quad (3)$$

which *C*<sub>1</sub> the concentration of Ti(IV) after spiking of standard solution and *C*<sub>2</sub> is a real concentration of Ti(IV) in the sample solution, and *C*<sub>3</sub> is the concentration of Ti(IV) that spiked to sample solution. According to the results in Table 4 all recoveries in the range 94-103%. Acceptable recovery results can confirm that the matrix effect cannot have significant effects on the efficiency of the proposed method, and the results indicate that the proposed method has a high potential for the determination

of TiO<sub>2</sub> commercial cosmetic samples.

### Conclusions

Due to the fact that a lot of amounts of titanium dioxide can cause threaten human health. Therefore, monitoring the amount of titanium dioxide in cosmetic samples is important. Our investigation revealed that the proposed method is a simple, sensitive, and user-friendly spectrophotometric procedure for the determination of TiO<sub>2</sub> in a wide dynamic range that can be applied to cosmetics with satisfactory recoveries. Method validation shows that the method has high accuracy and precision and is robust. Therefore, this report can be proposed as a reliable method with high accuracy for the determination of titanium dioxide in cosmetics. Also, the method proved to be suitable for routine quality control of commercial cosmetics and can be extended for further study in this field.

**Table 1. Optical and statistical parameters of regression equation and validation parameters**

Molar absorptivity, $\epsilon(L.mol^{-1} .cm^{-1})$	$7.22 \times 10^2$
Sandal's sensitivity ( $\mu g cm^{-2}$ )	66.3
Color of complex	Yellow
Measurement wavelength <sup>2</sup>	407
Regression equation	A= 0.0151 C +0.0066
Regression coefficient ( <i>r</i> <sup>2</sup> )	0.9994
Slop of calibration curve	0.0151
Linear range ( $\mu g/mL$ )	5.0 – 100
Limit of detection(LOD= 3sb/m)	3.0 $\mu g/mL$

**Table 2. Determination of TiO<sub>2</sub> in Cosmetic formulations by proposed method and comparison of it with ICP-AES method.**

Cosmetics samples	Origin	Wieght of sample	Proposed method		ICP-AES method		t-values (2.78) <sup>a</sup>	F-value (9.28) <sup>a</sup>
			%Found in sample $\pm$ SD	RSD%	Found in sample $\pm$ SD	RSD%		
Paint stick cream	USA	0.2	9.03 $\pm$ 0.015	0.16	9.05 $\pm$ 0.04	0.44	1.33	7.11
		0.3	9.05 $\pm$ 0.073	0.80	9.07 $\pm$ 0.036	0.40	0.57	4.11
		0.4	9.07 $\pm$ 0.108	1.19	9.08 $\pm$ 0.045	0.50	0.2	5.76
BB-cream	France	0.2	4.51 $\pm$ 0.041	0.90	4.54 $\pm$ 0.036	0.79	1.5	1.29

Cosmetics samples	Origin	Wieght of sample	Proposed method		ICP-AES method		t-values (2.78) <sup>a</sup>	F-value (9.28) <sup>a</sup>
			%Found in sample $\pm$ SD	RSD%	Found in sample $\pm$ SD	RSD%		
		0.3	4.49 $\pm$ 0.036	0.80	4.58 $\pm$ 0.017	0.37	3.60	4.48
		0.4	4.51 $\pm$ 0.047	1.03	4.53 $\pm$ 0.041	0.90	0.76	1.31
Cake make-up	France	0.2	2.15 $\pm$ 0.033	1.54	2.13 $\pm$ 0.041	1.92	1.0	1.54
		0.3	2.16 $\pm$ 0.026	1.20	2.14 $\pm$ 0.031	1.44	1.17	1.42
		0.4	2.15 $\pm$ 0.026	1.23	2.14 $\pm$ 0.025	1.16	0.66	1.08
		0.2	3.49 $\pm$ 0.026	0.74	3.48 $\pm$ 0.10	0.28	0.21	7.01
Foundation cream	France	0.3	3.46 $\pm$ 0.036	1.04	3.47 $\pm$ 0.017	0.143	0.66	6.0
		0.4	3.50 $\pm$ 0.017	0.49	3.51 $\pm$ 0.01	0.28	0.83	2.0
		0.2	5.69 $\pm$ 0.030	0.73	5.67 $\pm$ 0.10	0.25	1.21	6.01
Toothpaste	Iran	0.3	5.56 $\pm$ 0.039	1.17	5.58 $\pm$ 0.017	0.151	0.87	7.0
		0.4	5.30 $\pm$ 0.018	0.53	5.32 $\pm$ 0.01	0.24	0.76	2.1

<sup>a</sup> It is the theoretical value based on the paired *t*-test and F-test at the level of significance of  $p = 0.05$

**Table 3. Operating conditions of ICP-AES**

Operating conditions of ICP-AES	
Parameter	Value
Reflected power	1350 W
Radiofrequency (RF) generator	40 MHz, free-running
Auxiliary gas flow rate	0.50 L min <sup>-1</sup>
Viewing mode	Axial
Torch type	Fassel type
Injector, id	Alumina, 2.0 mm
Nebulizer	Gem tip cross flow
Sample uptake flow rate	2 mL min <sup>-1</sup>
Delay time	30 s
Wavelength	334.94 nm
Plasma gas flow rate	15 L min <sup>-1</sup>

**Table 4. Real samples analysis results**

Cosmetics samples	Ti <sup>4+</sup> ( $\mu\text{g mL}^{-1}$ )		
	spiked	Found ( $\mu\text{g mL}^{-1}$ ) $\pm$ <sup>a</sup> SD	Recovery%
TV paint stick	0.0	27.15 $\pm$ 0.015	-
	15	42.66 $\pm$ 1.52	103.4
	30	57.06 $\pm$ 0.89	99.7
	35	63.00 $\pm$ 1.00	102.4

Cosmetics samples	Ti <sup>4+</sup> (µg mL <sup>-1</sup> )		
	spiked	Found (µg mL <sup>-1</sup> ) ± <sup>a</sup> SD	Recovery%
BB-cream	0.0	18.21 ± 0.026	-
	15	32.33 ± 1.52	94.13
	30	47.8 ± 0.75	98.63
	45	63.53 ± 0.64	100.7
Cake make-up	0.0	12.79 ± 0.014	-
	15	27.55 ± 0.32	100
	30	43.03 ± 0.139	100.7
	45	58.3 ± 0.36	101.1
Foundation cream	0.0	14.1 ± 0.1	-
	15	29.56 ± 0.25	103.73
	30	44.73 ± 0.27	102.43
	45	58.5 ± 0.25	98.88
Toothpaste	0.0	13.1 ± 0.11	-
	15	28.54 ± 0.25	104.72
	30	43.73 ± 0.26	100.54
	45	62.00 ± 1.00	99.67

<sup>a</sup> Average of three determinations ± standard deviation.

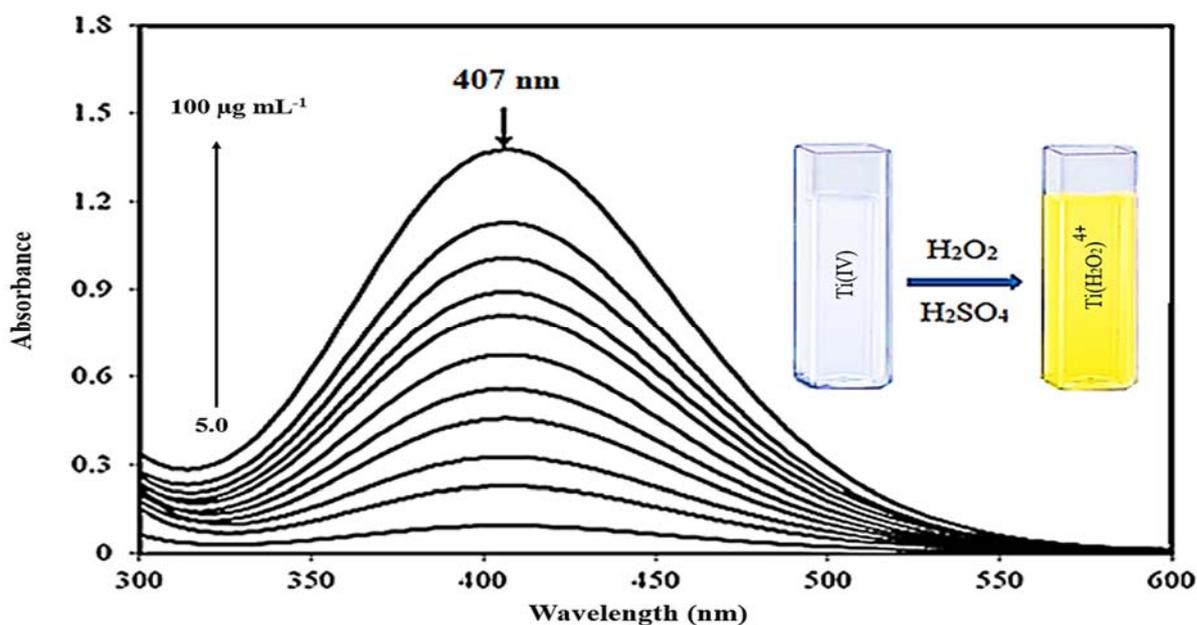
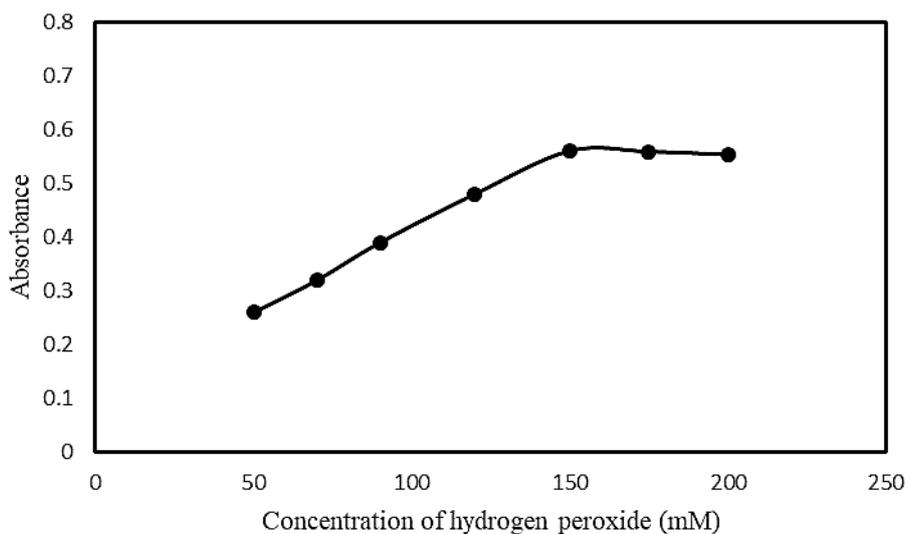
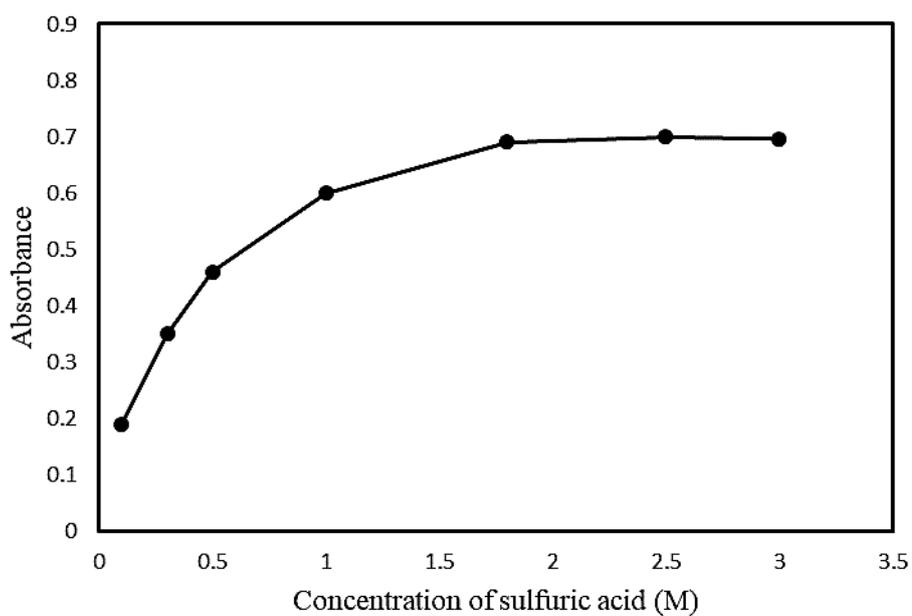


Figure 1. Absorption spectrum resulting from titanium and hydrogen peroxide reaction, Conditions: Concentration of Ti(IV): 5-100 µg mL<sup>-1</sup>, hydrogen peroxide: 150 mM, sulfuric acid: 1.8 M, temperature: 25 °C



**Figure 2: Effect of concentration of hydrogen peroxide on reaction, Conditions: Ti(IV):  $45 \mu\text{g mL}^{-1}$ , sulfuric acid: 1.8 M, temperature:  $25^\circ\text{C}$ .**



**Figure 3: Effect of concentration of sulfuric acid on reaction, Conditions: Ti(IV):  $45 \mu\text{g mL}^{-1}$ , hydrogen peroxide: 150 mM, temperature:  $25^\circ\text{C}$ .**

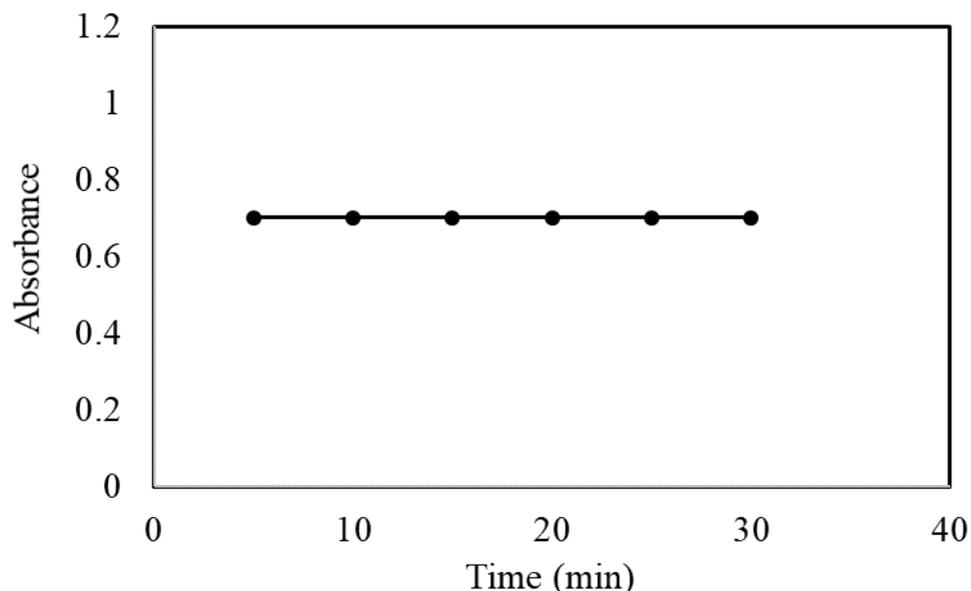


Figure 4: Effect of time on reaction, Conditions: Ti(IV):  $45 \mu\text{g mL}^{-1}$ , hydrogen peroxide: 150 mM, sulfuric acid: 1.8 M

### REFERENCES

- (1) Vimaladevi M. *Textbook of cosmetics*. CBS Publishers & Distributors. 2005.
- (2) De la Calle I, Menta M., Klein M. and Séby F. Screening of TiO<sub>2</sub> and Au nanoparticles in cosmetics and determination of elemental impurities by multiple techniques (DLS, SP-ICP-MS, ICP-MS and ICP-OES). *Talanta*. 2017; 171: 291-306.
- (3) Scher R. K. Cosmetics and ancillary preparations for the care of nails: Composition, chemistry, and adverse reactions. *Journal of the American Academy of Dermatology*. 1982; 6 (4): 523-528.
- (4) Sahota A. *Sustainability: how the cosmetics industry is greening up*. Wiley Online Library. 2014.
- (5) Salvador A. and Chisvert A. Sunscreen analysis: a critical survey on UV filters determination. *Analytica Chimica Acta*. 2005; 537 (1-2): 1-14.
- (6) Maarroof M. N. and Asmaa E. M. Determination of the Immunogenic and Hematologic Effects of Titanium Nanoparticles Manufactured from *Aspergillus flavus* in Vivo." *Jordan Journal of Biological Sciences*. (2019); 12 (2).
- (7) Kaniyas G. Determination of trace elements in eyeshadow face powder and rouge make-up cosmetics by neutron activation analysis. *Journal of radioanalytical and nuclear chemistry*. 1985; 89 (2): 487-496.
- (8) Bunhu T., Kindness A. and Martincigh B.S. Determination of titanium dioxide in commercial sunscreens by inductively coupled plasma-optical emission spectrometry. *South African Journal of Chemistry*. 2011; 64: 139-143.
- (9) Wilke K., Martin A., Terstegen L. and Biel S. A. short history of sweat gland biology. *International journal of cosmetic science*. 2007; 29 (3): 169-179.
- (10) Borowska S. and Brzóska M.M. Metals in cosmetics: implications for human health. *Journal of applied toxicology*. 2015; 35 (6): 551-572.
- (11) Wakefield G. and Stott J. Photostabilization of organic UV-absorbing and anti-oxidant cosmetic components in formulations containing micronized manganese-doped titanium oxide. *Journal of Cosmetic Science*. 2006; 57 (5): 385-395.
- (12) Kockler J., Oelgemöller M., Robertson S. and Glass B. D. Influence of titanium dioxide particle size on the photostability of the chemical UV-filters butyl methoxy dibenzoylmethane and octocrylene in a microemulsion. *Cosmetics*. 2014; 1 (2): 128-139.

- (13) Amro B., Saleh A. and Saja H. H. Innovative Non-Invasive Techniques in Skin Pharmaceutics as Valid Complements in Cosmetic Dermatology Practice. *Jordan Journal of Pharmaceutical Sciences*. (2011); 108(399): 1-14.
- (14) Lu P-J., Huang S-C., Chen Y-P., Chiueh L-C. and Shih D Y-C. Analysis of titanium dioxide and zinc oxide nanoparticles in cosmetics. *Journal of food and drug analysis*. 2015; 23 (3): 587-594.
- (15) Benítez-Martínez S., López-Lorente Á I. and Valcárcel M. Determination of TiO<sub>2</sub> nanoparticles in sunscreen using N-doped graphene quantum dots as a fluorescent probe. *Microchimica Acta*. 2016; 183 (2): 781-789.
- (16) Parsons L. and Vaughan F. The colorimetric determination of titanium dioxide in soaps. *Oil and Soap*. 1941; 18 (3): 64-65.
- (17) Melquiades F., Ferreira D., Appoloni C., Lopes F., Lonni A., Oliveira F. and Duarte J. Titanium dioxide determination in sunscreen by energy dispersive X-ray fluorescence methodology. *Analytica chimica acta*. 2008; 613 (2): 135-143.
- (18) Kawauchi A., Ishida M. and Saitoh I. Measurement of titanium dioxide in cosmetic products with X-ray fluorescence spectrometry. *Spectroscopy letters*. 1996; 29 (2): 345-366.
- (19) Ferreira D. D., Carlos R A., Audrey G L. and Fábio L M. Quantitative Analysis of Titanium Concentration in Cosmetic Sunscreen Products by Portable EDXRF, TEM and Portable Raman Spectrometry. *Latin American Journal of Pharmacy*. 2019; 38 (5): 1008-1013.
- (20) Lercari C., Sartorel B., Sedeo L. and Toninelli G. Determination of titanium dioxide in soaps by furnace atomic absorption spectrometry. *Journal of the American Oil Chemists' Society*. 1983; 60 (4): 856-857.
- (21) Mason Jr. J. T. Quantitative determination of titanium in a commercial sunscreen formulation by atomic absorption spectrometry. *Journal of pharmaceutical sciences*. 1980; 69 (1): 101-102.
- (22) Zachariadis G. and Sahanidou E. Analytical performance of a fast multi-element method for titanium and trace elements determination in cosmetics and pharmaceuticals by ICP-AES. *Open Chemistry*. 2011; 9 (2): 213-217.
- (23) Contado C. and Pagnoni A. TiO<sub>2</sub> in commercial sunscreen lotion: flow field-flow fractionation and ICP-AES together for size analysis. *Analytical chemistry*. 2008; 80 (19): 7594-7608.
- (24) Marczenko Z. and Balcerzak M. *Separation, preconcentration and spectrophotometry in inorganic analysis*. Elsevier: 2000.
- (25) Salvador A., Pascual-Martí M., Adell J., Requeni A. and March J. Analytical methodologies for atomic spectrometric determination of metallic oxides in UV sunscreen creams. *Journal of pharmaceutical and biomedical analysis*. 2000; 22 (2): 301-306.
- (26) Zaky M., Elgendy K. and El-Wahaab A. Spectrophotometric Determination of Titanium (IV) in Steel, Paint and a Cosmetic Cream Using Tannic Acid In Micellar Medium. *Bulletin of Faculty of Science, Zagazig University*. 2020; 2016 (1): 68-76.
- (27) Alahmad W. and Mahmoud A. A. Kinetic study of photocatalytic degradation of several pharmaceuticals assisted by SiO<sub>2</sub>/TiO<sub>2</sub> catalyst in solar bath system. *Jordan Journal of Pharmaceutical Sciences*. (2010); 3(2): 126-136.
- (28) Véber M. and Csanyi L.J. Study of The Formation of The Peroxotitanium (IV) Complex. *Acta Phys. Chem*. 1977; 23(2-3): 297-303.
- (29) Mori M., Shibata M., Kyuno E. and Ito S. Reaction of hydrogen peroxide with titanium (IV) at different pH values. *Bulletin of the Chemical Society of Japan*. 1956; 29 (8): 904-907.
- (30) O'Sullivan D. W. and Tyree M. The kinetics of complex formation between Ti(IV) and hydrogen peroxide. *International Journal of Chemical Kinetics*. 2007; 39 (8): 457-461.

## تطوير نهج سهل وانتقائي لتقدير ثاني أكسيد التيتانيوم في مستحضرات التجميل التجارية باستخدام تقنية قياس الطيف الضوئي بالأشعة المرئية وفوق البنفسجية

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### ملخص

تم وصف طريقة بسيطة وانتقائية لتقدير كمية ثاني أكسيد التيتانيوم (TiO<sub>2</sub>) في مستحضرات التجميل. تعتمد الطريقة على التكوين المعقد لـ Ti (IV) مع بيروكسيد الهيدروجين في الوسط الحمضي ومراقبة امتصاص المنتج الملون بتقنية القياس الطيفي عند  $\lambda_{max} = 407$  نانومتر. تم دراسة تأثير المتغيرات التجريبية على التفاعل وتحسينه. في ظل الظروف المثلى، في ، كان الرسم البياني للمعايرة خطياً في نطاق 5.0-100 ميكروغرام مل من Ti (IV) مع حد اكتشاف 3.0 ميكروغرام مل. تم تقييم صحة الطريقة عن طريق التحليل الإحصائي للبيانات. لهذا الغرض ، تم تطبيق الطريقة لتقدير ثاني أكسيد التيتانيوم في مستحضرات التجميل ، وتمت مقارنة النتائج إحصائياً بناءً على اختبارات -t و F مع تلك التي تم الحصول عليها عن طريق التحليل الطيفي للانبعاثات الذرية بالبلازما المقترنة حديثاً (ICP-AES). لم يكن هناك فرق كبير بين القيم المتوسطة ودقة الطريقتين عند مستوى ثقة 95%. أظهرت النتائج أن الطريقة المقترحة تقدم طريقة دقيقة وموثوقة لتحديد TiO<sub>2</sub> في مستحضرات التجميل التجارية ، ويمكن اقتراحها كطريقة روتينية في مختبرات مراقبة الجودة.

**الكلمات الدالة:** ثاني أكسيد التيتانيوم، مقياس الطيف الضوئي، مستحضرات التجميل، التحقق من الصحة.

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