

## DETERMINATION OF $\alpha$ -TOCOPHEROL IN COSMETOTEXTILES – UV/VIS SPECTROPHOTOMETRIC METHOD

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**Abstract:** Tocopherol is a chemical name for vitamin E that belongs to the group of amphipathic and lipid-soluble compounds that are easily oxidized when subjected to heat, light and alkaline conditions. Vitamin E can be used as active cosmetic substance for functionalization of textiles, acting as anti-ageing agent through gradual release on the skin. The paper deals with cosmetotextiles, carriers of Vitamin E contained in a suspension of cyclodextrine -  $\alpha$ -tocopherol complex, applied in finishing. Research is focused on development of a method for determination of  $\alpha$ -tocopherol in a form metal organic chelate complex using UV/VIS spectrophotometry.

**Keywords:** cosmetotextiles,  $\alpha$ -tocopherol, desorption, UV/VIS spectrophotometric analysis

### 1. Introduction

In the cosmetic industry, vitamin E is used as antioxidant and lipid-soluble compound with active substance. It has moisture binding capacity in aliphatic cosmetic creams, lotions, emulsions, body and face oils for dry skin care as well as for decorative cosmetics like lipsticks. Vitamin E is also successfully applied for various skin diseases. It belongs to the group of lipid-soluble vitamins and is found in nature in many vegetable oils and plant tissues.  $\alpha$ -tocopherol belongs to the group tocopherols that are consisting of a series of related benzopyranols (or methyl tococls). Hydrophobic part of a molecule is C16 side chain, that is saturated with 20-carbon phytyl tails (including the pyranol ring), with variable numbers of methyl groups attached to the benzene ring. The side-chain methyl groups of natural tocopherols are responsible for R,R,R stereochemistry. The four general constituents of the two classes are termed -  $\alpha$  (5,7,8-trimethyl),  $\beta$  (5,8-dimethyl),  $\gamma$  (7,8-dimethyl) and  $\delta$  (8-methyl) (Figure 1) [1, 2].

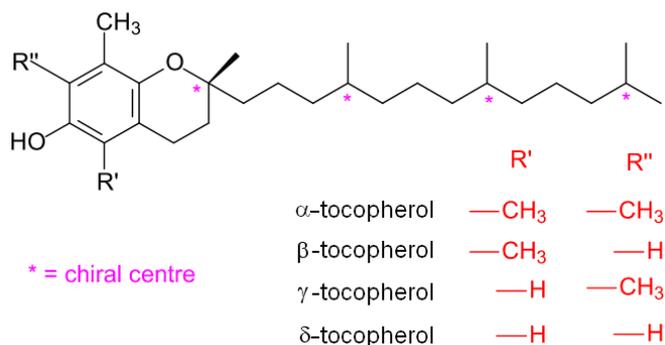
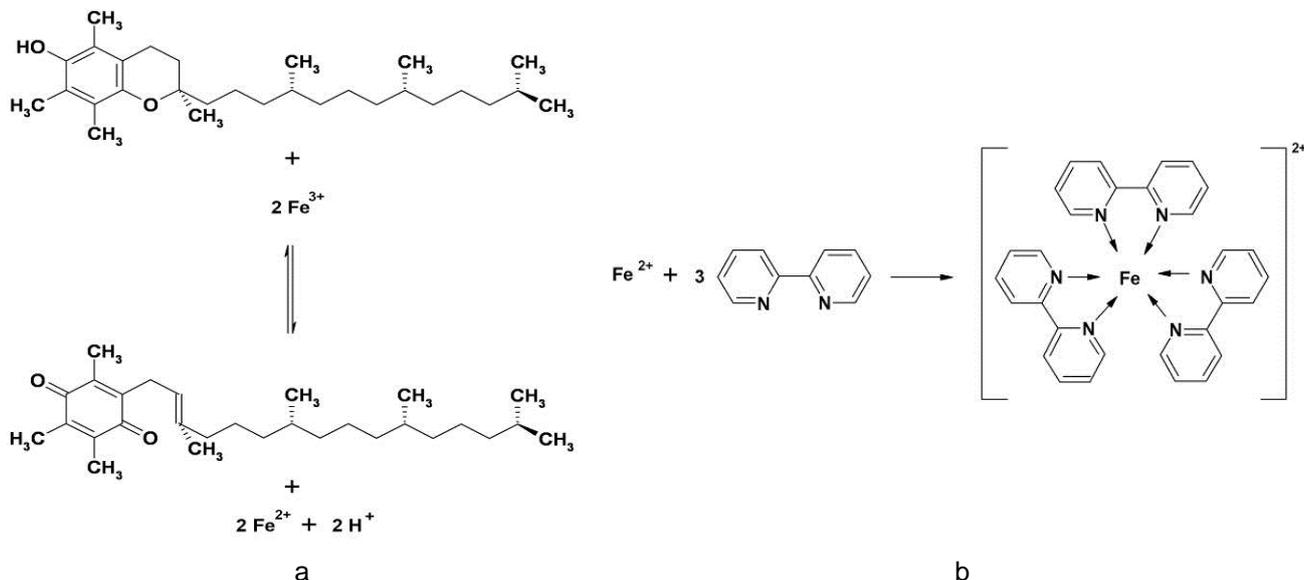


Figure 1. Structure and composition of tocopherols [1]

Vitamin E is well known as a youth source and for the purpose of slow release  $\alpha$ -tocopherol was applied to textiles resulting with the anti-ageing properties [3]. Microencapsulated vitamin E significantly increases the skin moisture and elasticity and also reduce the skin wrinkle and roughness [2]. Temperature and light sensitivity of  $\alpha$ -tocopherol is a reason why commercial products are prepared like complex, e.g. with cyclodextrin [4, 5]. In that form it is more stabile, adequate for application on textile (cosmetotextile) enabling controlled release during wearing [6]. NouWell E was selected for the application on cotton fabrics in this study. It is very important to control a release as well as a lifetime of Vitamin E with a proper methodology. In general, tocopherols can be analysed by gas and liquid chromatography. Quantification of tocopherols also requires flame-ionization and mass spectrometric detection [1, 7]. One of the convenient methods for qualitative detection of  $\alpha$ -tocopherol on the textile and other relating materials is a drop test [8]. The test is

based on a redox reaction between  $\alpha$ -tocopherol and iron (III)-chloride (solution A) that is reduced to iron (II), whereas  $\alpha$ -tocopherol is oxidised to tocoquinone, Figure 2a. After addition of the dipyriddy solution (solution B), the iron (II) ions form a red coloured metal organic chelate complex with dipyriddy, presented in Figure 2b [6].



**Figure 2.** Reaction mechanism: a. redox and b. complexation [8]

The aim of this preliminary study is to identify and quantify  $\alpha$ -tocopherol using a drop test chemicals using the UV/VIS spectrophotometric method. The first step included reference stock solution necessary for baseline or calibration curves. The second step included a spectral identification of desorbed NouWell E (source of  $\alpha$ -tocopherol) from cotton fabrics.

## 2. Materials and methods

The visible absorption spectra of  $\alpha$ -tocopherol were measured in matched quartz cuvettes using a Cary UV/VIS spectrophotometer (Cary 50 Solarscreen, Varian Inc, SAD) at a wavelength from 350 to 700 nm.

The preliminary part of the laboratory work is related to the agent NouWell E, dispersion of cyclodextrine - vitamin E complex (Bezema, Switzerland) that was diluted with methanol and water as necessary for UV/VIS spectrophotometric determination, Table 1.

**Table 1.** Prepared solutions of NouWell E with methanol and water

	NouWell E (ml)	H <sub>2</sub> O (ml)	MeOH (ml)
A	2.0	0.0	2.0
B	2.0	0.5	1.5
C	2.0	1.0	1.0
D	2.0	1.5	0.5
E	2.0	2.0	0.0

Qualitative determination of  $\alpha$ -tocopherol on cotton fabric is carried out by a drop test. Proposed protocol is not adequate for spectrophotometric determination of  $\alpha$ -tocopherol in solution and therefore should be optimized. So it was necessary to find an amount of iron (III) chloride and dipyriddy, which can produce a red coloured complex characterized by specific curve peaks (Table 2).

**Table 2.** Chemicals for spectrophotometric determination of  $\alpha$ -tocopherol

Chemicals	Volume			
	2			
NouWell E (ml)				
FeCl <sub>3</sub> (ml)	0.020	0.0250	0.040	0.050
Dipyriddy (ml)	0.020	0.0250	0.040	0.050

The stability of red coloured complex diluted with methanol and water was tested after 10 s, 30 min, 60 min and 120 min by spectrophotometric determination.

Reference of  $\alpha$ -tocopherol (Merck 613420 | DL-  $\alpha$ -Tocopherol - CAS 10191-41-0 . Calbiochem, purity  $\approx$  98%) was used for calibration procedure. The stock solution (1000 $\mu$ g/ml) was prepared by dissolving this form of  $\alpha$ -tocopherol in methanol. Different concentrations of  $\alpha$ -tocopherol were prepared by diluting of stock solution (0.01 ml, 0.02 ml, 0.05 ml, 0.10 ml, 0.20 ml) with water in volumetric flask (10 ml).

The final step of this work relates to desorption of  $\alpha$ -tocopherol from cotton fabric in plain weave, surface mass of 175.617 g/m<sup>2</sup> and yarn density of (warp/weft 25/25 yarns/cm) that was treated with NouWell E and additives (Table 3) [9].

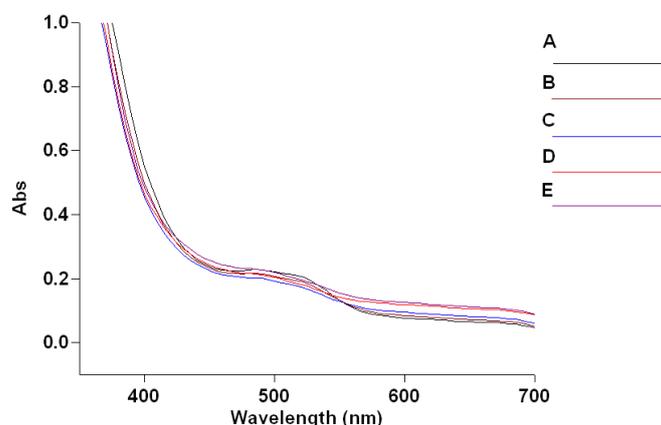
**Table 3.** The composition and concentration of products in recipe applied

Product*	Chemical composition	Concentration (g/l)
NouWell E	cyclodextrine- $\alpha$ -tocopherol complex	100
Sarabid OL	alkyl polyglycol ether	3
Arristan PMD	microdispersion of modified polyurethane	45
Tubingal SMF	microemulsified functional polysiloxane	35

\*All products were supplied by CHT-Bezema, Deutschland . Swiss company

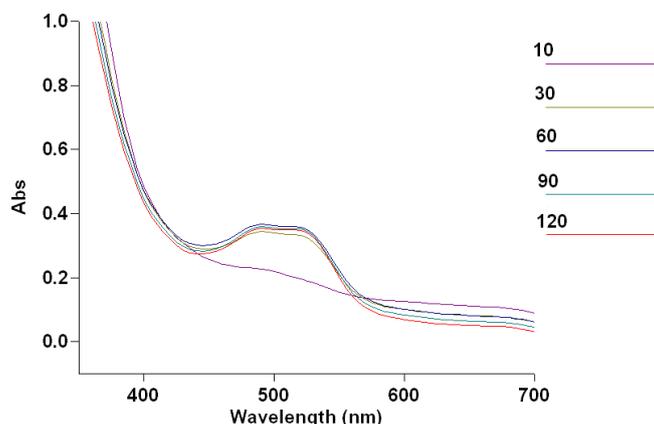
### 3. Results and discussion

UV/VIS spectrophotometric analysis of metal organic chelate complex diluted by different ratio of water and methanol (Table 1) is presented by spectral curves, Figure 3. It is clear that type of solvent has no impact on spectral curves to be created.



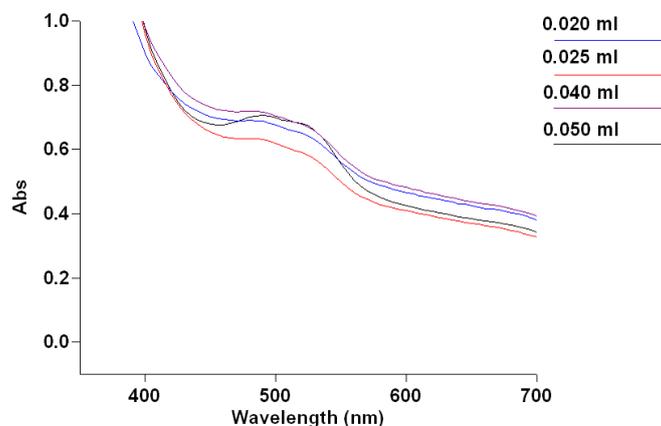
**Figure 3.** Spectral curves of metal organic chelate complex diluted with methanol and water

Colour stability of metal organic chelate complex in variation of standing time (10 s, and 30, 60, 90 & 120 min) was presented in Figure 4. Spectral curves showed that metal organic chelate complex possessed 2 characteristic peaks. It is clear that metal organic chelate complex possess no characteristic peak after 10 s.



**Figure 4.** Spectral curves of metal organic chelate complex versus standing time

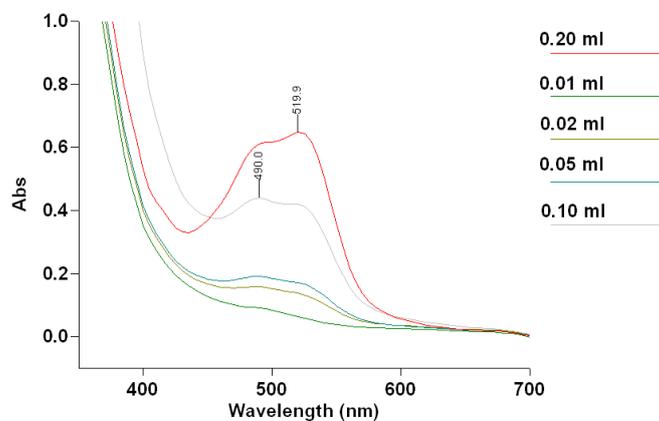
Volumes of chemicals proposed for qualitative determination of  $\alpha$ -tocopherol on cotton fabric by drop test method are not adequate for spectrophotometric determination of  $\alpha$ -tocopherol in solution. So, it was necessary to search an amount of iron (III) chloride and dipyrindil that can produce a red metal organic chelate complex that can be characterized by UV/VIS spectrograms, Figure 5.



**Figure 5.** Spectral curves of metal organic chelate complex

Spectral curves shape of metal organic chelate complex in wavelength range is different. The shape and intensity of a curve in green (2 ml NouWell + 0,025 ml solution A + 0,025 ml solution B) it is selected as the best. Therefore, it was the reason why this concentration was applied for further spectral analysis tests.

The next step was preparation of stock reference solutions required for calibration curves, Figure 6. Spectral curves showed characteristic peaks, observed at 490 nm (0,01 mg/ml of stock solution) and 520 nm (0,02 mg/ml of stock solution).

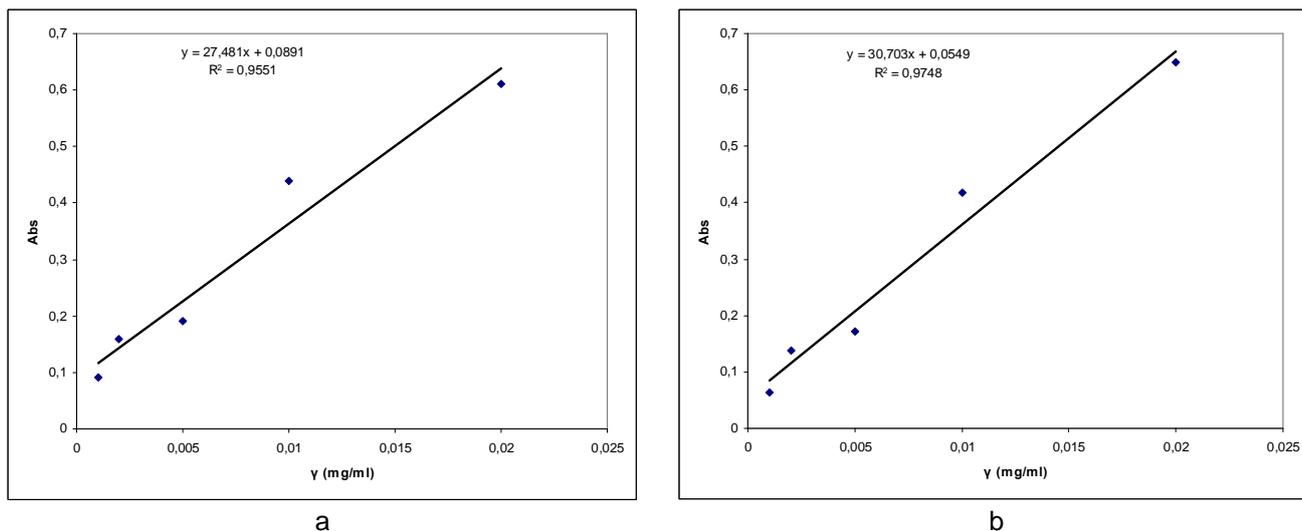


**Figure 6.** Spectral curves of stock solutions

Recorded spectrograms of stock solutions were decomposed by absorbance values and related concentration at two specific wavelengths (490 and 520 nm) necessary for calibration curve, Figure 7a and 7b.

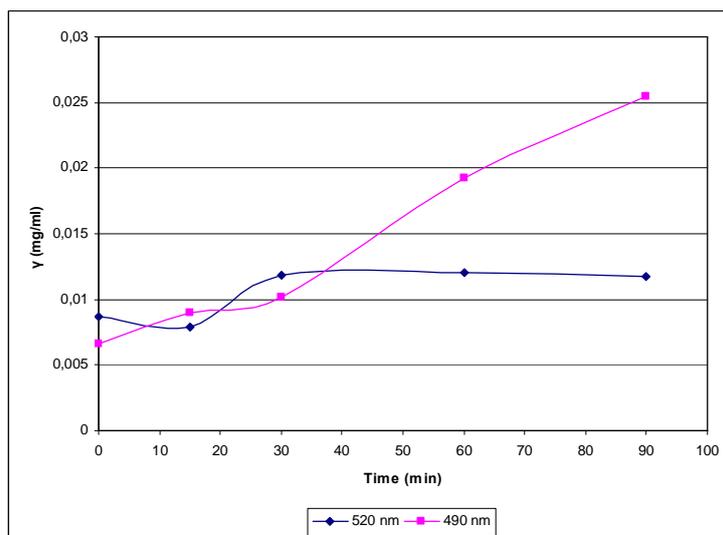
Calibration curves proved a high statistical reliability of absorbance versus concentration of stock reference ( $\alpha$ -tocopherol) at 490 nm (Fig 7a) and 520 nm (Fig 7 b). It was a platform for further work on desorption of  $\alpha$ -tocopherol from cosmetotextile (treated cotton fabric with NouWell E) [9].

Cosmetic cotton fabric was soaked in distilled water and stirred 90 min. Aliquots of that solution were taken at start and after 15, 30, 60 and 90 min.



**Figure 7.** Calibration curves of ( $\alpha$ -tocopherol - stock reference solution): a. 490 nm; b. 520 nm

Analytical protocol included a solution of  $\alpha$ -tocopherol (desorbed NouWell E from cotton fabric), solutions A and B, necessary for generating of metal organic chelate complex. Red coloured complexes were recorded by spectrophotometry wherein values of absorbance measured at 490 nm and 520 nm were related to a calibration curves, Figure 7. Identified concentration of desorbed  $\alpha$ -tocopherol versus time is shown in Figure 8.



**Figure 8.** The rate of  $\alpha$ -tocopherol desorption from cotton fabric

Figure 8 indicates that  $\alpha$ -tocopherol was desorbed from cotton fabric during the stirring phase. The amount of desorbed  $\alpha$ -tocopherol, in a metal organic chelate complex ( $\alpha$ -tocopherol - NouWell E) recorded at 490 nm and 520 nm is different. The equilibria of  $\alpha$ -tocopherol - NouWell E desorbed from cotton fabrics recorded in metal organic chelate complex at 490 nm is not achieved. The second characteristic peak of metal organic chelate complex at 520 nm resulted in equilibria amount after 30 minutes.

#### 4. Conclusion

This paper compares qualitative method with optimized quantitative method for UV/VIS spectroscopic identification of  $\alpha$ -tocopherol. Forming mechanism and sensitivity of a red coloured metal organic chelate complex should be further clarified. Peak at 520 nm is more reliable and therefore recommended for the quantitative determination. Taking into the consideration heterogeneity of the textile material there is a need for further research while preliminary results of spectral determination of  $\alpha$ -tocopherol on cosmetotextiles gave a good guidance for further progress on this issue.

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