

## THE CONNECTION BETWEEN TITANIUM CONTENT, BLEACHING AND FR PROTECTION OF WOOL

LJ. SINADINOVSKA<sup>A</sup>, S. ILIEVSKA<sup>A</sup>, I. SPIREVSKA<sup>B</sup>, J. SINADINOVSKI<sup>A</sup>

<sup>a</sup>Faculty of Technology and Metallurgy, <sup>b</sup>Institute of Chemistry, Faculty of Science, University "St. Cyril and Methodius", 1000 Skopje, Republic of Macedonia

### Abstract

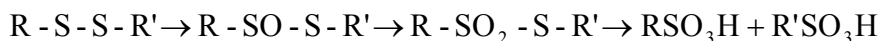
Potassium hexafluorotitanate was used for FR protection of wool (fibre and textile) in different conditions of treatment. Bleaching process was provided using of hydrogen peroxide and optical brightener bleaching system. The degree of FR protection was determined through the quantity of connected titanium in the wool. A modified procedure for the titanium determination in the wool using UV-vis spectrophotometry was developed. The connection between titanium content, bleaching and FR protection was established too.

**Key words:** wool, titanium determination, UV-Vis spectrophotometry, potassium hexafluorotitanat, FR protection, bleaching

### 1. Introduction

Wool is a low flammable material (LOI 25.2). Her wide use in the high risk environments needs FR treatment for better protection [1, 2, 3]. The particular challenge of bleaching wool involves the removal of dirt, vegetation, suint and other impurities [4]. In our earlier investigations potassium hexafluorotitanate was used as an effective durable finish [5, 6, 7]. Our interest in the earlier work was to find out the optimal conditions of FR protection of the home wool and with good qualitative characteristics for the further use [8]. The aim in the presented work is to find out the connection between bleaching (which is used very often for the wool fabrics) and the FR protection [9,10].

The very interesting problem for us was the find out what happened in the wool fibre really, using bleaching and FR treatment after that, i.e. whether the structure changes (breaking of S-S bonds during the bleaching process) have some influence to the connection of potassium hexafluorotitanate which we used. For that propose we prepared two types of samples: FR treated, only and bleached and FR treated after that, but in the same way as for the unbleached ones. It is known that cystine oxidation by hydrogen peroxide bleaching, resulting in cystine disulphide cleavage, and complete conversion to cysteic acid [11].



During bleaching the selective alteration of S-S linkage can be controlled by the appropriate choice of bleaching system. The presence of optical brightener contributed to increased whiteness [12].

The presented work uses UV-vis spectrophotometric determination of titanium, as an indirect way for the enabled FR protection, and through the quantity of connected titanium, the dependence between bleaching and FR protection is analysed.

## 2. Experimental

### *Samples*

Home wool and woolen textile.

### *Reagents:*

- $K_2TiF_6$  (Fluka Chemie AG, Switzerland, p.a.)
- $CH_3COOH$  (Alkaloid, Skopje, Macedonia, p.a.)
- Sulphuric acid (Merch, Germany, p.a.)
- $NH_4OH$  (Alkaloid, Skopje, Macedonia, p.a.)
- $H_2O_2$  (Alkaloid, Skopje, Macedonia, p.a.)
- Blankit IAN (Ciba Geigi, Switzerland)
- Rucogen RSK (Ciba Geigi, Switzerland)

### *Apparatus*

A Hewlett Packard 8452A Diode Array Spectrophotometer connected with a personal computer (commercial software for direct registration of data) was used. The measurements were made with quartz civette of 0.5 mm. Wavelength range 190 to 820 nm. All experiments were performed at room temperature.

### *Procedures*

#### *I. FR treatment*

Samples of wool and woolen textile were treated with 6%  $K_2TiF_6$  solution as a FR substance. Modulus of the treatment's bath was 1:20.

The variations of the other working parameters were made:

- \* pH = 2, 3, 4
- \* time of treatment: 30 min and 60 min for every pH value
- \* temperature of all these conditions was 323K

After FR treatment the samples were washed with distilled water at 323K during 30 min. Modulus of the bath was 1:40. The washed were dried 24 hours at room temperature.

#### *II. Bleaching process*

Samples of wool and woolen textile (20X20) were bleached with modulus of the bath 1:30, during 3 hours with  $10 \text{ cm}^3/\text{dm}^3$   $H_2O_2$  (30%). The pH wool was 8.5 (with  $NH_4OH$ ).

So bleached samples were treated with optical brightener further on using  $0.5 \text{ g}/\text{dm}^3$  Rucogen RSK, 7% Blankit IAN, during 3 hours at 323K temperature.

The bleached samples were treated for the FR protection using the procedure I.

So, FR treated dried samples (2g) were ignited in a covered platinum dish slowly and burned at 1103K during 3 hours. The ash was dissolved in conc. sulphuric acid. The solution was evaporated 3 times with  $H_2SO_4$  (1:2) to the  $SO_3$  fumes and diluted to  $100 \text{ cm}^3$  with distilled water in a volumetric flask. The final concentration of sulphuric acid in the solution was adjusted to  $1 \text{ mol}/\text{dm}^3$ .

A modification of the procedure was made [13].

$2 \text{ cm}^3$  of each probe was taken, added  $3 \text{ cm}^3$   $H_2O_2$  (3%) diluted to  $25 \text{ cm}^3$  in a volumetric flask and transferred in a 0.5 mm cell for the instrumental analyse.

Determination of titanium was made using calibration curve which was construed applying the prepared standard solution of  $K_2TiF_6$  with  $\gamma=0.5 \text{ mg}/\text{cm}^3$ .

### 3. Results and discussion

The quantity of titanium in the FR treated and in the bleached and FR treated wool samples was determined using UV-vis spectrophotometer, at 406 nm wavelength. All measurements of the yellow peroxide complex of titanium in the probes were determined after 2 min. A linear dependence between the concentration of Ti and absorbance was established. The next equation for the calibration curve was obtained:

$$A = 0.674C - 0.011 \quad R = 0.997$$

The relative standard deviation was 2.3% and the detection limit  $1.3 \mu\text{g}/\text{dm}^3$ .

From our earlier investigations, the obtained analyses showed that the use of 6%  $\text{K}_2\text{TiF}_6$  in the treatment's bath enabled a very good FR protection for the samples and good qualitative characteristics (alkaline solubility, dimensions stability etc.) which is important for the further use.

The aim of this work was to find out the connection between bleaching and FR procedure which are used for finishing of wool fabrics. The bonded titanium is a direct correspondence with the degree enabled FR protection. That's why we use the determination of the quantity of Ti in the FR treated and bleached and FR treated samples only as a measure trying to find out the connection between bleaching and the possibility of Ti salt penetration in the fibre. The results obtained from our investigations are given in the table 1 and 2.

**Table 1.** Results from determination of Ti ( $\mu\text{g}/\text{cm}^3$ ) in the FR treated wool, w ( $\text{K}_2\text{TiF}_6$ ) = 6%

pH	2		3		4	
time (min)	30	60	30	60	30	60
Ti	134.4	148.8	101.7	115.2	60.7	79.2

**Table 2.** Results from determination of Ti ( $\mu\text{g}/\text{cm}^3$ ) in the bleached and FR treated wool, w ( $\text{K}_2\text{TiF}_6$ ) = 6%

pH	2		3		4	
time (min)	30	60	30	60	30	60
Ti	118.7	137.0	86.9	108.3	61.2	80.7

As it can be seen, there is a difference in the quantity of connected titanium in the both types of samples. The pH value of FR solution in the bath and the time of treatment have influence for the enable protection. The lower quantity of titanium in the bleached and FR treated samples for the same conditions of FR treatment is an answer of our question what really happened in the wool, i.e. that the acceptance of the FR substance  $\text{K}_2\text{TiF}_6$  is happened to the S-S bond of the wool fibre, partly too.

The cystine oxidation by hydrogen peroxide bleaching, resulting in cystine disulphide cleavage. With our appropriate choice of bleaching system the selective alteration of S-S

linkage could be controlled. That is why we used a combination of bleaching system (hydrogen peroxide and optical brightener Blankit IAN) which contributed to increase whiteness. Our examinations showed that this way treated samples have some changes in the properties, but they still retain good qualitative characteristics. For that purposes alkaly solubility, strength of breaking, stretching, dimension stability were determined too. The degree of FR protection was controlled using vertical test of burning too.

#### 4. Conclusions

Spectrophotometric method is convenient for Ti determination. The quantity of connected Ti in the samples is a confirmation for the provided good FR protection. The appropriate used bleaching system enabled good degree of whiteness. The bleached and unbleached FR samples showed good qualitative characteristics.

This research is a contribution for the explanation of the connection between Ti content, bleaching and FR protection of the wool fibre (S-S bond in the wool fibre is a very possible place where Ti salt is connected).

#### References

- [1] Proposed Flammability Standard for Mattresses (DOL, PFF, 4-7) Feredeal Register No 175, 18096 (1971) 36
- [2] Motor Vehicle Safety Standard, Federal Register No. 5, 290 (1971) 36
- [3] G.C. Tesero, C.H. Meiser, Tex. Res. J., 40 (1970) 430
- [4] W. von Bergen, The Wool Handbook, vol.1, Interscience Publishers, NY, (1963), 143
- [5] M. Arifoglu, W.N. Marmer, C.M. Carr, Tex. Res. J., 60 (1990) 319
- [6] P.G. Gordon, D. T. Mc Mahon, W. Stephans, Tex. Res. J., 10 (1977) 699
- [7] S. Ilievska, Lj. Sinadinovska, I. Spirevska, Tekstilna industrija, 5-6 (1991) 19
- [8] Lj. Sinadinovska, I. Spirevska, S. Ilievska, T. Stafilov, Anal. Lab. vol. 7, 3 (1988), 134
- [9] W.S. Simpson, Proceed.of the 8<sup>th</sup> Inter.Woll Tex. Res. Conf., New Zeland, 4 (1990) 279
- [10] H.F. Launer, Tex. Res. J., Vol. 35, 9 (1965) 813
- [11] A. Maza, A.M. Manich, J.L. Parra, M.D. Castellan, Tex. Res. J., Vol. 62, 3 (1992) 162
- [12] R.A. Duffield, IWS Techn. Inf. Bull, Inter. Wool Secretatirat, Ilkley, England (1986)
- [13] E.B. Sandell, Colorimetric Determination of Traces of Metals, Int.Sci. Publ., NY (1950)