The Use of Pekoflam DPN for Fireproofing Cotton Textile Materials

CATALIN GHERASIMESCU[1*], ION SANDU[2*], ADELINE-CAMELIA CIOCAN[3], IOAN GABRIEL SANDU[4,5], MIHAIL LEVA[1], RAZVAN BERARIU[1]

[1] "Gheorghe Asachi" Technical University of Iaşi, Faculty of Materials Science and Engineering, 53 D. Mangeron Blv., 700050, Iaşi, România

[2] "Al. I. Cuza" University, Iaşi, Arheoinvest Platform, Scientific Investigation Laboratory, 11 Carol I Blv., 700506, Iaşi, Romania

[3] "Al. I. Cuza" University of Iaşi, Faculty of Physics, 11 Carol I Blv., 700506, Iasi, Romania

[4] "Gheorghe Asachi" Technical University of Iaşi, Faculty of Materials Science and Engineering, 50 D. Mangeron Blv., 700050, Iaşi, Romania

[5] Romania Inventors Forum, 3 Sf. Petru Movila Str., 700089, Iasi, Romania

The current study presents how the fireproofing substance Pekoflam DPN operates when treating the 100 % cotton fabrics and also its effect (permanent, semi-permanent, non-permanent) when it is used alone or followed by a creaseproof treatement with the Rucon FAN product. For this study we used the following methods of analysis,: Fourier Transform IR Reflectance Spectroscopy coupled with a microscope (micro-FT-IR) and Scanning Electron Microscopy (SEM) coupled with Electronic Dispersive X-ray Spectroscopy (EDX). The obtained data show that the fireproofing effect is semi-permanent for cotton, not suitable for products that require repeated washing, and the creaseproof treatment decreases the fireproofing effect.

Key words: fireproofing, cotton, textile materials, Pekoflam DPN, SEM-EDX, FTIR

Cellulosic textile materials are easy flammable materials. Therefore, for a wide range of consumer textile products, a fireproofing treatment is required in order to delay their self-ignition or to prevent burning and the maintenance of burning [1,2].

For that purpose different groups of chemicals, that act to delay burning can be used by releasing fireproof gases or by forming a glassy coating that prevents the access of oxygen to the flame. There are also fireproofing substances that have a more complex action, by intervening either in the mechanism of thermal decomposition of cellulose, or by absorbing combustion energy [1,3,4].

This paper aims to establish the action mechanism of the Pekoflam DPN fireproofing substance applied to cellulosic textile materials based on 100% cotton, as well as the evaluation of the effect (permanent, semi-permanent, impermanent) when used as such and followed by the creaseproof treatment with the Rucon FAN commercial product, in order to efficiently implement it on cellulosic textile articles compatible with that type of treatment.

Experimental part

For this study we chose the Pekoflam DPN commercial product, from the group of inorganic products of the cellulosic textile materials. It is used for cellulosic textile materials as such, as well as in combination with PAN fiber [2].

In order to determine how this fireproofing product acts, the textile material was analyzed, before and after treatment, by Scanning Electronic Microscopy (SEM), coupled with Energy-Dispersive X-ray Spectroscopy (EDX) and with Fourier Transform IR Spectroscopy coupled with a microscope, in reflection mode (micro-FTIR) [1].

The SEM electronic microscope from VEGA-TESCAN, coupled with EDX (detecting device from Bruker) operated at 30 KV, obtaining b.s.e images (backscatter electrons) and elemental composition, for which a mapping of the atoms in the analyzed surface has been carried out [5,6].

The micro-FT-IR spectrum was obtained with a Tensor 27 spectrometer, coupled with a Hyperion microscope 1000 (150 x) from Bruker, Germany [7]. The OPUS / VIDEO included software was used to obtain the spectrum. Spectral measurements were made in reflection mode[7].

The measuring range was 4000-600cm^{-1}, the resolution being 4 cm^{-1}.

The FTIR spectra in transmission were recorded with a Digilab/Excalibru FTS 2000 spectro-photometer for the 400-4000 cm^{-1} range, with a resolution of 4 cm^{-1}, by dispersing the sample in the KBr pill [1].

The cellulosic textile material as a 100% cotton fabric, has been treated for fireproofing, by using an appropriate working procedure and recipe. Thus, the treatments were conducted using the following baths for treatment:

- Fireproof substance(Pekoflam DPN).................300g/L;
- Fixing agent (Herasim dm 70)...............................14g/L;
- Softening agent (Datasoft CSF)...............................8g/L;
- Orthophosphoric acid 85% 18g/L;
- Wetting agent (Tececschneller)................................3g/L.

The technological flow used in the process of fireproofing consists of the next stages: impregnation, spinning, drying, heat treatment, washing and drying.

After impregnation, the samples of cotton-based textiles were spun by the roller squeezer device, then the 80% drying, was done in an oven, at 110°C, followed by a heat treatment at 145°C, for 4 min [8]. The washing was done in several steps, first with a calcinated soda solution 10-20 g/L, at 50°C, then with warm water 50-60°C, followed by rinsing.

Results and discussions

Figures 1-3 present the cross-section microphotographs made by the Scanning Electron Microscope, where the continuity of the fireproof layer can be noticed both on the surface and inside the fiber.

By comparing the microscopic image of the untreated fiber section with the recorded image at 3000X for the

* ion.sandu@uaic.ro; Tel. +40.232.201662

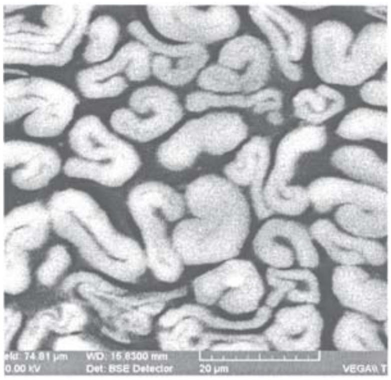

Fig. 1. The SEM image of the untreated fiber section (3000X)

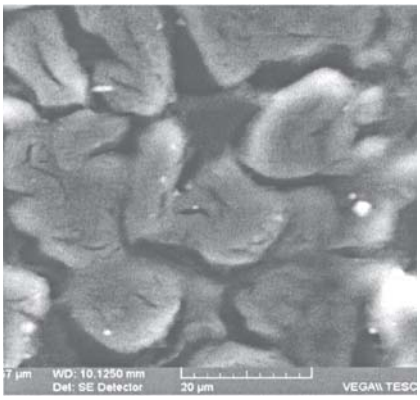

Fig. 2. The SEM image of a fiber section treated with Pekoflam DPN (3000X)

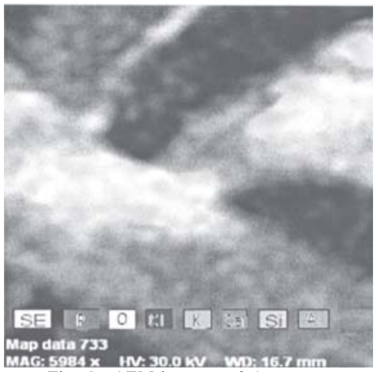

Fig. 3. SEM image of the atom distribution in the fireproofing substance (Pekoflam DPN) on the surface and inside the fiber (5984X)

treated samples at fireproofing resolution, , we noticed a swelling of the fiber, with a slight modification of the lumen shape, the fireproofing substance penetrating inside it. (fig.2).

This can be more easily seen, by analyzing the microscopic image of the section with the atom charting, made at a magnifying range much higher than 5984X (fig.3).

In this image we can see quite clearly that the fireproofing substance is distributed equally and in a large amount inside the fibers and unequally and in a smaller amount between them.

Figure 4 depicts the EDX spectrum for the fiber treated with Pekoflam DPN.

Table 1 shows the elementary chemical analysis evaluated from the EDX spectra, for the fibers treated with Pekoflam DPN.

These data confirm the fact that the analyzed fireproofing substance contains elements with fireproofing properties, such as: phosphorus, chlorine, silicone and aluminum, together with compensatory cations of alkaline and alkaline earth metals, among which we mention the phosphates, the silicates and the alums, with proven fireproofing properties. At high temperatures, these substances form a glassy layer, which blocks the access of oxygen to the cellulosic material, holding up ignition and burning.

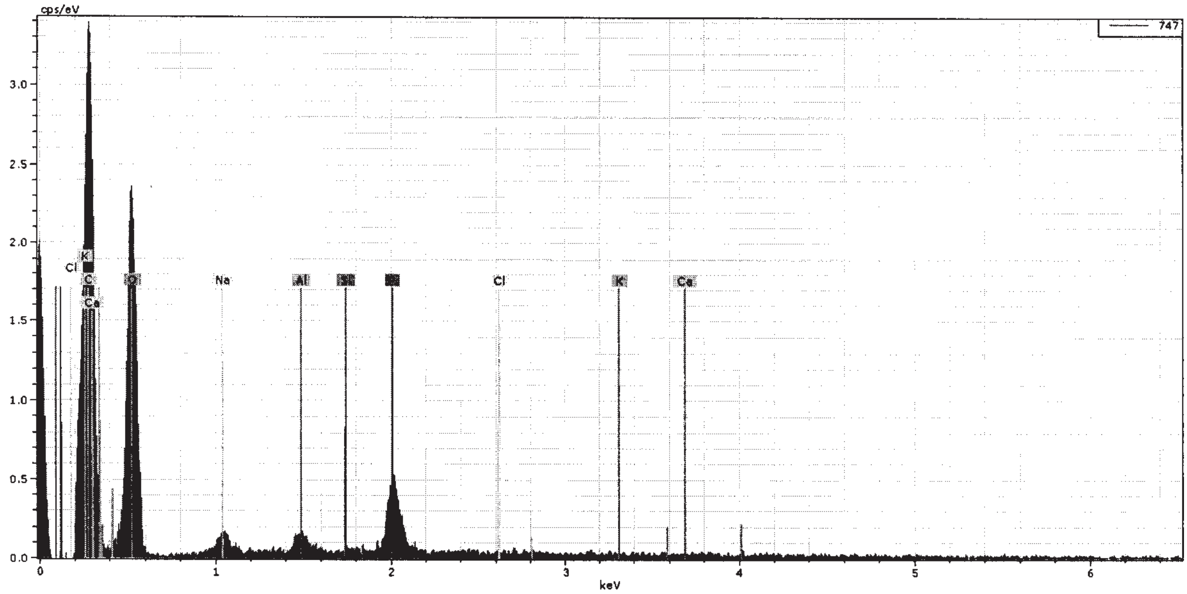

Fig. 4. The EDX spectrum of cotton fibers treated with Pekoflam DPN

The chemical element	[norm. wt. -%] (mass percentage)	[norm. at. -%] (atomic percentage)
Carbon	11.47	15.477
Phosphorus	4.0133	2.1
Sodium	5.3641	3.7817
Aluminum	2.0593	1.237
Chlorine	below 1	below 1
Potassium	below 1	below 1
Calcium	below 1	below 1
Silicon	below 1	below 1
Oxygen	75.785	76.772

Table 1
DATA CONCERNING THE ELEMENTARY CHEMICAL ANALYSIS OF THE FIREPROOFING SUBSTANCE PEKOFLAM DPN

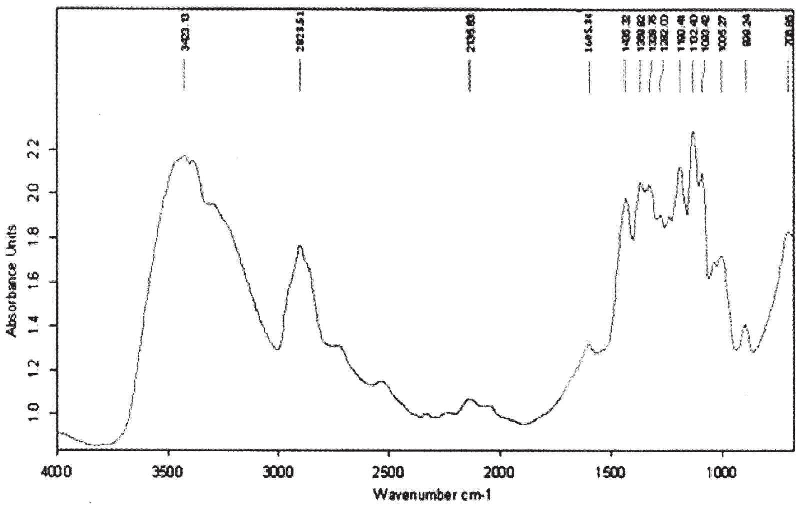

Fig.5. Micro-FT-IR spectrum in reflection, untreated cotton

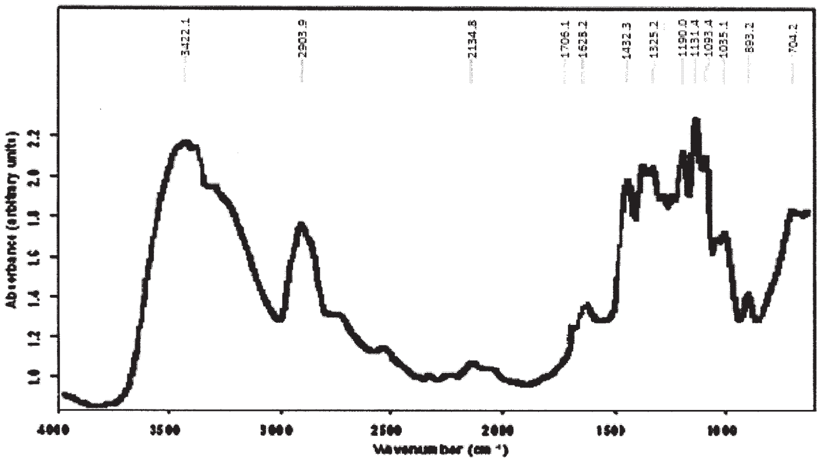

Fig. 6. Micro - FT-IR spectra in reflection of the Pekoflam DPN treated material

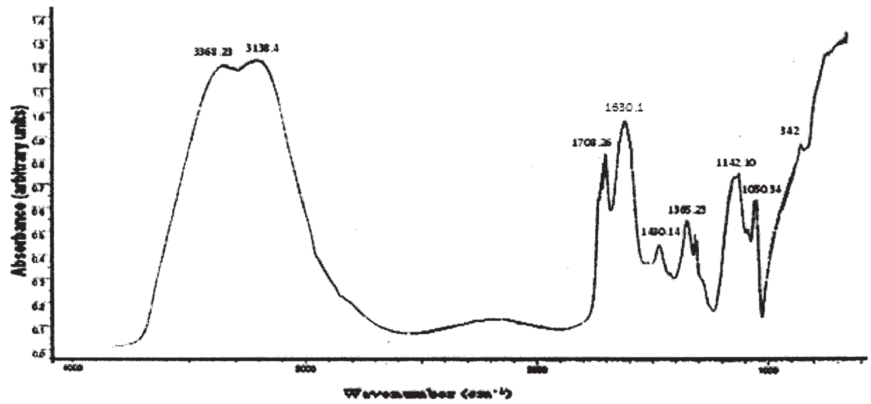

Fig. 7. The IR spectrum of the Pekoflam DPN fireproofing substance

Additional data concerning the action mechanism of the analyzed substance were also obtained through some comparative studies using micro-FTIR spectroscopy in reflection, for untreated cellulose fiber (fig. 5), for the Pekoflam DPN treated cellulose fiber (fig. 6) and for the FTIR spectrum of the individual fireproofing agent (fig. 7).

Relevant publications mention a series of studies on the application of the FTIR spectoscopy in diffuse reflection, on textile materials, in general for qualitative measurements but also for the quantitative ones [9 - 15].

Returning to the micro-FTIR spectra, in the spectrum of the fiber treated with Pekoflam DPN (fig. 6) there are two ranges at the wavelength of 1706 cm^{-1} and 1628.2 cm^{-1}, which are characteristic to the Pekoflam DPN product [16].

Those aspects lead to the conclusion that the fireproof agents analyzed do not link chemically to the cellulose fiber on which they were applied, but only by physical interactions of the Van der Waals type, or by hydrogen brackets [16]. That is why the fireproofing effect of this product is semi-permanent, an issue that will be evaluated further on.

In order to establish the effect of this fireproofing agent on the 100% cotton textile material, a number of treatments were applied, such as creaseproof finishing, the improvement of the handle and washing.

After making those treatments, samples of treated textile material were tested in compliance with STAS 6941/03 and we found that the time of burning in this case was much longer than in the case of materials treated for simple fireproofing, which led to the conclusion that the Pekoflam DPN fireproofing substance, has not a permanent effect on the cellulose textile materials, as mentioned in the technical data card of the product, but, at best, a semi-permanent effect.

To establish the cause which leads to the decrease in fire resistance as a result of the application of finishing treatments to materials treated with Pekoflam DPN, the samples treated for fireproofing and then for creaseproofing were subject to physico-chemical analyses.

The chemical element	[norm. wt. -%] (mass percentage)	[norm. at. -%] (atomic percentage)
Carbon	13.47	17.8993
Phosphorus	3.7189	1.9162
Sodium	3.4217	2.3754
Aluminum	0.8925	below 1
Chlorine	0.4550	below 1
Potassium	0.2829	below 1
Calcium	0.5019	below 1
Silicon	0.5386	below 1
Oxygen	76.5824	76.3937

Table 2
THE DATA REGARDING THE ELEMENTARY CHEMICAL ANALYSIS OF THE SYSTEM FIREPROOF AGENT-TEXTILE MATERIAL-SUPERFICIAL CREASE RESISTANT FINISH

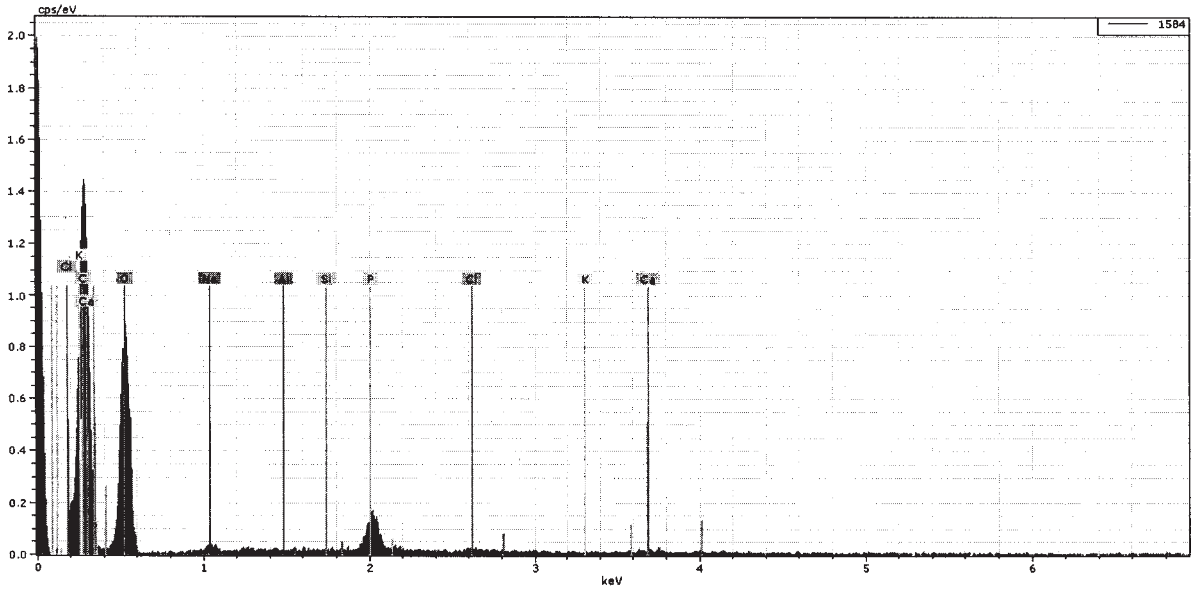

Fig. 8. EDX data for the Pekoflam DPN substance

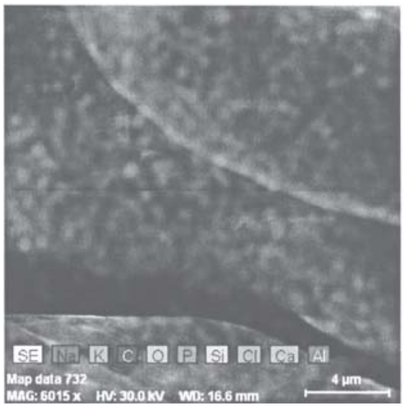

Fig. 9. The SEM image showing the distribution of the elements of the fireproofing substance (Pekoflam DPN) and of the creaseproofing substance (Rucon FAN) on the surface and inside the cellulosic fibers (6015X)

The stability of fixation on the fireproofing agent on the textile material was verified by determining the elementary composition (table 2) based on the EDX spectrum of the fireproofing agent integrated on the 100% cotton textile material which had also been treated for creaseproof, as presented in the figure 8.

The chemical composition shown in table 2 was confirmed by the SEM electronic microscopy images (see fig. 9 and 3), where the distribution of the components of the fireproofing substance and of the Rucon FAN product (used for crease resistant finish purposes) can be seen.

By comparing the percentage of the fireproofing elements , shown in table 1 (fireproofed textile), with the one in Table 2 (fireproofed and creaseproofed textile), we noted that the percentage of these elements is different, as in the case of the fireproofed and creaseproofed textiles the fireproof properties thereof were much lower. This fact explains the decrease in fire resistance of the fireproofed textiles, to which the creaseproofing treatment was applied, by using the Rucon FAN product.

Moreover, the distribution of the elements in the fireproofing substance used for the fireproofed and creaseproofed textile, shown in figure 9, is also slightly different from the one of the textile which was only fireproofed, shown in figure 3.

From the elementary analysis based on the EDX specra of the two subsystems: fireproofed fibers and fireproofed and creaseproofed fibers, we could not determine any marker element for the differentiation, but a simply reduction of composition as a result of the creaseproofing process, made after prior washing , and processing, which cause a decrease of the concentration of specific fireproofing elements especially between fibers and on their surface.

Conclusions

Based on SEM-EDX and micro-FTIR data , we studied the effects of fireproofing processes with Pekoflam DPN and of creaseproofing with Rucon FAN on 100% cotton cellulose fibers.

We found that the fireproofing agent Pekoflam DPN does not bind to the fiber chemically, but by Van der Walls interactions and by hydrogen brackets, and that it is distributed both on the surface and inside the fiber.

We established that the fireproofing substance Pekoflam DPN has a semi-permanent effect on 100% cotton textile fibers and not a permanent one. Moreover, fireproofing with Rucon FAN, with which it is compatible, decreases fireproof qualities.

References

1. GHERASIMESCU, C., BUTNARU, R., SANDU, I., CIOCAN, A.C., SANDU A.V., Rev. Chim.(Bucharest), **61**, no. 8 , 2010, p. 728.

2. WEIL, E.D., LEVCHIL, S.V., Flame Retardants in Commercial Use or Development for Textiles, Journal of Fire Sciences, **26**, 2009, p 243.

3. KAMATH, M.G., BHAT, G.S., PARIKH, D.V., CONDON, B.D., Processing and Characterization of Flame Retardant Cotton Blend Nonwovens for Soft Furnishings to Meet Federal Flammability Standards, Journal of Industrial Textiles, 38, 2009, p 251

4.WU, W.,YANG,C., Comparison of different reactive organophosphorus flame retardant agents for cotton. II. Fabric flame reistant performance and physical properties, Polymer, Degradation & Stability, **92**, 2007, p 363

5. BODIRLAU, R., SPIRIDON, I., TEACA, C., Rev. Chim. (Bucharest), **60**, , no. 5, 2009, p 508

6. CLAUDIA MORGOVAN, ELEONORA MARIAN, AUREL IOVI, IOAN BRATU, GHEORGHE BORODI, Rev. Chim. (Bucharest), **60**, no.12, 2009, p 1282.

7. RUFFOLO, S., BARONE, G., The Use of FTIR and Micro-FTIR Spectroscopy: An Example of Application to Cultural Heritage, International Journal of Spectroscopy, **10**, 2009, p 1155

8. BUTNARU, R., STOICHITESCU, L., Procedee special de finisare a materialelor textile, Ed. '' Gh. Asachi'' Iaşi, 1995, p 170

9. HEISE, H.M., KUCKUKA, R., BERECK, A., RIEGEL ,D., Mid-infrared diffuse reflectance spectroscopy of textiles containing finishing auxiliaries, Vibrational Spectroscopy, Vol. 35, Issues 1-2, 17 June 2004, p. 213-218

10. SAUPERL, O., STANA-KLEINSCHEK, K., VONCINA, B., SFILIGOJ-SMOLE, M., ETLINGER, B., Influence of the mercerization on the croslinking of the celullose fibers with BTCA, Tekstil, **51**, (10), 2002, p. 455.

11. SANDU, I.C.A., LUCA, C., SANDU, I., POHONTU, M., Rev. Chim. (Bucharest), **52**, no.7-8, 2001, p. 409.

12. SANDU, I., VRINCEANU, N., COMAN, D., Rev. Chim. (Bucharest), **60**, no. 9, 2009, p. 944.

13. SANDU, I.C.A., DE SA, M.H., PEREIRA, M.C., Ancient 'gilded' art objects from European cultural heritage: a review on different scales of characterization, SURFACE AND INTERFACE ANALYSIS, 43, 8 Special Issue: SI, 2011, p. 1134.

14. SANDU, I.C.A., BRACCI, S., SANDU, I., LOBEFARO, M., Integrated Analytical Study for the Authentication of Five Russian Icons (XVI-XVII centuries), Microscopy Research And Technique, 72, (10), 2009, p. 755.

15. TURA, V., TOFOLEANU, F., MANGALAGM, I., MINDRU, T.B., BRINZA, F., SULITANU, N., SANDU, I., RAILEANU, I.D., IONESCU, C., Electrospinning of gelatin/chitin composite nanofibers, JOURNAL OF OPTOELECTRONICS AND ADVANCED MATERIALS, 10, (12), 2008, p. 3505.

16. BODIRLAU, R., SPIRIDON, I., TEACA, C., Rev. Chim. (Bucharest), **60**, no. 5, 2009, p 508

Manuscript received: 14.07.2011