# Gamma Irradiation Effects on Natural Dyeing Performances of Wool Fabrics

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*The objective of this study was to improve the performance of natural dyeing on fabrics made of 100% wool by gamma pre-irradiation technique. For this purpose, the fabrics were subjected to irradiation with different doses in the range of 5 kGy - 40 kGy using a gamma irradiator Co-60 and then subjected to natural dyeing by discountinous exhaustion processes. Due to the lack of colour reproducibility in natural dyeing a natural commercial dye has been used - Itodye Nat Pomegranate. The mordanting of pre-irradiated and naturally dyed fabrics has been performed using iron alum. To establish the effectiveness of gamma pre-irradiation treatment on natural dyeing measurements for colour and color fastness to washing, light, wet and dry rubbing, acid and alkaline perspiration were performed. The results obtained were analyzed in comparison with those recorded for non-irradiated and dyed fabrics. The investigation of the effect of gamma radiation on textile materials dyed was carried out by assessing the physico-chemical, physico-mechanical and surface characteristics of the dyed textile materials. Infrared spectroscopy was used to monitor chemical and structural changes in proteinic fibers induced by gamma irradiation and dyeing process. Surface modifications of wool fibers, as an effect of gamma radiation treatment and natural dyeing processes were studied by electron microscopy.*

*Key words: gamma irradiation, natural dyeing, wool fabrics*

The current concerns for the environment as well as the norms and regulations in force have led to a growing demand for natural products in various industrial and industry-related branches. The market registers an increased demand for natural products, even in the textile field. The revival of interest towards the use of natural dyestuffs in the textile industry is attributed to the increased awareness about environmental contamination created by intermediates and chemicals being used in synthetic dyeing processes.

Natural dyes are biodegradable, compatible with the environment, they are not noxious and do not induce allergic reactions. Nevertheless, dyeing with this class of dyes presents some inconveniences such as the lack of stability and of colour fastness. Commercialization of natural dyes can be done successfully by a systematic and scientific approach to extraction, purification and use. Optimization of extraction condition is a must to minimize the investment cost and to avoid discrepancy in the dye shade quality. Extraction may include drying, pounding, soaking, crystallizing, condensing and liquidifying, among others, depending on the quality and species of the dye yielding plant [1].

A lot of techniques are being used to improve dyeing behaviour of natural dyes and modification of fabrics either by improving extraction process or by enhancing dye uptake of textile materials to get good shades and fastness properties [2]. These techniques may include colour isolation on fabrics [3], biopolishing, [4], bioscouring [5], cationization [6], chitosan treatment [7] enzyme treatment [8, 9], ultrasonic treatments [10], UV exposures [11, 12], microwave treatments [13], plasma treatments [14]. Several studies have clearly indicated that gamma rays treatment is very effective in improving dye extraction, colour strength and fastness properties of natural dyestuffs [15, 16].

Irradiation processes have several commercial applications; the advantages of this technology are well known energy saving being a low-temperature process, low environmental impact, simple, economical and high treatment speed. Despite of these advantages, there have been few applications of radiations in the textile industry, such as fabric coating, fabrics grafting pigment printing, effective methods of sterilization of textile dressings and others medical textiles, disinfection of various types of historical objects [17-21]. Also, radiation treatments of textile materials can add value in colouration. Modification of the surface fibers can allow more dye uptake, producing deeper shades, dyeing at low temperature and increase wettability. However, one should bear in mind, that ionizing radiation has a destructive effect on many polymeric materials and may lead to deterioration in their mechanical, chemical and structural properties [22, 23].

Due to their natural heterogeneity, wool fibers are complex materials. The conformational strength and rigidity of wool keratin are confered by the molecular architecture: partly crystalline, alpha-helical filaments embedded in an amorphous matrix [24]. Wool is a condensation product of

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a α-amino acids, and the presence of cysteine results in crosslinkages between the main protein chains wherever this amino acid occurs in the chain. After application of high-energy radiatoins, changes in mechanical, physico-chemical and structural properties of polymers can occurs. The direction of change in properties is governed by whether cross linking or cleavage is the predominating reaction. Cross linking increase the strength and modulus, but elongation decrease. Cleavage of long-chain molecules (degradation) produces decreases in strength, elongation and in modulus elasticity. Decreases in strength are indicative of a decrease in average molecular length or of chain scission while increases in modulus are regarded as evidence of cross linking [25]. The effect of high-energy radiation is not limited to cross linking or chain scission. Oxidation reactions that can profoundly influence fiber properties can also occur. Thus it is believed that the predominating effect of gamma irradiations on wool fibers when exposed in an atmosphere containing oxigen and water is one of oxidation of cysteine. Such oxidation causes the wool to become increasingly sensitive toward alkalies, and the solubility in alkalies can be used to detect the first traces of damages.

Keeping in view the beneficial effects of application of gamma radiations in natural dyeing, the objective of this study was to improve the performance of a natural commercial dye on 100% wool fabrics by gamma irradiation technique. We have conducted the study with the objective to enhance the colour strength of dyed wool fabrics through them surface modification using high energy radiations.

## Experimental part

*Materials and methods*

100% wool fabrics with 208 g/cm$^2$ have been used for the dyeing process. Dyeing has been performed using a natural commercial dye - Itodye Nat Pomegranate (LJ Specialities, UK), supplied as a stable solution. Post-mordanting operation has been performed using the conventional mordant – iron alum (Sigma Aldrich). Scouring process was carried out with a tensioactive product, a low foam non-ionic product based on polyethoxilated fatty alcohol Imerol JSF (Clariant).

*Preliminary treatments*

For the smooth running of subsequent natural dyeing, the wool fabrics have been treated according to the conventional scouring procedure. The 100% fabrics were scoured with a solution containing 2 g/L Imerol JSF, at a temperature of 40°C, for 30 min. After this operation the fabrics were subjected to repeated rinsing with warm water at 40°C and with cold water and afterwards dried freely at room temperature.

*Gamma irradiation*

100% wool fabrics were subjected to gamma irradiation with different doses in the range of 5 kGy - 40 kGy using a research Co60 irradiator GC-5000 (Gamma Chamber, BRIT, India). Irradiation has been performed in a cyclindrical geometry with a dose rate of minimum 4.9 kGy/h, the doses accumulated by fabrics for the irradiation with gamma radiations were 5 kGy, 10 kGy, 15 kGy, 25 kGy and 40 kGy. The irradiation doses were determined by the method of dosimetry with ethanol-chlorobenzene (ECB) (standard deviation of ± 2%).

*Dyeing*

Dyeing of 100% wool fabrics was carried out by discountinous exhaustion processes, at *p*H = 5 with 20% dye, MLR (material to liquor ratio) 1:35, at a temperature of 90°C for 60 minutes, followed by hot and cold rinsing and squeezing. After the dyeing operation, the fabrics were rinsed with hot and cold water, soaped with 1g/L Imerol JSF at a temperature of 80°C for 10 min, squeezed and dried freely at room temperature.

*Mordanting*

Mordanting was performed using the laboratory equipment Ugolini (Italy) with 3% iron alum, at MLR 1: 30, for 45 min, at a temperature of 80°C, followed by hot and cold rinsing, squeezing and drying at room temperature.

*Colour measurements*

To show the influence of gamma pre-irradiation on natural dyeing of textile materials made of protein fibers, spectrophotometric characterization of textile backings which were subjected to pre-irradiation and natural dyeing was performed by evaluating the parameters of color difference and color intensity. In this regard color measurements were made in accordance with standard ISO 105 J03:2001, using for the determination the spectrophotometer Spectroflash 650 (Datacolor, Switzerland), and the illuminant D65/10 as light source. The values obtained for the color difference parameters represent the average of 10 individual determinations carried out on dyed samples. The colour difference parameters were determined considering as reference for each type of textile backing the non-irradiated sample (code 0 kGy) which was dyed with natural dye.

To assess the colour intensity of pre-irradiated and dyed samples, K/S parameter was calculated at the dominant wavelength ($\lambda_{dominant}$), based on the formula derived from the theory of Kubelka–Munk:

$$K/S = (1-R)^2/2R, \qquad (1)$$

where:
K - is the coefficient of absorption;
R - reflectance at $\lambda_{dominant}$;
S - is the coefficient of scattering.

*Dyeing fastness*

The influence of gamma treatments on natural dyeing was determined by evaluating colour fastness to washing, light, acid and alkaline perspiration. Standard SR EN ISO 105-C 10:2010 was used to determine fastness to washing, in respect of colour change in the washing solution and colour staining on the multifibre standard, the assessment was made on the grey scale basis. Dyeing fastness to acid (*p*H = 5.5) and alkaline (*p*H=8) perspiration was evaluated on the basis of standard SR EN ISO 105-E 04:2013. Dyed samples were tested to artificial light, using Xenotest Apolo-James Heal device, according to standard SR EN ISO 105-B02:03 and the results were assessed based on the blue scale. Color fastness to rubbing (wet and dry) of dyed wool fabrics were tested according with SR EN ISO 105-X12: 2003 standard.

*Determination of physico-mechanical and physico-chemical characteristics*

In order to evidence the influence of irradiation treatment on natural dyeing on the integrity of the analysed fabrics, the main physical-mechanical characteristics were determined in comparison with un-irradiated and dyed fabrics. Density (SR EN 1049-2:2000), breaking strength and breaking elongation (SR EN ISO 13934-1/2013) were used. The alkali solubility index of keratin fibres was determined by the solubilisation of yarns for 1h in a NaOH 0.1N solution, at a temperature of 65°C, after having first been scoured of fats by extraction in Soxhlet with oil ether and ethylic alcohol, in accordance with standard STAS 8398-69.

*Diffuse Reflectance Infrared Fourier Transform (DRIFT) Spectroscopy*

Infrared spectroscopy was used to monitor chemical and structural changes in wool fibers induced by gamma irradiation. For the above mentioned samples, Fourier transform infrared spectra were collected on a Vertex 70 model spectrometer (Bruker, Germany) equipped with a Helios micro DRIFT accessory, in the wavenumber range of 4,000–600 $cm^{-1}$, at a resolution of 4 $cm^{-1}$, with 64 consecutive scans.

*Electron microscopy*

The surface morphology of scaled cuticle layer of keratin fibres as an effect of gamma radiation was investigated by a FEI Quanta 200 Scanning Electron Microscope with a GSED detector, at a 2000X –magnification and accelerating voltage of 12.5 kV – 20 kV.

## Results and discussions

*Color measurements*

The use of colour measurement technique by determining the chromatic parameters enabled the determination of colour changes of pre-irradiated and naturally dyed samples, compared to those non-irradiated. Terms of colour difference were determined considering as a reference for each type of fabric, the sample dyed with natural dye without preliminary treatment with gamma rays and the values determined are given in Table 1. The values obtained for K/S are graphically represented in figure 1.

Analyzing the data presented in table 1 can be observed that gamma pre-irradiation of fabrics made of protein fibres positively influences the adsorption capacity of natural dye, effect materialized in getting darker (negative values of DL*) and more saturated shades when using irradiation with doses of 10 kGy, 15 kGy, 40 kGy. There was no evidence of dyeing affinity increase in the case of pre-irradiation with doses of 5 kGy and 25 kGy, respectively. The values of K/S parameter recorded at a maximum wavelength ($\lambda_{dominant}$ = 572 nm) for the 100% wool fabrics record a significant increase with increasing the dose, and when applying the maximum dose there was an increase of approximately 1.8% compared with the non-irradiated sample (fig. 1).

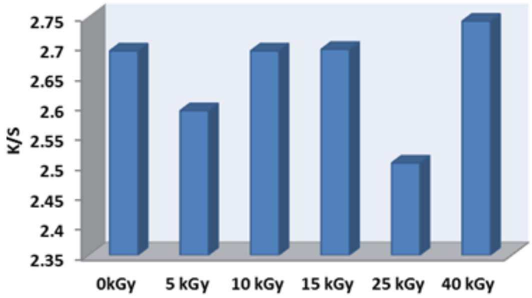


Fig. 1. K/S values obtained for 100% wool fabrics irradiated with different doses, naturally dyed and post-mordanted with iron alum

*Colour fastness*

The values of colour fastness made with ITODYE NAT POMEGRANATE for experimental variants on fabrics made of natural fibers are given in table 2. From the data presented it can be seen that, regardless of the applied dose for activating protein fibers, colour fastness to washing is generally weak and moderate, both in terms of change in the washing solution and colur bleeding on the multifibre standard. Colour fastness to acid or alkaline perspiration of pre-irradiated and dyed samples are moderated, and the grades are similar to those obtained for non-irradiated samples. In terms of colour fastness to dry friction the samples behave similarly, without significant differences between them; colour fastness to wet friction is differentiated, but pre-irradiation treatments did not induce an improvement thereof. Also, it can be seen that gamma pre-irradiation does not influence positively colour fastness to light, and the grades are similar to those obtained for the non-irradiated sample.

Mechanical and physico-chemical characteristics of irradiated 100% wool fabrics, naturally dyed and mordanted with iron alum are presented in table 3.

The preliminary gamma radiation process followed by natural dyeing and mordanting induces the percentage decrease of the tensile strength of 100% wool fabrics by maximum 11% in the warp direction and by 18% in the weft direction at 40 kGy (according with recalculated breaking strength N/100 yarns). The application of different doses of gamma radiation did not affect the elongation at break and the values obtained had an increasing trend compared to the untreated sample.

When wool fibers are subjected to gamma irradiations, the first noticeable changes in properties are them susceptibility to damage by alkalies [26]. Analyzing the data in table 3 it can be seen that alkali solubility values gradually increase with increasing the irradiation dose, the highest value being obtained in the case of irradiation with a dose of 25 kGy. These results suggest the structural

**Table 1**

COLOUR DIFFERENCE PARAMETERS OBTAINED FOR 100% WOOL FABRICS NATURALLY DYED

| Dose | Color difference parameters | | | | | | Observations |
|---|---|---|---|---|---|---|---|
| | DL* | Da* | Db* | DC* | DH* | DE | |
| 0 kGy | Reference | | | | | | - |
| 5 kGy | 0.71 | -0.14 | -1.27 | -1.28 | 0.09 | 1.46 | lighter, unsaturated, greener |
| 10 kGy | -0.30 | 0.71 | 2.09 | 2.13 | -0.57 | 2.22 | darker, more saturated, redder |
| 15 kGy | -0.16 | 0.30 | 0.30 | 0.32 | -0.29 | 0.46 | darker, more saturated, redder |
| 25 kGy | 1.21 | -0.31 | -0.91 | -0.92 | 0.28 | 1.54 | lighter, unsaturated, greener |
| 40 kGy | -0.36 | -0.16 | 0.17 | 0.16 | 0.17 | 0.43 | darker, more saturated, greener |

*Obs: Positive values of DL * indicate lighter samples compared to the reference; negative values of DL* indicate darker samples compared to the reference. Positive values of DC* indicate more saturated samples compared to the reference; negative values of DC* indicate nore unsaturated samples compared to the reference. DE* represents the total colour difference between the sample and the reference.*

**Table 2**
COLOR FASTNESS OBTAINED FOR 100% WOOL FABRICS IRRADIATED WITH DIFFERENT DOSES, NATURALLY DYED AND POST-MORDANTED WITH IRON ALUM

| Dose | Color fastness | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Washing | | | | Acidic perspiration | | | | Alkaline perspiration | | | | Light | Rubbing | |
| | Colour changing | Colour staining | | | Colour changing | Colour staining | | | Colour changing | Colour staining | | | 50 ore | Dry | Wet |
| | | BBC | PA | L | | BBC | PA | L | | BBC | PA | L | | | |
| 0 kGy | 3 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 3-4 | 4-5 | 4-5 | 4-5 | 4 | 2 | 3 |
| 5 kGy | 2-3 | 4-5 | 4-5 | 4-5 | 3-4 | 4-5 | 4-5 | 4-5 | 3 | 4-5 | 4-5 | 4-5 | 4 | 2 | 3-4 |
| 10 kGy | 2-3 | 4-5 | 4-5 | 4-5 | 3-4 | 4-5 | 4-5 | 4-5 | 3-4 | 4-5 | 4-5 | 4-5 | 4 | 2 | 3 |
| 15 kGy | 2-3 | 4-5 | 4-5 | 4-5 | 3-4 | 4-5 | 4-5 | 4-5 | 3 | 4-5 | 4-5 | 4-5 | 4 | 2 | 2-3 |
| 25 kGy | 2-3 | 4-5 | 4-5 | 4-5 | 3-4 | 4-5 | 4-5 | 4-5 | 3-4 | 4-5 | 4-5 | 4-5 | 4 | 2 | 3 |
| 40 kGy | 3 | 4-5 | 4-5 | 4-5 | 3-4 | 4-5 | 4-5 | 4-5 | 3-4 | 4-5 | 4-5 | 4-5 | 3-4 | 2 | 2-3 |

*Note: **Grey scale assessment**: grade 5: very good resistance; grade 4/4-5: good resistance; grade 3/3-4: moderate resistance; grade 2/2-3: weak resistance; grade 1/1-2: very weak resistance. **Blue scale assessment**: grade 8- exceptional resistance; grade 7-excellent resistance; grade 6-very good resistance; grade 5- good resistance; grade 4- acceptable resistance; grade 3- moderate resistance; grade 2- weak resistance; grade 2- very weak resistance.*

modification of the protein fibres as a result of destructive degradation caused by irradiation, modification which results in high alkali solubility values. Also, the dyeing and mordanting processes are factors contributing by increasing the humid-thermal operations of the process flow to the increase in the degradation degree of fabrics.

*Diffuse Reflectance Infrared Fourier Transform (DRIFT) Spectroscopy*

The micro-DRIFT spectra of the untreated and irradiated naturally dyed wool showed the characteristic bands of keratin at around (1692-1695) $cm^{-1}$ for amide I, 1558-1559 $cm^{-1}$ for amide II and around 1234 $cm^{-1}$ for amide III. Amide I absorption is associated with the C=O stretching vibrations, amide II vibration leads to the N–H bending and C–N stretching vibration. Both the Amide III vibration is

**Table 3**
MECHANICAL AND PHYSICO-CHEMICAL CHARACTERISTICS FOR 100% WOOL FABRICS, IRRADIATED, NATURALLY DYED AND MORDANTED WITH IRON ALUM

| Code | Mechanical and physical-chemical characteristics | | | | | | |
|---|---|---|---|---|---|---|---|
| | Density [No. of yarns /10cm] | | Breaking strength [N] | | Elongation at break [%] | | Alkali solubility |
| | Warp | Weft | Warp | Weft | Warp | Weft | |
| Untreated | 209 | 162 | 376 | 292 | 50.1 | 30.50 | 15.42 |
| 0 kGy | 230 | 177 | 388 | 314 | 64.6 | 43.00 | 16.30 |
| 5 kGy | 223 | 183 | 371 | 330 | 61.5 | 44.50 | 17.19 |
| 10 kGy | 227 | 180 | 365 | 312 | 59.7 | 44.30 | 18.65 |
| 15 kGy | 227 | 183 | 374 | 297 | 59.5 | 42.80 | 21.43 |
| 25 kGy | 225 | 180 | 377 | 330 | 61.0 | 42.00 | 26.31 |
| 40 kGy | 230 | 187 | 369 | 275 | 62.2 | 36.80 | 23.20 |

associated to the combination of N–H bending and CN stretching of the peptide group. Markers of alteration in wool keratin indicated by the literature and evaluated in this study are: the ratio of amide I and II bands and the separation between amide I and II bands ($\Delta\nu = \nu_{AI}-\nu_{AII}$) (table 4).

The increase of the amide I/amide II ratio can be associated with hydrolysis of the polypeptide chains; the increase in the distance between the two amidic structures (amide I – amide II) indicates a denaturation of the amide structure [27]. Analyzing the data from the table 4 one may conclude that pre-irradiation treatment did not significantly changed the chemical structure of the treated fabrics (fig. 2).

*Scanning electron microscopy*

In the case of 100% wool fabrics no differential corrodation occurs from one side to another of the surface, the cracks in the scales are present to a lesser extent, with a random distribution on the surface of the fiber, with rare spacing and separation of fibre body scales, the type and the quantity of these changes having a random distribution

**Table 4**
CALCULATED SPECTRAL PARAMETERS*

| Code | $IA_I/IA_{II}$ | $\Delta\nu = \nu_{AI}-\nu_{AII}$ ($cm^{-1}$) |
|---|---|---|
| Untreated | 2.97 | 132.87 |
| 0 kGy | 2.68 | 134.25 |
| 5 kGy | 2.75 | 135.81 |
| 10 kGy | 2.69 | 134.74 |
| 15 kGy | 2.65 | 136.85 |
| 25 kGy | 2.65 | 134.72 |
| 40 kGy | 2.69 | 134.08 |

* data are obtained after averaging 5 spectra

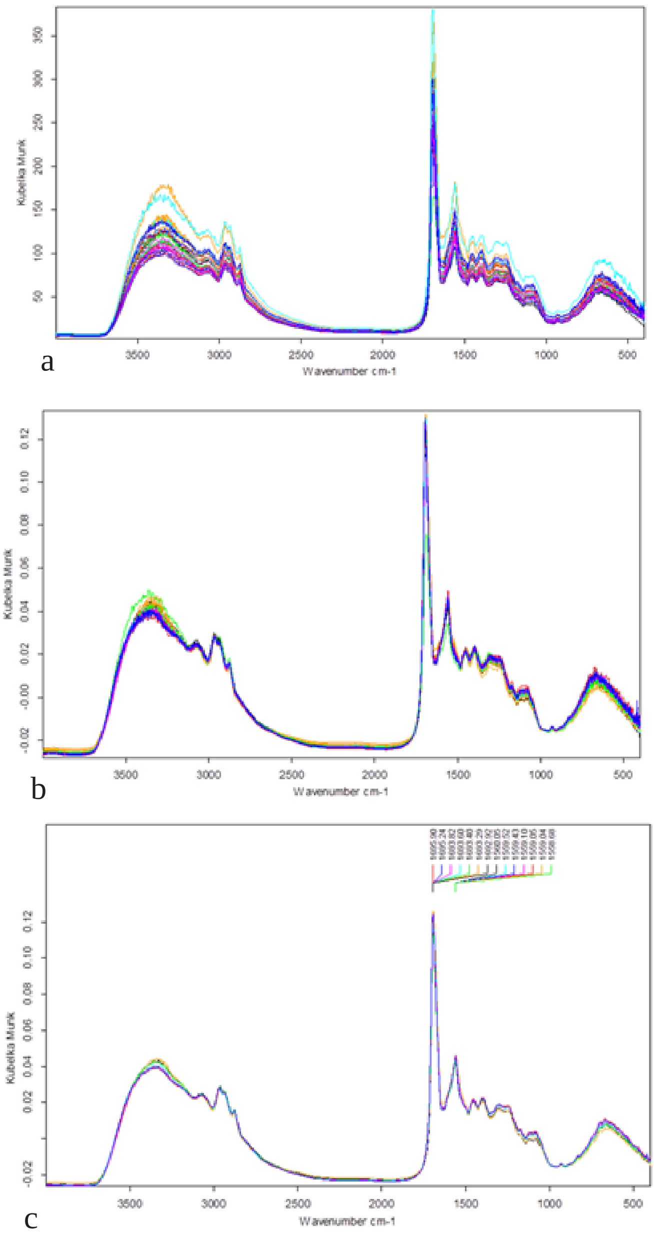


Fig. 2. Spectra obtained for 100% wool fabrics, irradiated with different doses, naturally dyed and mordanted with iron alum a - original spectra, b - vectorial normalised spectra, c - average spectra untreated: 0 kGy, 5 kGy, 10 kGy, 15 kGy, 25 kGy, 40 kGy

on the surface of wool fibers. In conclusion, the application of pre-irradiation processes in order to improve the adsorption capacity of fabrics made of natural fibers for further natural dyeing performed according to the mentioned dyeing recipe, will not lead to the modification of the constituent fiber surface morphology.

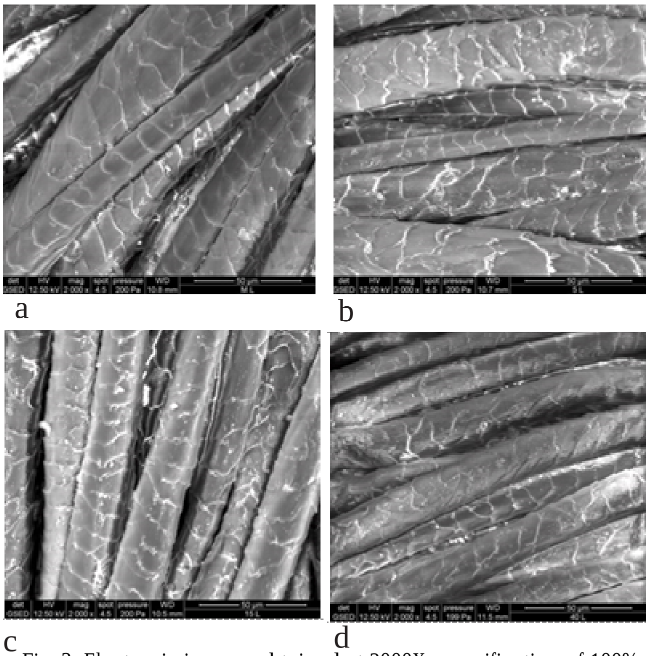


Fig. 3. Electronic images obtained at 2000X magnification of 100% wool fabrics subjected to gamma pre-irradiation and natural dyeing: a - untreated, b - dose of 5 kGy, c - dose of 15 kGy, d - dose of 40 kGy

## Conclusions

The conclusions summarize the aspects made during research on the application of pre-treatments with gamma radiation on textile materials in order to improve the adsorption capacity of natural dyes and to obtain additional benefits. Pre-irradiation with doses of 10 kGy, 25 kGy, 40 kGy of wool fabrics positively influences the adsorption capacity of natural dye, effect resulted in getting darker and more saturated shades. The SEM observations show slight visible damage to the surface morphology. Analysis of infrared data indicates that pre-irradiation treatment did not significantly change the chemical structure of treated fabrics. Gamma irradiation with doses not exceeding 40 kGy does not cause significant deterioration in the mechanical properties of wool fabrics. The irradiation dose of 25 kGy leads to higher value of alkali solubility. The colour fastness to light, washing, perspiration and rubbing has not been improved by dyeing irradiated wool fabrics.

It is important to suggest here that two kinds of effects might occur in parallel in wool during the irradiation. The first effect as manifests as an evident decrease in dye accessibility at lower doses may not be altogether independent of crosslinking. On the other hand, the remarkable increase in the take-up at higher doses seems to be associated with structural damage of fibers.

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