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Sumar

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Analysis of thermal decomposition of selected natural fibres on the basis of visual changes during heating

IZABELA JASIŃSKA

REZUMAT – ABSTRACT

Analiza descompunerii termice a fibrelor naturale selectate pe baza evaluării vizuale în timpul procesului de încălzire

Proprietățile termice ale fibrelor liotrope sunt importante pentru procesele de tratare şi finisare (vopsire, stabilizare și imprimare), precum și pentru procesele de întreținere. Schimbările care au avut loc în structura fibrelor sub influența energiei termice au fost caracterizate prin modificarea culorii fibrelor (distrugerea termică a legăturilor chimice) și contracția acestora. Analiza influenței energiei termice asupra rezistenţei structurii fibrelor, prezentate în manuscris, s-a bazat pe evaluarea microscopică a modificărilor survenite în structura fibrei. Nouă tipuri de fibre au fost evaluate în acest studiu. Acestea sunt: fibre celulozice naturale, fibre celulozice regenerate – fibre de viscoză şi proteice – soia și mătase. Fibrele provin din surse diferite – mănunchiuri de fibre, semitort și fire. Pe baza testelor efectuate s-a stabilit că, pentru fibrele de bumbac şi in, sursa materiei prime poate fi importantă în procesul de identificare, cu referire la descompunerea termică. În ceea ce priveşte fibrele de viscoză (sau atunci când există probabilitatea apariției lor), s-a constatat că aceste fibre sunt caracterizate printr-o contracție termică variată, în funcție de tipul lor. Se poate concluziona că fibrele cu aceeași compoziție a materiei prime pot prezenta proprietăți diferite în ceea ce privește contracția termică și ritmul de descompunere la o anumită temperatură, care ar trebui să fie luată în considerare în timpul procesului de tratare și întreţinere la temperaturi mari.

Cuvinte-cheie: fibre celulozice, descompunerea termică a fibrelor, fibre non-termoplastice, contracția termică a fibrei

Analysis of thermal decomposition of selected natural fibres on the basis of visual changes during heating

Thermal properties of lytropic fibres are important for finishing treatment processes (dying, stabilization, and printing) as well as for maintenance processes. The changes, which occurred in fibres structure under influence of thermal energy were characterized by colour changing of fibres (thermal destruction of chemical bonds) and their shrinkage. The analysis of influence of thermal energy to fibres structure fastness, presented in manuscript, was based on visual, microscopic, assessment of changes occurred in fibre structure. The nine genres of fibres were evaluated in this study. There were natural cellulose fibres, regenerated cellulose fibres – viscose and protein fibres- soya and silk. Fibres came from different sources – loose fibres, roving, and yarn. Based on the performed tests, it was stated, that for cotton and flax fibres, the source of raw material may be important during the process of identification with reference to thermal decomposition. As for viscose fibres (or when there is likelihood of their occurrence), it was found that these fibres are characterised by varied thermal shrinkage, depending on their type. It may be concluded that fibres of the same raw material composition may present different properties in terms of thermal shrinkage and pace of decomposition at certain temperature, which should be considered during the treatment process and maintenance using high temperature.

Keywords: cellulose fibres, thermal decomposition of fibres, non- thermoplastic fibres, thermal shrinkage of fibre

INTRODUCTION

Evaluation of properties of fibres during the process of thermal decomposition is an important issue in the process of analysis of textile products. Properties of fibres such as the pace of change propagation in the structure of a fibre caused by the increasing temperature of the product or occurrence of the phenomenon of thermal shrinkage of a fibre are relevant when designing, e.g. processes of finishing treatment of textiles or preservation processes. In laboratory practice this is described as temperature of decomposition (non-thermoplastic fibres) or melting, softening temperatures for thermo-plastic fibres as one of the methods of identifying raw material, as well as microscopic analysis, attempts to burn, chemical methods or evaluation using FTIR spectrum – these are methods listed in the Technical Report ISO/TR 11827:2012 [1–2].

Due to the preparation of new types of fibres, their characteristics are presented as well as their dissimilarity towards other types of materials [3–5]. Moreover, the already known techniques of fibre identification undergo constant improvement, especially those closely related to each other. In their publication, the authors elaborated an improved method of differential identification of flax and ramie fibres [6]. In the publication the thermal properties and thermal durability of some natural fibres – cotton, ramie and wool were investigated [7]. The TD-FTIR (temperature-dependent Fourier infrared transform) and differential thermal analyses (TG-DSC) were used for determination of degradation and water loss of mentioned fibres. Researchers found, that in the range of temperature from 25°C to 210°C for all tested fibres thermal degradation phenomenon was not detected. In another research work the improvement of physical and mechanical properties of hemp fibres and their thermal resistance was investigated [8]. The large group of research works in scope of properties, such as thermal degradation, mechanical strength, for new-investigated composites were undertaken. Most of composites consisted of some natural fibre – sisal, Boraussus fruit fibre, short curaua fibre and synthetic polymer – PET, polypropylene [9–12]. As an example, in the research work the thermogravimetric analysis (TGA) was used for determination thermal decomposition of all composite components (recycled PET, sisal fibres) [9]. It is important feature in composite investigation, because the differences of thermal degradation for composite substitutes made decomposition process faster and easier. Mostly the techniques of processing and image analysis supported by data mining technique are mostly used in fibre identification process [13–15].

Summarising the above literature review, it can be stated that in order to differentiate textile products of natural origin and analysis of composites consisted of natural and synthetic fibres, there is a wide range of methods of evaluation and identification applied, such as TD-FTIR, TGA or TG-DSC. The microscopic evaluation of fibres is used rather in case of investigation of image analysis-based identification techniques or in form of SEM images. There were not found in literature instances of thermal degradation analysis based on continuously observation of heated fibre. The microscopic analysis of thermal degradation process provides another kind of information than above mentioned techniques (e.g. TD-FTIR). The direct observation of heated fibres allows to investigate even slight alteration of fibre's particular parts – shape of longitudinal view, existence of shrinkage, fibre colour. This kind of information could be important for finishing treatment process or setting of appropriate cleaning parameters for the end users (washing, drying or ironing temperature). Moreover, optical microscopy with additional devices to control of degradation temperature is economical, less time consuming (easy way of preparing samples) in comparison to other advanced techniques.

The aim of this work is to present the analysis and evaluation of the process of fibre thermal decomposition that is based on the microscopic, continuously observation method. The final result of thermal decomposition, according to standards is only the value of ignition temperature [1, 17]. In the presented paper, the analysis of the whole process of thermal decomposition of fibres in a function of temperature together with establishing their final temperature of thermal decomposition was investigated. The typical standard test of ignition temperature has been enlarged; the analysis of detail changes during thermal decomposition process was investigated. Additionally, phenomena accompanying fibre decomposition were evaluated, such as longitudinal or transverse directions shrinkage. Applying the technique of identification (microscopic method and attempt to burn) is based on methodology included in the standards [1, 17]. Identification of materials using the microscopic method together with the thermal analysis were performed in the context of influence of fibre origin, i.e. establishing whether fibres maintain their characteristic features in a ready-made product.

EXPERIMENTAL WORK

Materials and Method

The material selected for tests comprised of a group of natural plant fibres and synthetic fibres on the basis of regenerated cellulose. Fibres that were evaluated were of the following origin: roving, knitting yarn, weaving yarn, final products (fabrics, knitwear). The different sources of tested fibres had implemented due to simulation of real laboratory testing conditions (identification of raw material composition using optical microscopy). Due to the fact that all the analysed fibres are non- thermoplastic, most frequently it is usually only the temperature of their decomposition and the temperature of disintegration that is determined. In table 1 there are the types of fibres together with their characteristic. All yarns taking under consideration in this paper and being a source for tested fibres were not finished (died or blenched). The proposal of end usage of yarn (knitted or woven fabrics) was connected with yarn structure and characteristic (twist).

Since all of the tested materials belong to the group of non-thermoplastic fibres, their physical state did not change during rising the temperature of fibres, but the chemical structure was altered as a result of chemical decomposition of the polymer that created the fibre [3]. Observations of the thermal degradation process of the fibres were performed in the conditions of heating using Dialux microscope equipped with a heated table (maximum table heat temperature was 350°C). The changes taking place during the process of heating were noted together with the values of temperature at which they took place. The aim of the test was not only to establish the temperature of fibre decomposition, but also to characterise the stages of thermal degradation that could be observed in the change of colour or shrinkage. During the analysis, the behaviour of the fibre in the time of the controlled burn process was described, including the following:

- change of fibre colour in time resulting from its progressive chemical destruction,
- fibre shrinkage and its intensity, and directivity (change of the transverse dimension of the fibre or a shrinkage along the fibre length),
- fibre deformation understood as a change in a fibre shape and not classified as a shrinkage in any direction.

In order to unify the results during their further presentation and analysis, the new qualitative scales were assumed that included natural numbers. The scales of changes in colour and longitudinal view have been investigated during research process and presented form is a result of evaluation of alternation in different fibre structure and properties. Table 2

Table 1

RAW MATERIALS AND GENRES OF FIBRE				
No.	**Raw material**	**Sample no.**	**Fibre**	**Fibre source**
1	cellulose (cotton)	1a	cotton	20 tex yarn dedicated for knitted fabrics
		1b	cotton	roving
2	cellulose (hemp)	2a	hemp	25 tex yarn dedicated for knitted fabrics
3	cellulose (jute)	3a	jute	loose fibre
4	cellulose (ramie)	4a	ramie	loose fibre
5	cellulose (flax)	5a	flax	30 tex yarn from oakum, dry spinning, dedicated for weaving fabrics
		5b	flax	30 tex yarn, wet spinning dedicated for weaving fabrics
		5c	flax	yarn took from final product
6	regenerated cellulose	6a	viscose	20 tex yarn dedicated for knitted fabrics
		6b	viscose FR	20 tex yarn dedicated for knitted fabrics
7	bamboo cellulose	7	bamboo	20 tex yarn dedicated for knitted fabrics
8	soya protein	8	soya	25 tex yarn dedicated for knitted fabrics
9	silk skleroproteine (fibroine, sericin)	9	silk	loose fibre

Table 2

THE SCALE OF THERMAL PROPERTIES ASSESSMENT		
Scale degree	**Colour of fibre**	**Shrinkage intensity**
0	no changes	no shrinkage
1	light brown	light longitudinal shrinkage
2	middle brown	severe longitudinal shrinkage
3	dark brown	slight transverse shrinkage
4	light black	severe transverse shrinkage
5	middle black	deformation - curl
6	dark black	
7	carbonized fibre	

includes descriptions referring to individual degrees, however, there is a separate scale for colour change and intensity of a shrinkage of fibres during heating.
On the basis of the new assumed qualitative scale describing the process of thermal degradation of fibres, the obtained results are presented in form of figures.

THERMAL ANALYSIS OF FIBRES

As part of the preformed analysis, for each fibre a characteristic of its thermal properties was prepared, including temperature values of thermal decomposition. These values are accessible for some fibres in literature, e.g. [2, 15]. Detailed results obtained for fibres are presented in form of figures:

- change of fibre color in its temperature growth function;
- intensity of fibre shrinkage during the temperature increase.

Colour change of fibre's structure

Figures 1–4 present a characteristic of colour change (caused by thermal decomposition) that takes place in fibres during the process of the temperature increase. Figure 1 presents a summary of results for fibres not presented in other figures. Figures 2–4 show detailed thermal characteristics for cotton, viscose and flax fibres.
Having analyzed the graphs presented above with thermal decomposition of fibres, the following can be concluded:

- silk fiber decomposition (fig. 1) proceeded in a different manner due to the presence of sericin coating of the fibre. Initially, the destruction of the fibre took place from inside of its structure and black colour was a dominant one. Then, decomposition included also the inside of the fibre where the brown colour prevailed and finally, the fibre was carbonized at the temperature of 290°C;

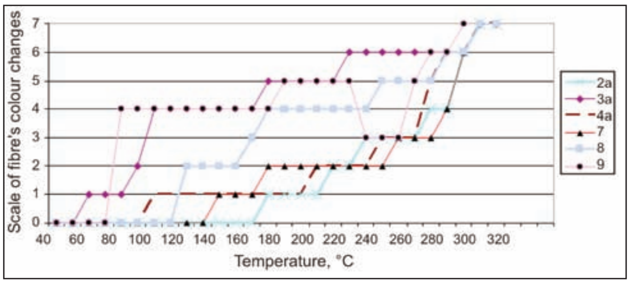

Fig. 1. Thermal resistance of fibres

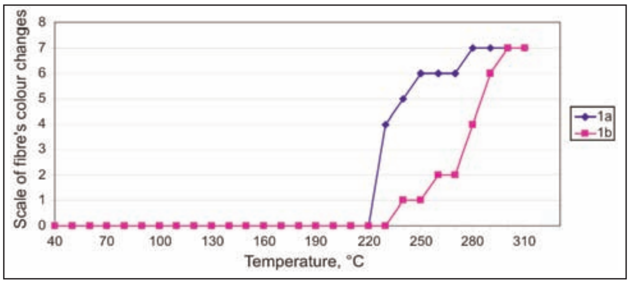

Fig. 2. Thermal resistance of cotton

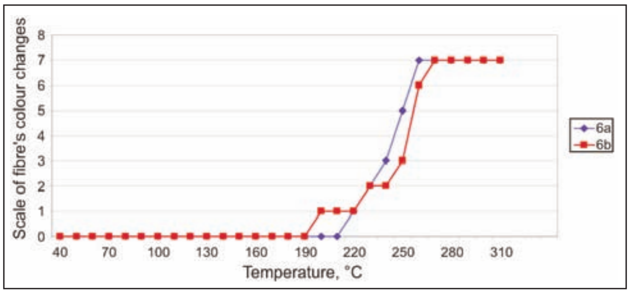

Fig. 3. Thermal resistance of viscose

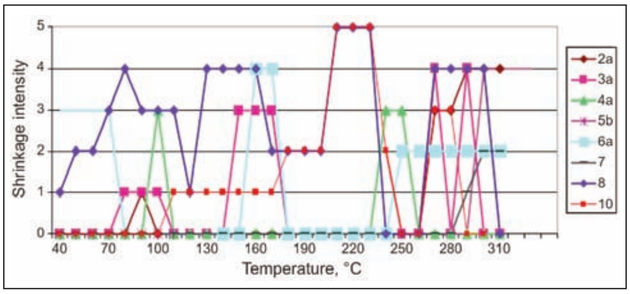

Fig. 5. Thermal resistance of fibres – shrinkage

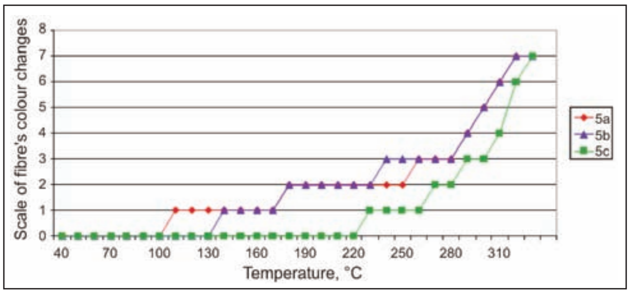

Fig. 4. Thermal resistance of flax

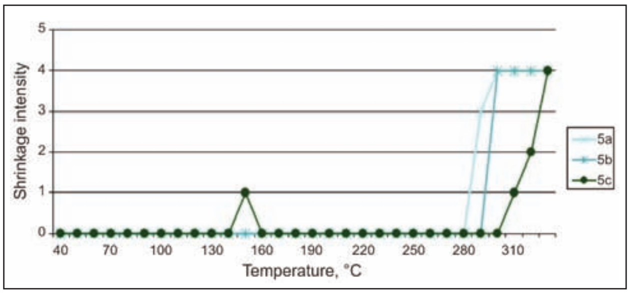

Fig. 6. Thermal resistance – flax fibre shrinkage

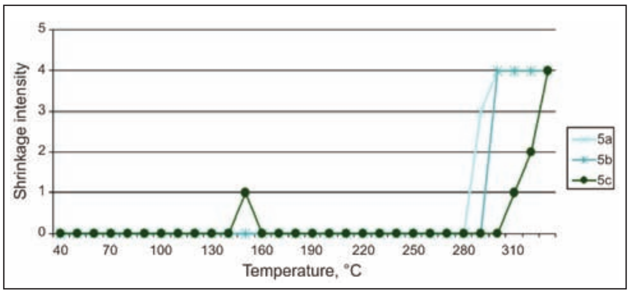

Fig. 7. Thermal resistance – viscose fibre shrinkage

- the highest thermal resistance was observed for the flax fibre – about 330°C, whereas the lowest for viscose fiber – 270°C;
- among the flax fibres, the highest thermal resistance was observed for fibres in the final product, however, this may be connected with the presence of finishing (during the process of heating, there were bubbles of liquid around the fibres). The level of temperature value of the thermal decomposition of the fibre is similar for fibres from all analyzed sources (differences are around 10°C);
- viscose fibres present a similar thermal resistance and slight differences in the characteristic picturing their decomposition do not point to any distinct properties in this area. With reference to viscose fibres, modifications are possible (e.g. as a result of incorporating a substance into the fibre) that allow to obtain various thermal properties – e.g. fibres with higher thermal resistance.

Thermal shrinkage evaluation

Figures 5–7 present the intensity of shrinkage process of the fibres under higher temperature conditions. Figure 5 presents all types of fibres that were analysed and for which there was a shrinkage during the process of heating. Figure 6 and 7 concern the fibres of flax and viscose.

While analysing the figures presenting the process of thermal shrinkage of fibres during heating, the following can be observed:

- for cotton and silk fibres, the phenomenon of shrinkage during the process of thermal destruction did not occur;
- the strongest shrinkage, both longitudinal to the axis of the fibre (shrinkage on the axis) and in the direction perpendicular to the axis of the fibre (reducing the dimension of the fibre) appeared for the fibres of viscose and soya;
- above the temperature of 260°C in all evaluated fibres, a shrinkage is visible, however, for viscose fibres it is continuous whereas for all the others the shrinkage is of temporary character (it appears at certain temperatures to disappear later and reappears at higher temperature values);
- for soya fibres, deformation appears that is indirectly connected with the phenomenon of shrinkage in both directions mentioned above. Changing the shape of the fibre is its distinct property (a fibre becomes crimped and twisted which suggests uneven shrinkage of its individual fragments);
- in the group of flax fibres, the highest thermal resistance (with reference to shrinkage) are presented by fibres taken from a final product, which was noted during the analysis of the change in the fibre colour, for other fibres shrinkage process begins rather rapidly, at about 280–290°C and lasts until fibre carbonization.
- in case of viscose fibres, fibres taken from yarn (6a) present lower thermal resistance, shrinkage appears at the very beginning of the process of fibre heating and is rapid (presence of a shrinkage in both directions against the axis of the fibre). For FR viscose, shrinkage appears later and is limited

to a longitudinal direction against the axis of the fibre. The shrinkage becomes stronger at about 240–250°C and lasts until fibre carbonization.

RESULTS

Summarising the presented results of research into identification of the selected natural and man-made fibres using a microscopic method on the basis of a tested sample the following may be concluded:

- the source of cotton, flax and viscose fibre (in relation to the degree of a textile material treatment) may become the reason of their various thermal properties both in terms of temperature of thermal decomposition and its characteristics;
- the lowest thermal resistance among the evaluated fibres have viscose fibres and they are characterised by shrinkages occurring at much lower temperatures than for the other fibres that were evaluated;
- the highest thermal resistance is presented by flax fibres collected from the final product, which was also confirmed by the analysis of their colour change;
- soya fibre was the most distinct one as it was characterised by a strong deformation (curl) directly after the increase of temperature (around 45°C).

The aim of this work was to establish whether fibres maintain their characteristic features in terms of their thermal properties in a final product (knitting yarn, fabric knitwear) as fibres evaluated in a laboratory most frequently are obtained from products. Basing of the performed tests, it was stated that for cotton and flax fibres, the source of raw material may be important during the process of identification with reference to thermal decomposition. As for viscose fibres (or when there is likelihood of their occurrence), it was found that these fibres are characterised by varied thermal shrinkage, depending on their type. On the basis of the presented results, it may be concluded that fibres of the same raw material composition may present different properties in terms of thermal shrinkage and pace of decomposition at certain temperature, which should be considered during the treatment process and maintenance using higher or high temperature.

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Development of a machine vision system for yarn bobbin inspection

H. İBRAHIM ÇELİK

REZUMAT – ABSTRACT

Dezvoltarea unui sistem de control vizual pentru inspecţia bobinelor de fire

Când diferite materii prime sunt amestecate în orice etapă a producției de fire, se creează defecte de fibre pe bobina de fire. Deoarece fiecare fibră are caracteristici diferite de afinitate a colorantului, acest element poate determina producerea unei ţesături de calitate mai slabă. În ciuda noilor sisteme de automatizare utilizate în fabricile de producţie a firelor, defectele amestecurilor de fibre sunt încă inspectate de către ochiul uman. Acest proces este mare consumator de timp, iar evaluarea se face în mod subiectiv. Mai mult decât atât, multe bobine cu defecte de amestec al fibrelor pot fi trecute cu vederea de către operator. În acest studiu, a fost dezvoltat un prototip de sistem de control vizual pentru a detecta defectele amestecului de fibre prin utilizarea metodei de procesare a imaginii. S-a urmărit ca defectele amestecului de fibre al bobinelor de fire să poată fi detectate automat, iar evaluarea defectului să poată fi făcută în mod obiectiv. Algoritmul de detectare a defectelor s-a bazat pe filtrul Wiener, filtrul Gaussian și pe operațiile morfologice. Șase bobine de fire: trei cu defecte ale amestecului de fibre și alte trei fără defecte ale amestecului, în calitate de grup de control, au fost verificate cu succes, iar zonele cu defecte au fost etichetate.

Cuvinte-cheie: control vizual, amestec de fibre, inspecţia bobinelor de fire, procesarea imaginii, identificarea defectelor

Development of a machine vision system for yarn bobbin inspection

When the different raw materials are blended in any step of the yarn production, it causes fiber mixture fault on the yarn bobbin. Since each fiber has different dye affinity characteristics, this fault causes off-quality fabric production. Despite the new automation systems used in yarn manufacturing mills, the fiber mixture fault is still inspected by human eye. This process takes long time and the evaluation is made subjectively. Furthermore, many bobbins with fiber mixture fault may be escaped from the worker notice. In this study, a prototype of vision inspection system was developed to detect the yarn fiber mixture fault by using image processing method. It was aimed that the fiber mixture faults of the yarn bobbins can be detected automatically and the fault evaluation can be made objectively. The fault detection algorithm was based on Wiener filtering, Gaussian filtering and morphological operations. Six yarn bobbins; three of them with fiber mixture fault and other three having no mixture fault as control group were detected successfully and the fault areas were labeled.

Key-words: Machine vision, fiber mixture, yarn bobbin inspection, image processing, fault detection

INTRODUCTION

The basic staple spun yarn manufacturing in ring spinning system consists of the main production processes namely; opening, cleaning, blending, carding, drawing, roving, ring spinning, winding and packaging [1]. The raw material of the yarn is introduced to the blowroom as fiber bales and they are converted into yarn form by following the production steps sequentially. Each yarn production is performed by using distinctive raw material. So, unique code is used for each yarn production even they have the same fiber type such as cotton, viscose etc. In fully automated mills, the material transfer between the production steps are performed by using automation systems. Finally, the yarn bobbins are packaged by using a robotic system.

When the different raw materials are accidentally mixed in any step of the yarn production, it causes fiber mixture fault on the yarn bobbin. Despite the new automation systems used in yarn manufacturing mills, the fiber mixture fault is still inspected by human eye. The yarn packages are loaded on a creel and they are detected in a UV lightened room. This process is time consuming and it has the risk of yarn fault missing. For fully automated mills, this inspection method causes more problems, since the human intervention is not permitted during manufacturing. The fiber mixture bobbins cause more problems after weaving operation. Since each fiber has different dye affinity characteristics, this fault causes high cost due to off-quality fabric production.

In the literature, there are some studies on foreign material and fiber detection [2–7]. In these studies, machine vision systems have been designed for fiber lint inspection. The studies are especially performed for cotton lint. Any study or commercially developed system that achieves the fiber mixture fault inspection on yarn bobbin has not been encountered.

In this study, a prototype vision inspection system was developed to inspect the yarn bobbins for fiber mixture fault and image processing algorithm was prepared. Yarn samples with fiber mixture fault and no fault were used. The samples are inspected by using the prototype vision inspection system to determine the mixture fault existence.

EXPERIMENTAL PART

Material

Six 100 % cotton yarn samples were used. Three of them comprise mixture fault caused by several reasons i.e. cotton fiber comes from different bales, winding of different types of yarn cops on the same bobbin etc. Other three samples did not have any fiber mixture, they are evaluated as control group. The yarn bobbins were supplied from different yarn spinning mills, they were selected randomly.

Vision inspection system for yarn package inspection

Prototype vision inspection system has been developed in order to acquire image of the yarn bobbin properly and to analyze it for the fiber mixture existence. The system consists of lightening unit, High Density (HD) web camera, cabin and computer (figure 1). The most important parameter that determines the reflectance of the yarn is the fiber type comprising the yarn structure. Each fiber type has different reflectance values under the same lightening condition. So, Ultra Violet (UV) fluorescents are used as lightening unit to identify different fiber groups according to their different luminance values [8]. The image acquisition process is achieved by using a Logitech HD Pro Webcam C920. The resolution of the camera 1920×108 pixels. The camera is connected to the computer via USB port. Since any light entrance expects the UV lightening source will change the image analyzing result, the cabin is designed to provide a darkroom condition.

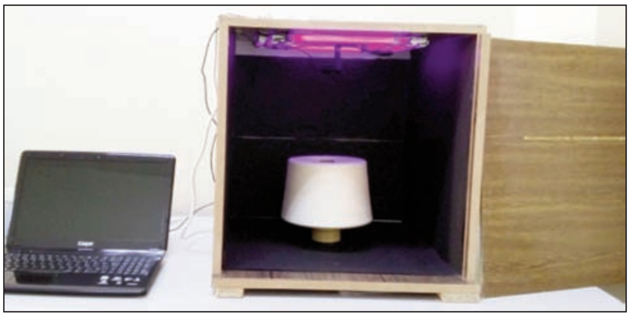


Fig. 1. Prototype yarn package vision inspection system

Method

The algorithm developed for detection of fiber mixture in a yarn package consists of noise removing filter, Gaussian filter and morphological operation (figure 2). Gaussian filter is used to segment the regions that have different intensity values according to the neighborhood relation. The morphological operations are used to remove the noises and to make the regions clearer.

The image frame may have noises because of illumination condition. It is recommended that the noise should be removed to get the true pixel intensity values. So, the image frame captured from the HD camera is applied Wiener low-pass filter for noise removing. Wiener estimates the local mean and the variance around each pixel. Wiener filter makes its estimation by using following equation [9]:

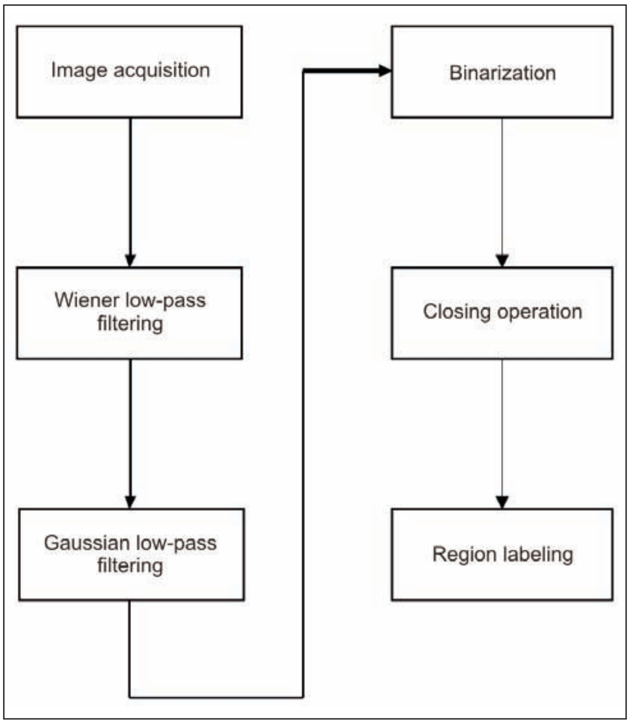


Fig. 2. Flow chart of the algorithm

$$b_{(n_1,n_2)} = \mu + \frac{\sigma^2 - \nu^2}{\sigma^2}\left(a_{(n_1,n_2)} - \mu\right) \qquad (1)$$

where ν^2 is the noise variance, μ and σ^2 are the local mean and local variance around each pixel respectively.

The noise filtered image is then applied Gaussian filtering. The Gaussian filter is used for image smoothing as low-pass filter. The image frame is convolved with Gaussian function [9]:

$$h_{(n_1,n_2)} = e^{\frac{-(n_1^2+n_2^2)}{2\sigma^2}} \qquad (2)$$

where, n_1 and n_2 are the locations of the related pixel, σ^2 is the variance of the neighborhood. The filtered image is converted into binary form. The binarization operation is carried out by using a threshold level as following:

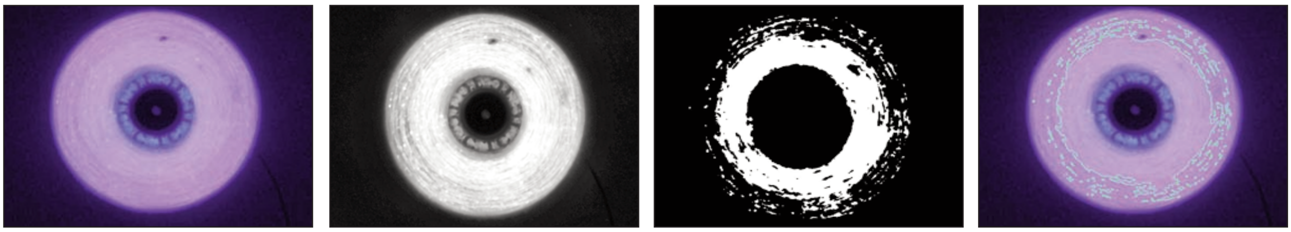
$$P_{(i,j)} = \begin{cases} 0 & a(i,j) < T \\ 1 & a(i,j) > T \end{cases} \qquad (3)$$

All pixel values $a(i,j)$ greater than the threshold value (T) in the input image is replaced with the value 1 (white) and all other pixels values are replaced with 0 (black). The threshold value (T) is determined by trial and error method. Thus, the pixels that have close gray level values are separated and detected.

The binary image is applied closing morphological operation. Closing is the name given to the morphological operation of dilation followed by erosion with the same structuring element

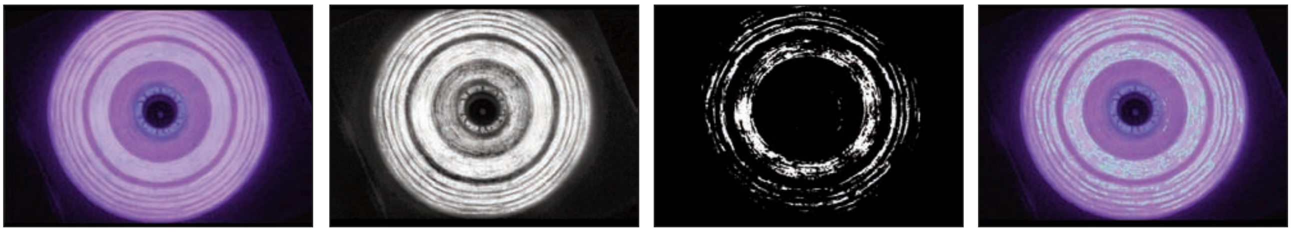
$$A \cdot B = (A \oplus B) \ominus B \qquad (4)$$

(i) yarn bobbin sample



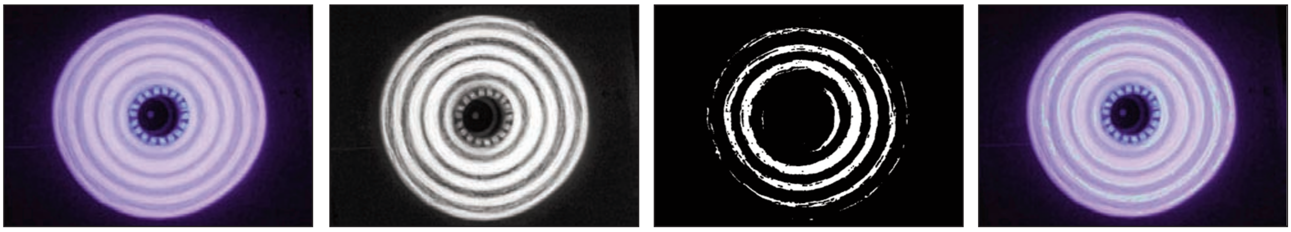
(a) RGB (b) Gaussian filtered (c) Binary (d) Labeled

(ii) yarn bobbin sample



(a) RGB (b) Gaussian filtered (c) Binary (d) Labeled

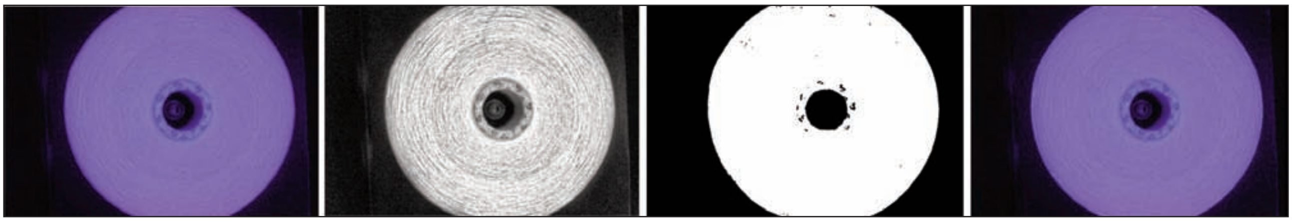
(iii) yarn bobbin sample



(a) RGB (b) Gaussian filtered (c) Binary (d) Labeled

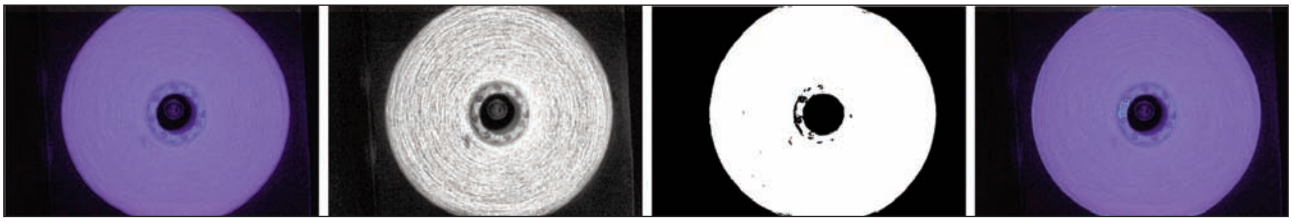
Fig. 3. Fiber mixture inspection of yarn bobbins

(iv) yarn bobbin sample



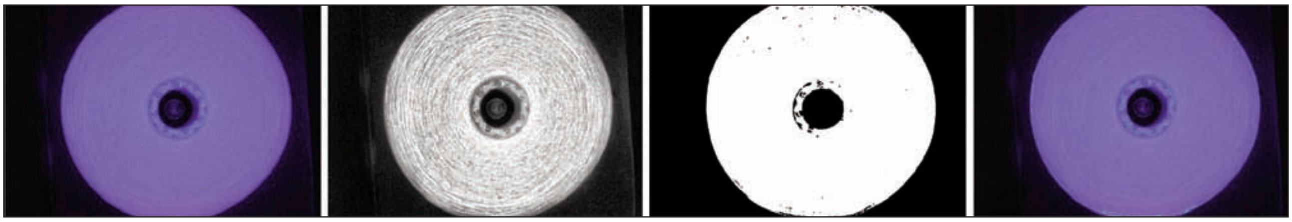
(a) RGB (b) Gaussian filtered (c) Binary (d) Labeled

(v) yarn bobbin sample



(a) RGB (b) Gaussian filtered (c) Binary (d) Labeled

(vi) yarn bobbin sample



(a) RGB (b) Gaussian filtered (c) Binary (d) Labeled

Fig. 4. Inspection of faultless yarn bobbins

Closing operation is used to eliminate specific image details smaller than the structuring element. This morphologic operation connects objects that are close to each other and fills up small holes. Thus the boundary of the object is made smoother [10]. Finally, the boundaries of the detected regions are labeled.

RESULTS AND DISCUSSION

The fiber mixture existence of the six yarn bobbins are detected by using prototype vision inspection system. After the yarn bobbin image frames are acquired via the developed system, the image frames are analyzed by applying the developed algorithm. Thus, the boundaries of three bobbins that have fiber mixture faults are detected and labeled successfully (figure 3). Other three yarn bobbins that consist of single fiber type are detected as control group (figure 4). The yarn samples with fiber mixture faults have different amount of mixtures at different places of the bobbins.

A user interface is prepared on MATLAB® program to apply the algorithm easily (figure 5). The user interface consists of *Exit, Image Acquisition, Fiber Mixture Inspection* and *Fault Labeling* buttons. After the yarn bobbin is placed into the cabin, the camera is activated and the image frame is acquired from the top of the yarn bobbin by using *Image Acquisition* button (figure 5). By using *Fiber Mixture Inspection* button, the image frame is analyzed by applying the developed algorithm (figure 2) and the result is displayed on the screen (figure 6). The detected regions are labeled by using *Fault Labeling* button (figure 7).

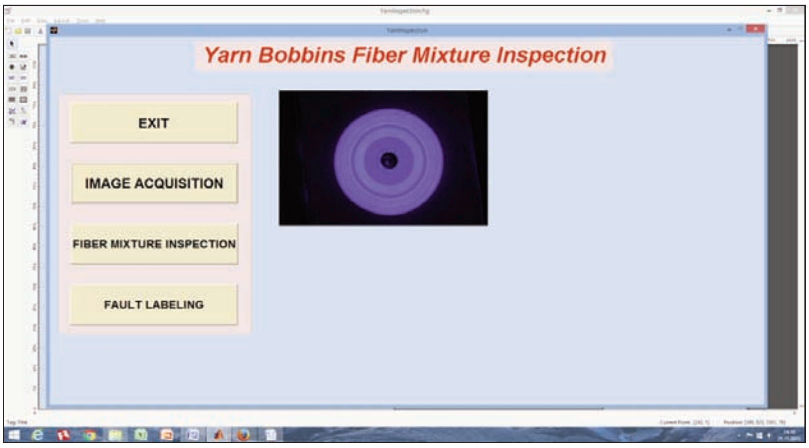


Fig. 5. User interface and image acquisition application

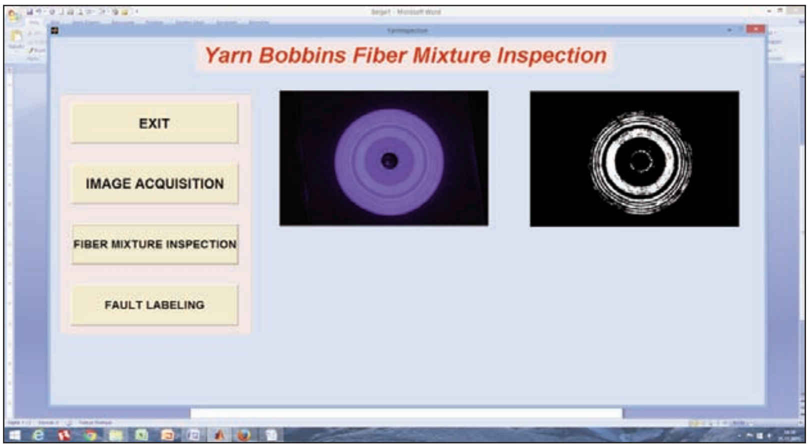


Fig. 6. Fiber mixture inspection algorithm application

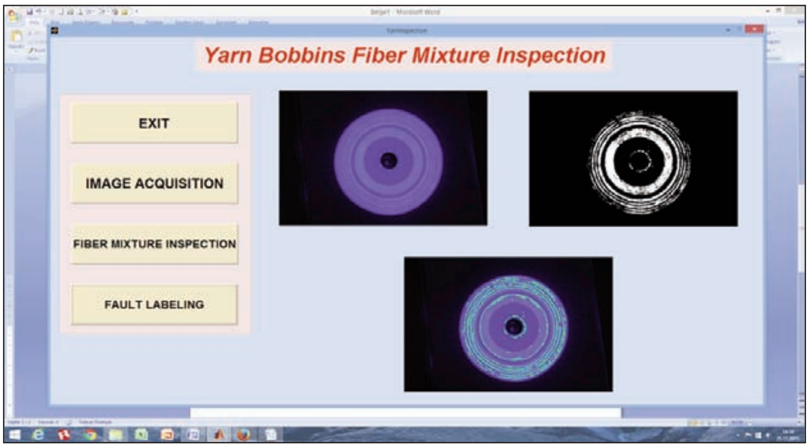


Fig. 7. Fault labeling application

CONCLUSION

In this study a prototype vision inspection system was developed for detection of yarn bobbin fiber mixture fault. Under the same lighting condition the fibers have different luminance values. This property provides the distinction of the fiber mixtures. The image processing algorithm developed for detection of the regions having different gray level values was based on Wiener filter, Gaussian filter, binarizaiton and morphological operation. The fiber mixture faults with different amount and locations were detected successfully and the boundaries are labeled by applying the developed algorithm. The fault detection algorithm was applied via a user interface prepared by using MATLAB®.

The study was performed as a prototype system design. The system will be improved and adapted to yarn manufacturing mill. It was aimed that the yarn bobbin inspection can be achieved in shorter time, objectively and accurately. By using this machine vision system, the employment cost would be reduced and the costs related with off-quality would be decreased.

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Electromagnetic shielding effectiveness of spacer knitted hybrid fabrics

HUSEYIN GAZI TURKSOY SINEM BILGIN

REZUMAT – ABSTRACT

Eficacitatea ecranării electromagnetice a tricoturilor spațiale hibride

În acest studiu, au fost realizate tricoturi spațiale având eficacitate de ecranare electromagnetică prin utilizarea diferitelor tipuri de fire conductive electric. Eficacitatea ecranării țesăturilor dezvoltate a fost testată prin standardul ASTM 4935-10. Încercările au fost efectuate la intervale de frecvență de 100–1000 MHz. Analiza varianței și a metodelor de Comparații multiple au fost folosite pentru evaluarea rezultatelor testelor. Rezultatele testelor indică faptul că tipurile de fire conductoare și dispunerea acestor fire în structurile ţesăturilor afectează în mod semnificativ eficacitatea ecranării tricoturilor spațiale hibride.

Cuvinte-cheie: eficacitatea ecranării (SE), fir metalic, fir cu miez, țesătură spațială

Electromagnetic shielding effectiveness of spacer knitted hybrid fabrics

In this study, spacer knitted fabrics having electromagnetic shielding effectiveness have been developed by using various types of electrically conductive yarns. Shielding effectiveness of developed fabrics has been tested by standard ASTM 4935-10. The tests have been carried out at 100–1000 MHz frequency ranges. Analysis of variance and Multiple Comparisons methods have been used for evaluating the test outcomes. The test results indicate that the conductive yarn types and arrangement of these yarns in fabrics' structures significantly affect the shielding effectiveness of spacer knitted fabrics.

Keywords: Shielding effectiveness (SE), metal wire, core yarn, spacer fabric

INTRODUCTION

In recent years, the usage of electrical and electronic devices increases rapidly. Electronic devices generate intentionally or unintentionally electromagnetic (EM) energy. EM fields have both electric and magnetic field components, and they have hazardous effects on the living tissues and electronic systems [1–11]. Unwanted reception of EM waves may create an interference phenomenon that has adverse effect called electromagnetic interference (EMI) problem, on the performance of electrical/electronic equipment [12]. The hazardous effect of EM waves on human health can also be considered as an EMI problem since the human body is actually an electrical system with a huge nervous system [4]. Because of the detected negative effects of electromagnetic waves, it has become a necessity to find the protection ways from electromagnetic radiation.

EM shielding is a process by which a material is able to reduce the transmission of electromagnetic radiation that affects the humans/equipment [13]. Traditionally, metals are used for electromagnetic shielding; however, these materials are heavy, expensive, inflexible and may be subject to thermal expansion and metal oxidation, or corrosion problems associated with their use [14]. Owing to these disadvantages of classical materials used for shielding, more flexible, lighter and low cost electrically conductive textile products have become increasingly more preferable.

There are various studies about fabric structures having electromagnetic shielding effectiveness (SE) in literature. Cheng investigated the SE of plain, 1×1 rib and 1×2 rib knitted fabrics which were produced with stainless steel (SS)/PES blended yarns, SS/Stainless Steel Wire (SW)/PES, and SW/PES core-spun yarns in different blend ratios. The SE tests of fabrics were performed at **30 kHz–3000 MHz** frequency range by using coaxial transmission line method. It was found that as the steel ratio of the yarn increases, the SE of the fabrics increases. It was interpreted that the SE of the 1×2 rib knitted structures are higher than that of the other knitted fabric structures due to the closer structure compared to the other fabrics [14].

Ciesielska and Grabwska investigated the SE of knitted fabrics with three types of knit structures, namely left-right stitch, double-layer, and double-layer with additional fillings, which contained ferromagnetic materials and electro conductive materials. They interpreted that the SE of knit structures depends on the arrangement of ferromagnetic and electro conductive materials in the knit structure [15].

Lin et. al. investigated the SE of double jersey fabrics knitted with hybrid core spun yarns. The core spun yarns were produced with SS wire and Cu wire as core materials and, rayon yarn and polyester/rayon (65/35) yarn as cover materials. The SE of double jersey fabrics were tested with a coaxial transmission line holder in the frequency range of 30 MHz to 3 GHz. It was reported that double jersey fabrics have the highest SE value in the 30–100 MHz frequency range and they have about 8–15 dB SE in this frequency range [16].

Soyaslan et al. examined the SE of seven knitted fabrics by using ASTM D 4935-10 Coaxial holder method. The knitted fabrics in plain knitting, weft in-laid plain knitting, 1×1 rib and weft in-laid 1×1 rib knitting structures were produced with copper wire and cotton blended yarns. The SE of these fabrics was made at **27 MHz – 3 GHz** frequency range. Test results showed that the weft-knitted structures have 10–40 dB shielding effectiveness under the frequency of 500 MHz [17].

As mentioned above, conventional knit structures have been preferred in many studies which are about developing fabrics offering electromagnetic shielding. However, conventional knit structures have a handicap for electromagnetic shielding property when they compared to woven fabrics. Differently from woven fabrics, the textile yarns can be found only in production direction or only vertically to it for knitted fabrics. Therefore, the conducting yarns can only be incorporated into one direction and consequently a shielding is achieved for electrical field components in just that direction for knit structures.

In the last few years, spacer knitted fabrics have been reached a greater level of importance for technical applications. Spacer fabrics have three dimensional structures consisting of two outer surfaces and a connection layer which combines two outer surfaces. In this study, due to their special construction, the SE properties of spacer knitted fabrics containing various types of electrically conductive yarns were investigated at the frequency ranges of 100–300 MHz and 300–1000 MHz.

MATERIALS AND METHODS

In the scope of study, 13 spacer fabrics having the same knitting structure were fabricated from different raw materials in front face, connection, back face, filling parts. Mayer & Cie, OVJA 1.6 E double-plate circular knitting machine was used to knit spacer fabric samples. The yarn types and their arrangements in the fabric structures are summarized in table 1. The microscopic images of conductive yarns used in this study are given in figure 1. In addition knitted needle diagram belonging to front face, connection, back face and filling yarns are given in figure 2.

SE measurements of fabric samples were made with coaxial holder method based on ASTM D 4935 standard for 100–1000 MHz frequency ranges [18]. This standard determined the shielding effectiveness of the fabric samples by using the transmission line method. The measurement device consists of a network analyzer (R & S ZVB20) generating and receiving the electromagnetic signals and a coaxial transmission line test fixture [19]. The shielding effectiveness is determined by comparing the difference in attenuation of a reference sample to the test sample, taking into account the transmission lines with this device. The reference and the test measurement were performed on the same material in this study (figure 3).

The shielding effectiveness was determined from equation (1):

$$SE = 20 \log \frac{E_1}{E_2} \tag{1}$$

Table 1

YARN TYPES AND ARRANGEMENT IN THE FABRIC STRUCTURES

Fabric Code	Front Face	Connection	Back Face	Filling
1	Ne 24/1 hybrid[1]	Ne 24/1 cotton	Ne 24/1 cotton	300 denier PES
2	Ne 24/1 cotton	Ne 24/1 cotton	Ne 24/1 hybrid	300 denier PES
3	Ne 24/1 hybrid	Ne 24/1 cotton	Ne 24/1 hybrid	300 denier PES
4	Ne 24/1 cotton	Ne 24/1 hybrid	Ne 24/1 cotton	300 denier PES
5	Ne 24/1 cotton	Ne 24/1 cotton	Ne 24/1 cotton	Ne 24/1 hybrid
6	Ne 24/1 hybrid	Ne 24/1 hybrid	Ne 24/1 hybrid	Ne 18/2 hybrid[2]
7	Ne 24/1 hybrid	Ne 24/1 hybrid	Ne 24/1 hybrid	Ne 20/2 hybrid-a[3]
8	Ne 24/1 hybrid	Ne 24/1 hybrid	Ne 24/1 hybrid	Ne 20/2 hybrid-b[4]
9	Ne 24/1 hybrid	Ne 24/1 hybrid	Ne 24/1 hybrid	Ne 30/1 carbon/ cotton[5]
10	Ne 24/1 hybrid	Ne 24/1 hybrid	Ne 24/1 hybrid	Ne 30/1 silcot[6]
11	Ne 24/1 cotton	Ne 24/1 cotton	Ne 30/1 silcot	300 denier PES
12	Ne 30/1 silcot	Ne 24/1 cotton	Ne 24/1 cotton	300 denier PES
13	Ne 30/1 carbon/ cotton	Ne 24/1 cotton	Ne 24/1 cotton	300 denier PES

[1] Ne 24/1 hybrid: core spun yarn containing 20 μ SS

[2] Ne 18/2 hybrid: twisted two 18/1 (core spun yarn containing 20 μ SS) yarn

[3] Ne 20/2 hybrid-a: twisted two Ne 20/1 cotton yarn and 20 μ SS

[4] Ne 20/2 hybrid-b: twisted two Ne 20/1 cotton yarn and 35 μ SS

[5] Ne 30/1 carbon/cotton – 10/90

[6] Ne 30/1 silcot: silver covered polyamide/cotton – 10/90

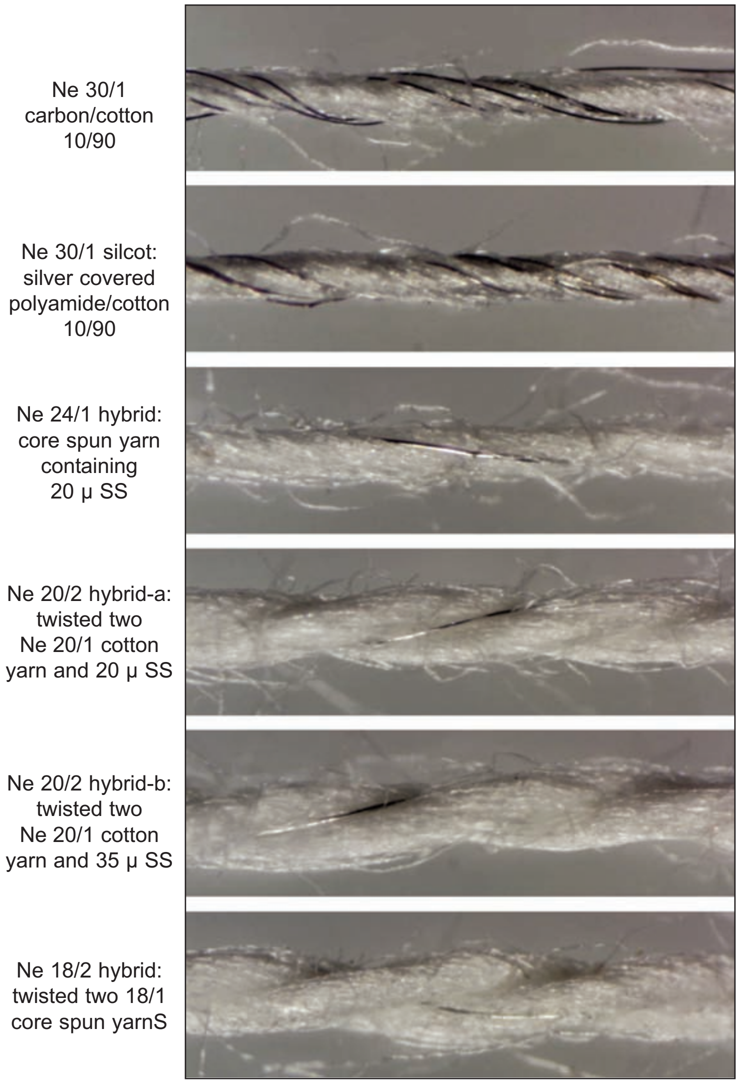

Fig. 1. Microscopic images of conductive yarns used in this study

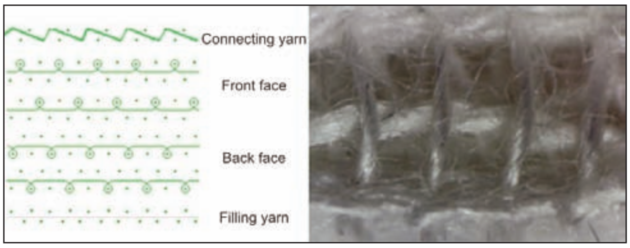

Fig. 2. The knitted report and interface section of fabric samples

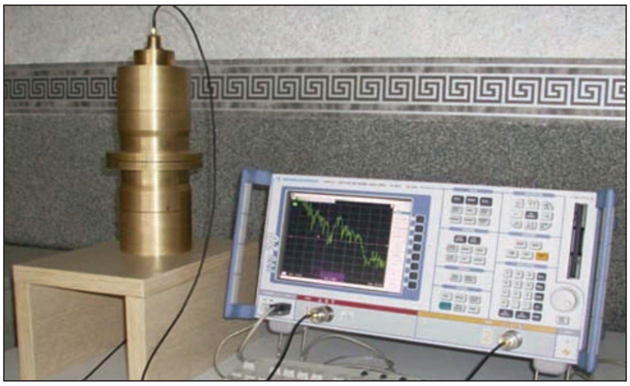

Fig. 3. Test set up of coaxial transmission line method

E_1 is the value of the electrical component and is measured with the reference sample. E_2 is measured with the test sample.

In daily life, electromagnetic fields which we have been exposed are at the **VHF (30–300 MHz)** and **UHF (300–3000 MHz)** frequency ranges. The devices frequently used such as radio, television, mobile phones, cordless phone emit electromagnetic radiation in these two frequency ranges. For this reason, after measuring SE values for 100 different frequencies between 100 and 1000 MHz at 3 different locations for each fabric type, means of the SE values of these selected locations of the samples were calculated separately for the frequency intervals 100–300 MHz as low frequency interval and 300–1000 MHz as high frequency interval. In order to evaluate the shielding performances of each fabric type, these mean values were compared and statistically evaluated for mentioned frequency intervals.

RESULTS AND DISCUSSIONS

The mean SE values of spacer fabric samples for 100–300 MHz and 300–1000 MHz frequency intervals are given graphically in figure 4 and figure 5 respectively. According to ANOVA results, the fabric type has statistically significant effect on SE values at both 100–300 MHz and 300–1000 MHz frequency intervals (p=0.000).

When SE values at 100–300 MHz frequency range of 1, 12 and 13 coded fabrics having same yarns as connection, back face and filling yarns are compared, it is seen that SE values of 12 coded fabric containing silcot in front face is lower than that of 1 coded fabric with core yarn in front face and 13 coded fabric with carbon/cotton yarn in front face (figure 4). This result shows that usage of silcot in front face doesn't contribute to SE value of fabric positively comparing to usage of core yarn and carbon/cotton yarn in front face. Also, according to Tukey test results, difference between means of SE at 100–300 MHz frequency range of 1 and 13 coded fabrics is not statistically significant (p=0.989).

When SE values at 100–300 MHz frequency range of 2 and 11 coded fabrics having same yarns as front face, connection and filling but having different back face yarns are compared, it is seen that 2 coded fabric having core yarn containing metal as back face yarn provides SE, 11 coded fabric having silcot yarn on its back face does not have any SE (figure 4). According to Tukey test results, difference between means of SE at 100–300 MHz frequency range of 2 and 11 coded fabrics is statistically significant (p=0.00).

When 1 and 3 coded fabrics are compared, 1 coded fabric having cotton yarn in back face has lower SE value than that of 3 coded fabric having core yarn in

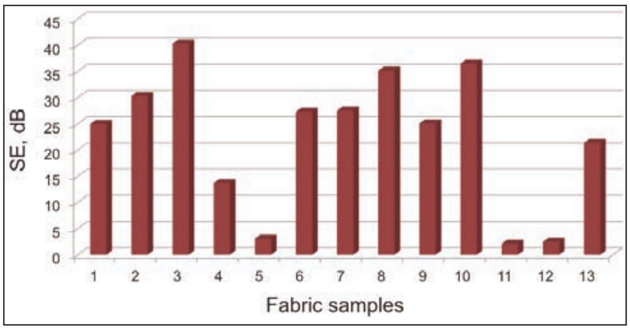

Fig. 4. The mean SE values of fabric samples in the frequency interval of 100–300 MHz

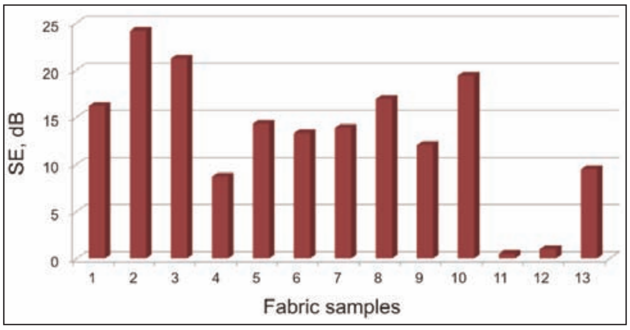

Fig. 5. Comparison of SE mean values of fabric samples in the frequency range of 300–1000 MHz

back face (figure 4). It shows that usage of core yarn containing metal wire in the fabric effects SE value positively.

6, 7, 8, 9 and 10 coded fabrics have same front face, connection and back face, but their filling yarns are different. When these fabrics are compared by using Tukey test, the differences between mean values of SE at 100–300 MHz frequency interval of 9 and 10 coded fabrics are statistically significant (p=0.032), but differences between mean values of SE belonging to other fabrics in this group are not statistically significant (p>0.05). 10 coded fabric containing silcot yarn in filling has higher SE value at 100–300 MHz frequency range than 9 coded fabric containing carbon/cotton yarn in filling (figure 4). Contrast to usage of silcot in front face, when it is used as filling yarn, it has positive effect on SE value comparing to usage of carbon/cotton yarn as filling yarn.

According to Tukey test results, when SE values at 300–1000 MHz frequency range of 1, 12, and 13 coded fabrics which have respectively core yarn, silcot and carbon as front face but have same yarns as connection, back face and filling yarns are compared, it is found that differences between mean values of SE at 300–1000 MHz frequency range of these fabrics are statistically significant (p=0.00). 1 coded fabric has the highest SE value. It is followed by 13 and 12 coded fabrics (figure 5).

When SE values at 300–1000 MHz frequency range of 2 and 11 coded fabrics having same front face, connection and filling yarns, but having different back face yarns are compared, it is seen that 2 coded fabric containing core yarn with wire in back face shows SE (24.16 dB), but 11 coded fabric containing silcot does not have SE (0.51 dB) ability as shown in figure 5.

According to Tukey test results, when SE values at 300–1000 MHz frequency range of 6, 7, 8, 9, and 10 coded fabrics having same front face, connection and back face yarns, but having different filling yarns are compared, 10 coded fabric having silcot in filling yarn has statistically higher SE value than that of other fabrics in this group except for 8 coded fabric (p=0.00). Because difference between means of SE values at 300–1000 MHz frequency of 8 and 10 coded fabrics is not statistically significant (p=0.513). On the other hand, difference between means of SE values at 300–1000 MHz frequency range of 6, 7, and 8 coded fabrics is not statistically significant (p>0.005).

CONCLUSIONS

In our daily life, electromagnetic fields which we often have been exposed are at the **VHF (30–300 MHz)** and **UHF (300–3000 MHz)** frequency ranges. In this study, the SE properties of spacer knitted fabrics containing various types of electrically conductive yarns were investigated in the frequency ranges of 100–300 MHz and 300–1000 MHz. Research results indicate that when silcot yarn is used in the fabric without metal wire, it does not have electromagnetic shielding. On the other hand, it is seen that when core spun yarns containing metal wire are used in front face, connection and back face, the fabric having silcot yarn in the filling has higher SE value than that of the fabrics having 18 (core)/2 yarn, twisted (20/2 p – 20 μ SS) and carbon/cotton as filling yarns. Analysis results justified that the conductive yarn types and arrangement of these yarns in fabric structure have an important effect on electromagnetic shielding effectiveness of spacer knitted fabrics.

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The effect of clothing insulation on the thermophysiological comfort of workers in artificial cold environment

RADOSTINA A. ANGELOVA

REZUMAT – ABSTRACT

Efectul izolației termice a îmbrăcămintei asupra confortului termofiziologic al lucrătorilor din mediul rece artificial

Studiul de față prezintă efectul izolației termice a îmbrăcămintei asupra confortului termofiziologic al lucrătorilor din mediul rece artificial. Diferite tipuri de mediu rece artificial din industria alimentară au fost identificate, împreună cu activitatea lucrătorilor. Pentru evaluarea expunerii la rece și a izolației îmbrăcămintei necesare pentru starea de confort termofiziologic, a fost calculat Indicele de Izolație termică a îmbrăcămintei (IREQ). Pe baza comparației cu nivelul utilizat al izolației termice a îmbrăcămintei (2,5 clo), au fost determinați indicele de expunere cu durată limitată (DLE) și timpul de recuperare (RT). Diferite condiții ale mediului cald, în care lucrătorii trebuiau să se recupereze din cauza expunerii la rece, au fost, de asemenea, testate: temperatura mediului ambiant, izolația termică a îmbrăcămintei și activitatea. Rezultatele obținute sugerează strategii de gestionare a expunerii la rece a lucrătorilor din industria alimentară, în legătură cu activitatea, izolația termică a îmbrăcămintei și condițiile de mediu.

Cuvinte-cheie : izolația termică a îmbrăcămintei, confort termofiziologic, mediu rece artificial

The effect of clothing insulation on the thermophysiological comfort of workers in artificial cold environment

The present study deals with the effect of the clothing insulation on the thermophysiological comfort of workers in artificial cold environment. Different types of artificial cold environment, related to food industry, have been identified, together with the activity of the workers. For the assessment of the cold exposure and the clothing insulation needed for the state of thermophysiological comfort, the Required Clothing Insulation (IREQ) index was calculated. On the basis of comparison with the used level of clothing insulation (2.5 clo), the Duration Limited Exposure (DLE) index and Recovery Time (RT) were determined. Different conditions of the warm environment, where the workers had to recover from the cold exposure, were also tested: temperature of the environment, clothing insulation and activity. The results obtained suggest strategies for management of the cold exposure of workers in the food industry, related to their activity, clothing insulation and environmental conditions.

Keywords: clothing insulation, thermophysiological comfort, artificial cold

INTRODUCTION

There are many working places where the work is performed in artificial cold environment: manual operations and storage of raw materials, processing of food in cooling chambers of warehouses, air explosive freezing of raw materials in the manufacture of frozen products, etc. The study of has shown that much more workers, than supposed, are occupied in artificial cold environment: in food processing industry, freezers, cold departments of supermarkets, etc. [1]

The work in natural and artificial cold has very similar features: in both cases the impact of cold could be dangerous for the human body. Therefore, workers must be provided with cold protective clothing. However, there are also specific characteristics of the occupation in artificial cold environment:

- The artificial cold environment is more stable in temperature, air velocity and humidity fluctuations, which helps the proper selection of protective clothing. In natural cold environment the temperature changes during the 24-hour period, which requires variation in the protective clothing's insulation.
- Continuous cold air flows in the artificial cold facilities lead to appearance of body temperature asymmetry (asymmetric cooling) and faster heat loses due to forced convection.
- The workers in artificial cold environment move more frequently between cold and warmer environment, which provokes higher strain on the thermoregulation system of the body.
- Work in a sitting posture or static work is more frequently observed in artificial cold, which is particularly harmful for the body.
- The clothing for artificial cold environment may be required to have additional protective features: i.e. against chemical hazards, mechanical hazards, etc., because of occupational safety regulations.

One of the approaches to assess the cold stress in cold environment is to assess the wind-chill temperature [2]. The wind-chill temperature is much more appropriate to be used in outdoor environment, however; though there are some exceptions, associated with artificial cold environment (i.e. large tunnel freezers with fast moving cold air). The Required Clothing Insulation (IREQ) index is very suitable for the assessment of the activities in artificial cold environment, as

Table 1

COLD RELATED WORK PLACES IN FOOD INDUSTRY			
Type of coldenvironment	**Temperature, ºC**	**Humidity, %**	**Application**
Cooling warehouse	0	80 ÷ 85	meat, poultry, fruits, vegetables
Freezer warehouse	–18 ÷ –23	85 ÷ 90	meat, poultry, frozen eggs, vegetables
Freezer warehouse	–20 ÷ –30	90 ÷ 95	fish, sea food, ice cream
Refrigerated warehouse for cooled food	12	85 ÷ 90	bananas
Refrigerated warehouse for cooled food	0 ÷ 8	85 ÷ 90	fruits, vegetables
Refrigerated warehouse for cooled food	–2 ÷ 0	80÷85	fresh eggs
Refrigerated warehouse for cooled food	–4 ÷ –10	85 ÷ 90	ice cubes
Refrigerated warehouse for cooled food	–15 ÷ –20	85 ÷ 90	frozen meat, frozen poultry, frozen fruits, frozen vegetables
Refrigerated warehouse for cooled food	–18 ÷ –23	85 ÷ 90	frozen fish, frozen sea food

it presents a method for assessment of the clothing insulation, needed to protect the human body in low temperatures. IREQ was developed by Holmér and included in the ISO Technical Report [3–4]. Later on it was proposed in ISO 11079 as a tool for designing and management of the activities in cold environment [5].

IREQ index is used to assess the cold stress of the human body on two levels: neutral and minimal [5]. The neutral value of IREQ corresponds to the thermophysiological comfort of the human body, when a thermal equilibrium between the generated by the body heat and the heat losses to the environment exists [6]. The minimal value of IREQ corresponds to the situation when cold strain already appears, the body is constantly cooling, but the thermoregulatory system of the body still can react and maintain the core body temperature within the desired limits.

Several researchers have investigated different problems related to thermal comfort or contributed to the development and application of the IREQ index [7–16]. The main reason is that extreme temperature of the environment can provoke injuries, decrease the working performance and create overall dissatisfaction [17]. The Unsafe Behaviour Index (UBI), which gives a relationship between the workers behavior and the thermal environment, was defined in [18]. It was found that the risk behavior is minimal within the temperature interval 17–23°C, while beyond these limits the proportion of unsafe workers' behaviour increases. Obviously, the cold artificial environment is a risk factor, and appropriate clothing must be used to decrease its negative influence on workers' performance and health, as well as on workers efficiency at the workplace.

The aim of the present study was to identify cold-exposed work places, related in general to food industry (food production, storage and selling) and to assess the working environment from the point of view of the clothing insulation of the protective ensembles used, the performed activity, the exposure to the cold environment, and the time and conditions needed for recovering of the body from the cold strain. The performed analysis gives recommendations for the adequate protective clothing insulation and recovery for different working loads and exposure times. The results obtained suggest strategies for management of the cold exposure of workers in the food industry, related to their activity, clothing insulation and environmental conditions.

EXPERIMENTAL WORK

Identification of artificial cold environment in food industry

During a preliminary survey on the work-places in food industry different types of artificial cold environment have been identified, dependent on the foods to be processes and the stage of their storage [19–23]. The cold-related work-places and the characteristics of the cold environment (temperature and humidity) are summarized in table 1.

Identification of activity level

Four levels of activity were identified in the cold working environment for food processing and storage: standing, standing with light activity, slow walking, and fast walking. The metabolic rates per activity, taken from ISO 8996:2004 are summarized in table 2 [24].

Table 2

METABOLIC RATES PER ACTIVITY IN THE ARTIFICIAL COLD ENVIRONMENT		
Metabolic rate		**Activity**
W/m^2	met	
70	1.2	Standing, immobile
93	1.6	Light activity in upright position, electric car driving
110	1.9	Walking with 2 km/h
200	3.4	Walking with 5 km/h

Clothing insulation

The literature survey has shown that there is no systematic study on clothing insulation levels of protective clothing, used in the artificial cold environment of different types of refrigerated warehouses, cooling warehouses and freezer warehouses. However, there are studies on the work in cold environment (both artificial and natural), which deal with the clothing insulation levels [7–9, 16–17]. It has been found that a clothing insulation of up to 1.5 clo is carefully selected by the workers, exposed to artificial cold (from –30°C to +10°C), while for temperatures of –20°C and below it is difficult for the workers to increase the clothing insulation if it is higher than 2.0 clo [7].

The study has reported three different clothing insulation values, used for protection of workers in the petroleum industry: from 1.7 to 2.6 clo, needed for exposure in outdoor cold environment from 0 °C to –34°C [25]. The working conditions in several Portuguese supermarkets were investigated through IREQ analysis, field studies and questionnaire studies [16]. It was reported an average value of the protective clothing insulation between 1.02 and 1.55 clo, for a temperature interval of the working conditions from –17.4°C (freezing chamber) to +12°C (food processing workplaces).

A clothing ensemble for cold protection with an insulation value Icl of 2.5 clo was selected to perform the present analysis. The thermal insulation was intentionally chosen to be on the upper possible limit (from ergonomic point of view). The idea was to overcome the wide-spread understanding to suggest clothing with higher insulation when the temperature of the working environment is low, as in many cases even the most insulating ensembles cannot guarantee thermophysiological comfort of the worker. Therefore, when the increment of the clothing insulation is exhausted, other strategies for ensuring the human thermophysiological comfort have to be applied, which will be shown below. The selected ensemble was a composition between a clothing ensemble for outdoor and indoor activities [16, 25]. It was selected to include underwear (underpants and a long sleeves shirt) from wool with Lenzing FR® cellulose fibers (50/50%); 3-layers overall with GORE-TEX® membrane, highly insulated jacket with GORE-TEX® membrane, calf-length woolen socks, hard-soled shoes, polar (polyethylene terephthalate) fleece gloves and a hat.

Calculation of IREQ, DLE, RT

The protocol for calculation of the Required Clothing Insulation (IREQ), given in ISO 11079, was applied to build a software code, using Pascal programming language [5]. The simulation of the IREQ index required the following initial and boundary conditions to be used:

- air temperature, air humidity;
- air velocity;
- activity level (metabolic heat production);
- thermal insulation of the clothing ensemble used.

The *IREQ* index was calculated using eq. (1):

$$IREQ = \frac{t_{sk} - t_{cl}}{R + C} \qquad (1)$$

where t_{sk} is the mean skin temperature, °C; t_{cl} is the temperature of the clothing surface, °C; *R* are the heat loses due to radiation, W/m^2; *C* are the heat loses due to convection, W/m^2, and *S* is the body heat storage rate, W/m^2.

Since both *IREQ* index and the temperature of the clothing surface are unknown, eq. (1) was solved together with the general balance eq. (2) using iterations:

$$M - W - E_{res} - C_{res} - E - K - R - C - S = 0 \qquad (2)$$

where *M* is the metabolic rate, W/m^2; *W* is the effective mechanical power, W/m^2; E_{res} are the heat losses due to respiration, W/m^2; C_{res} are the respiratory convective heat losses, W/m^2; *E* are the heat losses due to evaporation, W/m^2; *K* are the heat losses due to conduction, W/m^2.

Two values of *IREQ* were calculated: $IREQ_{neutral}$, which corresponds to the state of thermophysiological comfort of the person, and $IREQ_{min}$, which is related to all other cases when the clothing still can protect the body, but it is already in a state of continuous cooling.

Eleven temperature regimes were identified, following the working conditions in food warehouses, described in table 1: from +12°C to –28°C with a step of 4°C. The relative humidity was set to 85% for all cases. The two values $IREQ_{neutral}$ and $IREQ_{min}$ were compared with the thermal insulation of the clothing ensemble (2.5 clo) and the Duration of Limited Exposure to cold environment (DLE index) was determined.

DLE index was calculated using eq. (3):

$$DLE = \frac{Q_{lim}}{S} \qquad (3)$$

where Q_{lim} is the limit value of body heat gain or loss, kJ/m^2, and *S* is calculated from eq. (1).

Two values of *DLE* index were calculated: DLE_{min}, which corresponded to $IREQ_{min}$, and DLE_{max}, which corresponded to $IREQ_{neutral}$. When the predicted exposure time to cold environment was less than one working shift (*DLE* < 8 hours), than Recovery Time (RT index) was predicted.

RT index was used to assess the needed environmental conditions and period for rest, so as the workers to be able to continue their work in the cold environment without a danger from cold injuries and appearance of negative behavior. *RT* was calculated using eq. (3), substituting the conditions of the cold environment with the conditions for resting in warmer environment:

$$RT = \frac{Q_{lim}}{S} \qquad (4)$$

DLE and *RT* were calculated following the mathematical model and protocol, given in ISO 11079 [5].

RESULTS AND DISCUSSIONS

Figure 1 presents the results from calculation of the Required Clothing Insulation for the respective temperature and humidity of the artificial cold environment

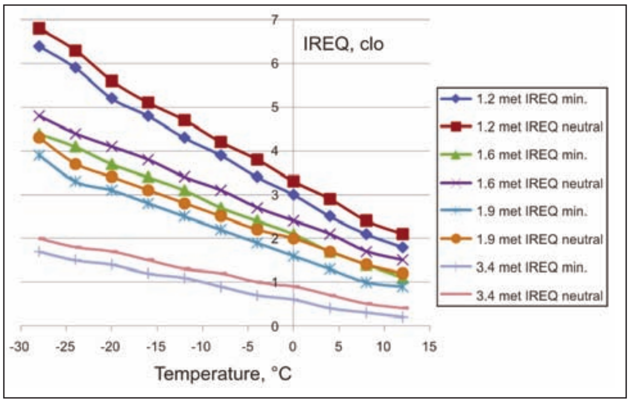

Fig. 1. Required clothing insulation for working in temperature interval (+12°C ÷ –28°C), four levels of activity

(see table 1) and 4 levels of activity (see table 2). *$IREQ_{min}$*, and *$IREQ_{neutral}$* were determined for each case of cold exposure.

The results show that as lower the temperature and the metabolic values are, as higher the required insulation of the clothing ensemble must be. The thermophysiological comfort ($IREQ_{neutral}$) of a sitting person with the selected protective clothing will be guaranteed for up to 8°C. In colder environment (4°C) a cooling of the body will appear ($IREQ_{neutral} > I_{cl}$). If the person has to continue his work in the environment of 4°C, three strategies for management of the cold exposure of the worker are possible:

- additional clothing item must be added so as to increase the clothing insulation Icl to 2.9 clo (to reach $IREQ_{neutral}$) or
- the exposure in the cold environment to be reduced from 8 hours to limited exposure time (*DLE* index must be calculated) or
- the metabolic activity has to increase.

The increment of the metabolic rate from standing (70 W/m^2) to light activity in upright position (93 W/m^2) guarantees the thermophysiological comfort of the worker in 0°C, up to –4°C, when a cooling of the body will start. The new increment of the activity through slow walking (110 W/m^2) will allow the worker to remain in thermophysiological comfort in cold environment of up to –8°C. The cooling of the body, which starts at –8°C, will require more heat to be produced by the body through activity (muscle work). The last tested metabolic rate of 200 W/m^2 (fast walking in the cold environment) allows the human body to be in a thermophysiological comfort in all investigated regimes of cold environment (see table 1), up to –28°C (in freezer warehouses).

The analysis of the results from the calculation of the *IREQ* index shows that the work in standing, immobile position (i.e. monitoring of a process) must be avoided in the cold environment. The standing posture has to be combined at least with light activity or slow walking. Fast walking is advisable for workers who enter periodically the warehouse. It is also advisable for workers in freezer warehouses where the temperature is below –20°C.

Figure 2 summarizes the results from the calculation of Duration Limited Exposure index (DLE). Both the minimum DLE_{min} and the maximum DLE_{max} time of exposure in the cold environment were calculated, taking into account the activity. The results show that the activity strongly influences the effect of the cold exposure, when the same clothing insulation level Icl is used: in a standing position the generated heat from the body is able to ensure the thermophysiological comfort during full working shift (8 h) in a cold environment with a temperature of up to +4°C. The generated heat during slow walking can ensure continuous work (8 h) in a temperature of –8°C. If the worker walks with 5 km/h, then 2.5 clo is enough to ensure the thermophysiological comfort of the body even in freezer warehouses with temperature of –28°C.

The analysis of the results in figure 2 shows that if the insulation of the clothing ensemble cannot be increased (as in this case), due, for example, of awkward movements or other ergonomic reasons, than the strategy for management of the cold exposure is to increase the activity in the cold work-place. However, the activity may provoke sweating, which lead to augmentation of the heat loses by evaporation. This would negatively influence the thermophysiological comfort of the worker, due to appearance of faster cooling. Therefore the combination between the clothing insulation and the activity must be carefully selected and a feedback from the workers must be received (i.e. via questionnaire studies for thermal comfort evaluation).

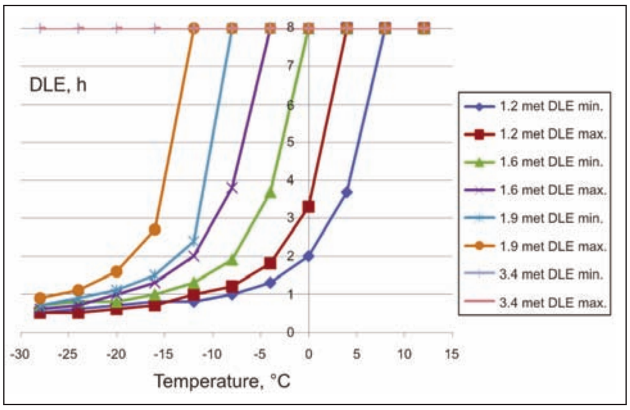

Fig. 2. Duration of limited exposure for working in temperature interval (+12°C ÷ –28°C), four levels of activity

If the clothing insulation is not high enough to protect the human body from cold and to ensure the thermophysiological comfort, the strategy for management of the cold exposure is to interrupt the exposure in the cold and a period for recovery of the body in warm indoor environment to be secured. ISO 11079 does not deal with the conditions for recovery of the body from the cold exposure, however [5].

The present study tested different cases of "warm exposure" during the recovery period from the cold. The temperature of the warm environment was increased from 18°C to 30°C, with a step of 2°C (the

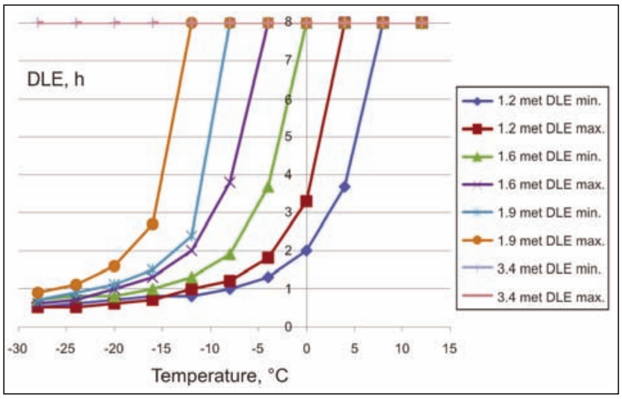

Fig. 3. Recovery time in temperature interval (18°C ÷ 30°C), two levels of activity, two levels of clothing insulation

relative humidity of the environment was kept constant, 60%). Two body postures were considered during resting: seated relaxed (1 met, 58 W/m^2) and standing relaxed (1.2 met, 70 W/m^2). Two values of clothing insulation were also used for the calculations: 2.5 clo, assuming that during the rest period the worker stays with the whole cold protective ensemble, and 1 clo, assuming that the person undresses. The results from the calculations of the Recovery Time (RT) are summarized in figure 3.

The smaller period for recovery of the body from the cold exposure is 42 min in a warm environment with temperature of 30°C, when the worker is standing in a relaxed posture, without taking-off part of the clothing ensemble. In case the worker takes-off his hat, gloves, overall and jacket during resting, then the temperature of the environment of the resting place (room) has to be over 24°C, if the person rests seated, and at least 24°C, if the person remains standing (the values for the heat storage are negative for lower temperatures of the warm environment) [5]. Although the insulation is reduced when the cold protective clothing is removed, there are some benefits to do so, for example the trapped moisture inside the clothing ensembles can be evaporated during recovery period.

Obviously, the recommendation of ISO 11079 for recovery from the cold exposure in “warm environment” is not enough [5]. The calculation of RT shows that the fast recovery appears if the workers rest with the whole clothing ensemble and do not seat. The decrement of the clothing insulation during rest requires higher temperatures of the warm indoor environment to be provided, which is related with more energy for the heating of the resting room.

CONCLUSIONS

There were significant differences in the Required Clothing Insulation (IREQ) for different cold-exposed work places in food production and food storage industry. The level of cold stress increased with the temperature drop and the decrement of activity. The results for IREQ showed that the increase of clothing insulation was not the possible management solution for workers in freezers, as the required insulation calculated had the impossible values of 5 ÷ 8 clo. The only solution could be increased activity and limited exposure, as well as regular change of the working place from cold to much warmer environment.

The results for the Recovery Time (RT) showed that in warmer resting rooms, organized for recovery of the workers from cold environment, the workers must keep their clothes on for fast recovery. The workers had to be involved in activities, which could keep them moving (playing darts, doing light exercises, dancing). This would stimulate the heat production in the muscles and warm the body faster.

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Photocatlytic nanomaterials based on doped TiO_2 for leather garments and upholstery with self-cleaning properties

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REZUMAT – ABSTRACT

Nanomateriale fotocatalitice pe bază de TiO_2 dopat pentru articole de îmbrăcăminte din piele şi tapiţerie cu proprietăţi de auto-curăţare

În ultimii ani a existat o preocupare intensă pentru dezvoltarea, promovarea și comercializarea produselor destinate acoperirilor fotocatalitice, în special pe bază de TiO_2, cu impact minim asupra mediului și cu efecte benefice asupra calității vieții. Cererea tot mai mare pentru tapițerie de mobilă, automobile și articole de îmbrăcăminte din piele în culori deschise (alb, bej, gri) a stimulat producătorii să îmbunătățească proprietățile anti-murdărire ale polimerilor de finisare. Prin aplicarea tehnologiilor inovatoare pentru finisarea pieilor cu compozite polimerice care conțin fotocatalizatori pe bază de nanoparticule de TiO_2 dopate cu metale/nemetale se pot obține produse din piele cu proprietăți avansate de suprafață (auto-curățare, auto-sterilizare, anti-ceață, rezistenţă la foc). În lucrarea de față s-au sintetizat nanoparticule de TiO_2 dopate cu diferite elemente care au arătat o îmbunătățire a descompunerii fotocatalitice a modelului de poluanți organici cum ar fi Orange II, albastru de metilen sau pastă de pix în soluție sau pe suprafața pieilor expuse la iradiere UV și/sau Vis.

Caracterizarea nanoparticulelor fotocatalitice și proprietăților de suprafață ale pielii s-a realizat prin teste de fotodegradare, microscopie electronică (SEM), spectroscopie de raze X (EDX), spectre de reflexie difuză (DLS), împrăștierea dinamică a luminii (DLS) și determinări ale unghiului de contact. Utilizarea nanoparticulelor de TiO_2 dopat, pe suprafața de piele, generează proprietăți de respingere a murdăriei prin creșterea valorilor unghiului de contact și de auto-curățare sub influența radiațiilor UV-Vis.

Cuvinte-cheie: piei tapițerie și îmbrăcăminte, finisări de suprafață, proprietăți de auto-curățare, acoperiri fotocatalitice

Photocatlytic nanomaterials based on doped TiO_2 for leather garments and upholstery with self-cleaning properties

In the recent years there has been a growing concern in the development, promotion and marketing of products for photocatalytic coatings, especially based on TiO_2, with minimum environmental impact and beneficial effects on quality of life. The increasing demand for light colors (white, beige, grey) in upholstery, automotive and garment leathers has spurred the producers to improve the anti-soiling properties of finishing polymers.

By applying innovative technologies for leather finishing with composite polymers containing photocatalysts based on TiO_2 nanoparticles doped with metals/non-metals one can obtain leather products with advanced surface properties (self-cleaning, self-sterilization, antifogging, fire resistance). In our work there were synthesized TiO_2 nanoparticles doped with different elements which have shown an improvement of the photocatalytic decomposition of organic pollutant models such as Orange II, Methylene Blue or ball pen ink in solution or on leather surface exposed to UV and/or Vis radiations.

Photocatalytic nanoparticles and leather surface properties were characterised by photodegradation tests, electron microscopy (SEM), X-ray spectroscopy (EDX), UV-Vis diffuse reflectance (DRS), dynamic light scattering (DLS) and contact angle determinations. The use of doped TiO_2 nanoparticles on leather surface showed soil repellency through contact angle increased values and self-cleaning properties under UV-Vis radiation exposure.

Keywords: leather garments and upholstery, surface finishing, self-cleaning properties, photocatalytic coatings

INTRODUCTION

Titanium dioxide, which is one of the most basic materials in our daily life in microscale size, because of its physical and chemical stability, very high refractive index, has high catalytic activity, high oxidative power in nano state and has emerged as an excellent photocatalyst material for environmental purification. Photocatalytic oxidation of various harmful pollutants over TiO_2 and ZnO semiconductor oxides under UV light irradiation is already known [1]. The UV dependence (because of its wide band gap of 3.2 eV) and high rate of electron–hole recombination in TiO_2 nanoparticles (TiO_2NPs) have been addressed as the main drawbacks for its practical applications [2]. Research is now focused on achieving high photocatalytic efficiency under visible light, taking into account that sunlight consists of about 5–7% UV light, 46% visible light and the rest is infrared radiation, and is very important to use the vast potential of solar photocatalysis [3]. Various techniques have been employed to make them absorb photons of lower energy as well, and among these there are metal doping and co-doping of metals and non-metals [3–7]. Doped TiO_2-based photocatalyst can be used like photocatalytic coatings to decompose surface organic dirt, creating self-cleaning surfaces [8–10]. Moreover, TiO_2NPs have antibacterial and antifungal properties, conferring a self-sterilizing effect to finished surfaces. Nowadays, nanoparticles have a large application for surface functionalization in order to provide different properties [11–12].

There is a high interest for leather finishing in white or light colors with anti-soiling properties and the progresses in this area are related to the use of silicon, fluoropolyethers or polyurethane dispersions based on polyester/polycarbonate diols [13–14]. The main applications of these new materials are for upholstery, automotive and garment leathers.

The present paper presents innovative nanomaterials for development of self-cleaning properties on leather surface with beneficial effect on items durability and solvent pollution reduction.

EXPERIMENTAL

Materials and Methods

The investigations were carried out using the following materials:

- TiO_2 powder having an anatase phase and a content of min. 99.5% TiO_2 was obtained from Metall Rare Earth ltd (China);
- deionized water with conductivity < 1 μS, resistivity of 18 μΩ·cm and pH = 5–7;
- urea (CH_4ON_2), 98% purity from Chimopar;
- sodium polyacrylate (Na-PAA) with Mw of 2100 from Sigma – Aldrich;
- $FeCl_3$, $CoCl_2$ were purchased from Sigma – Aldrich;
- Orange II sodium salt, methylene blue (C16H18ClN3S.3H2O) from Fluka;
- natural leathers processed in INCDTP-ICPI Division.

Doping of TiO_2 with N was performed using urea and a thermal procedure: TiO_2 and urea, in a molar ratio of 1:2 are mixed in an agate mortar and calcined at 450°C temperature for 4h, for solid phase reaction, resulting in a light yellow powder (TiO_2 doped with N) [15]. Using a chemical method, N doped TiO_2 powder was co-doped with Fe and Co.

In order to test leather surface properties, several leather samples were functionalized with TiO_2 based nanoparticles by covering their surfaces with colloidal dispersions embedded in film forming polymers.

Characterization of TiO_2 nanoparticles and their photoactivity on leather surface

Particle size and stability of TiO_2 nanoparticles in aqueous dispersions

Particle size and Zeta potential were determined using dynamic light scattering (DLS) technique before doping and co-doping in 1% dispersions of TiO_2 with 10%Na-PAA against TiO_2 content by using Zetasizer Nano ZS, Malvern.

Photocatalytic testing of TiO_2 nanoparticles

Photocatalytic activity of doped and undoped TiO_2NPs was evaluated by investigating degradation of an azo dye, namely Orange II (OII) with λmax = = 484 nm as model pollutants by using a UV lamp type VL 204 with irradiation at 365 nm. The absorbance spectra of the dye were determined (UV-Vis spectrophotometer Jasco V570) and in order to check the applicability of the Lambert-Beer law for the dye solutions, calibration lines were experimentally plotted (figure 1).

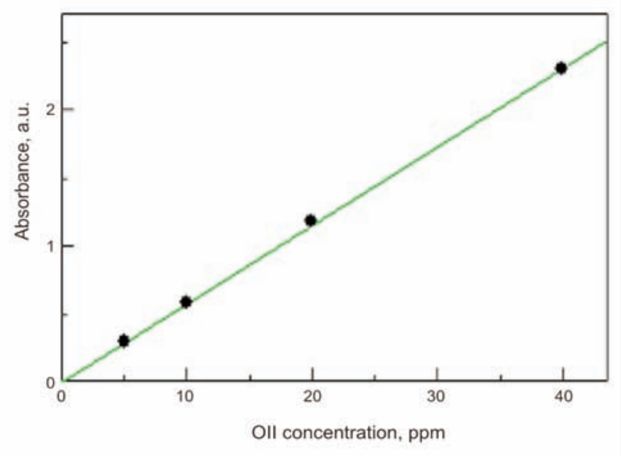


Fig. 1. Calibration line of OII dye

Kinetics of photodegradation of OII was also recorded. The experiments were carried out in a Berzelius glass with 50 mL sample and above this, at a 15 cm distance, a UV lamp was placed. The catalyst loading was 0.1 g/L. The suspension was first stirred in the dark for 30 min. to reach equilibrium sorption of the dye and after that UV illumination was started. Aliquot samples of 3 mL were taken at determined time intervals (1 h) and filtered through 0.1 μm membrane filters to remove the TiO_2 particles. To ensure a constant transfer rate of the OII to the catalyst and a uniform illumination of TiO_2 and TiO_2 doped nanoparticles, magnetic stirring was used.

UV-Vis diffuse reflectance (DRS) spectra were made on undoped and doped powders of TiO_2NPs using a JASCO V 570 spectrophotometer, equipped with integrating sphere accessory.

Leather surface preparation and characterization

After testing the photocatalytic properties of undoped, doped and co-doped TiO_2 nanoparticles, the sample with the highest photocatalytic activity was used for leather surface finishing. The procedure consisted in ultrasound mixing of powder nanoparticles in base coat polymer finishing solutions with Na-PAA and then spraying it on the leather surface.

The leather samples, treated and untreated (control) with selected nanoparticles, were analyzed by SEM, EDX (Quanta FEI 200) techniques and contact angle (VGA, Optima XE) measurements.

The leather samples were stained with ball pen ink, OII and methylene blue (MB) dyes and irradiated using a 500 W halogen Vis lamp and an UV lamp at 365 nm. The discoloration of model stains was recorded by photography up to 360 hours of exposure.

RESULTS

TiO_2 nanoparticles dispersion stabilized with Na-PAA was analysed by using DLS technique before doping and co-doping and the results show that nanoparticles average size is 12 nm (figure 2).

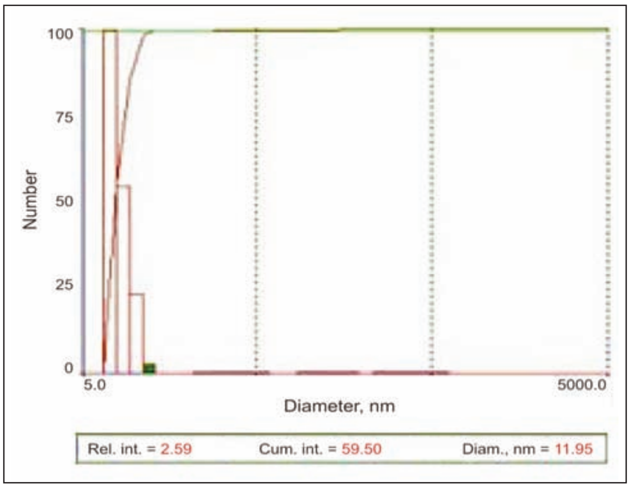


Fig. 2. Histogram of a 1% TiO_2/Na-PAA dispersion

Zeta Potential was used as a stability indicator for TiO_2 nanoparticles in dispersion. If all TiO_2 species present in dispersion have high Zeta potential values, either negative or positive outside the range –30 ÷ +30 mV, they tend to repel each other and to avoid aggregation. Therefore, Zeta potential values of 45 mV presented in figure 3 showed that the dispersion has a high stability.

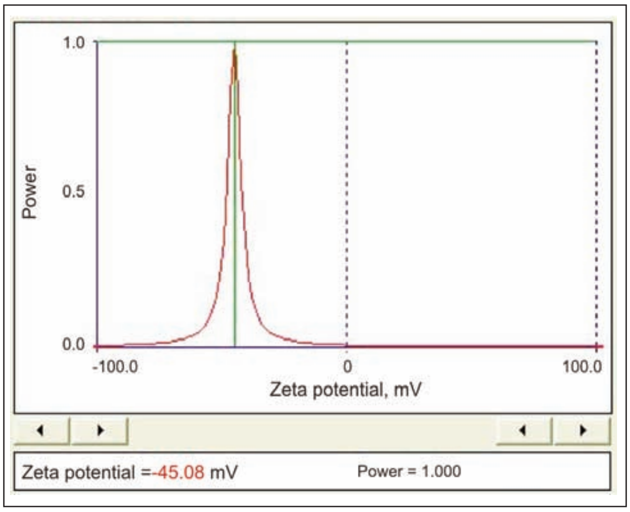


Fig. 3. Zeta Potential for TiO_2 nanoparticles dispersion

Table 1

UV-VIS ABSORBANCE OF OII SOLUTIONS WITH TiO_2 BASED NANOPARTICLES AFTER 30 MIN OF IRRADIATION	
Nanoparticles	**Absorbance**
TiO_2	0.36
N-TiO_2	0.44
Fe-TiO_2	0.08
Fe-N-TiO_2	0.04
Co-TiO_2	0.54

To evidence photocatalytic degradation for a 20 ppm OII solution, in the presence of 0.1 g/L TiO_2 and doped and codoped-TiO_2, absorbance spectra under UV irradiation ($\lambda = 365$ nm) were recorded (table 1).

Knowing that the initial value for the absorbance of OII was around 1 one can observe that the best value was obtained for Fe-N-TiO_2 (TiO_2 doped with N and co-doped with Fe) nanoparticles.

In order to have more information about the reactions of the photocatalyst materials with photon energies, UV-Vis diffuse reflectance spectra were made.

Figure 4 presents diffuse reflectance spectra of doped TiO_2 comparative with undoped TiO_2.

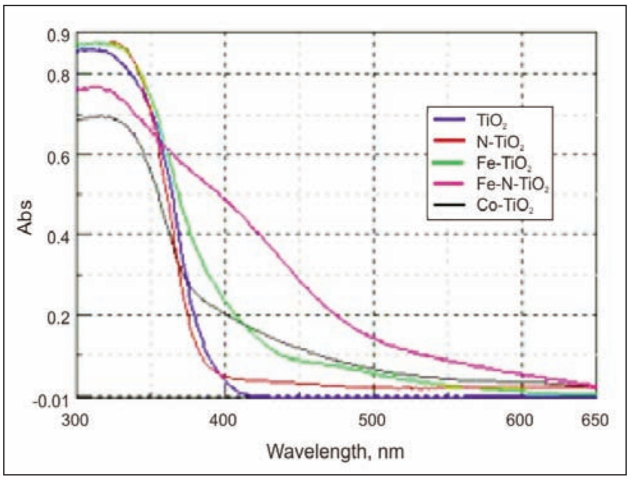


Fig. 4. Diffuse reflectance spectra for nano TiO_2 with different doping elements

Figure 4 shows a significant absorption shift in the visible domain for Fe-TiO_2 powder and also an increase for N-TiO_2 powder co-doped with Fe. Cobalt cation showed an appreciable increase of TiO_2 absorption in the visible domain.

SEM (figure 5) and EDX (figure 6) characterizations show the morphology of leather surface and the presence of Fe-N-TiO_2 nanoparticles (1.15% Wt% of Fe) on leather samples surface.

From contact angle determination results (table 2) one can see that the samples become hydrophobic after having been treated with Fe-N-TiO_2 nanoparticles. These results show soil repellency property of leather surface finished with the use of Fe-N-TiO_2 nanoparticles.

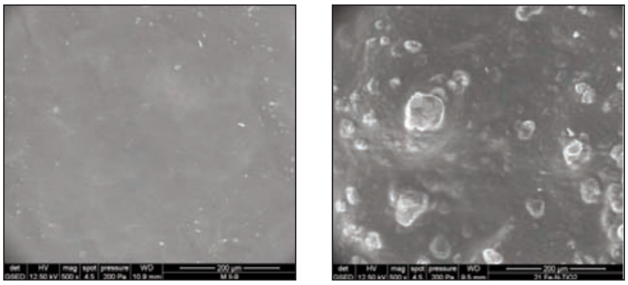
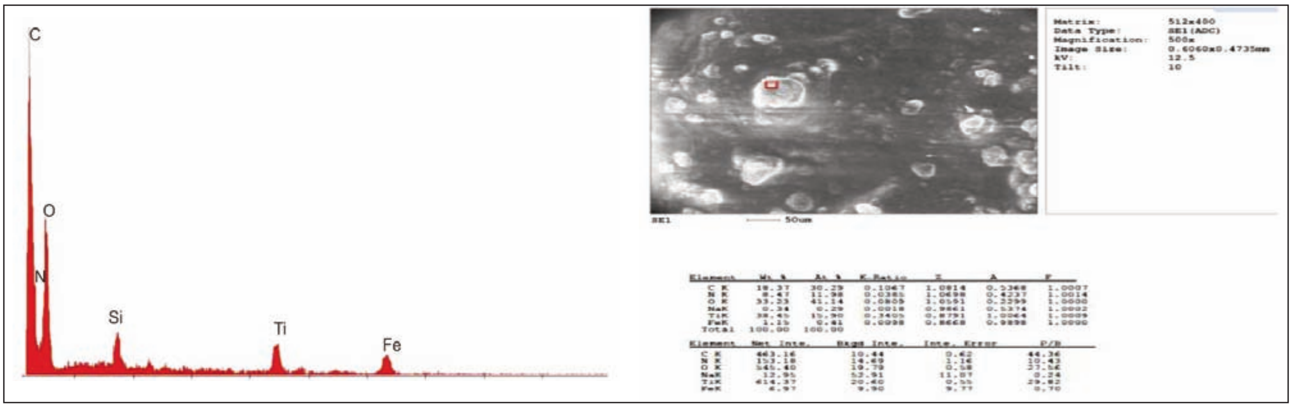


Fig. 5. SEM images (500x) before (left) and after (right) finishing leather surface with Fe-N-TiO_2

Table 2

CONTACT ANGLE FOR LEATHER SURFACE UNTREATED AND TREATED WITH FE-N-TIO_2 NPS		
Leather sample	**Contact angle, °**	
	untreated	**treated**
Fe-N-TiO_2-1	70	96
Fe-N-TiO_2-2	52	85
Fe-N-TiO_2-3	63	92



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Matrix:            512x400
Data Type:         SE1(ADC)
Magnification:     500x
Image Size:        0.6060x0.4735mm
kV:                12.5
Tilt:              10
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Element   Wt %    At %    K-Ratio    Z        A        F
 C K      18.37   30.29   0.1067    1.0814   0.5368   1.0007
 N K       8.47   11.98   0.0385    1.0698   0.4237   1.0014
 O K      33.23   41.14   0.0805    1.0591   0.2299   1.0000
 NaK       0.34    0.29   0.0018    0.9861   0.5374   1.0002
 TiK      38.45   15.90   0.3405    0.8791   1.0064   1.0009
 FeK       1.15    0.41   0.0098    0.8668   0.9898   1.0000
Total    100.00  100.00
Element  Net Inte.    Bkgd Inte.    Inte. Error    P/B
 C K      463.16        10.44          0.62        44.36
 N K      153.18        14.69          1.16        10.43
 O K      545.40        19.79          0.58        27.56
 NaK       12.95        52.91         11.87         0.24
 TiK      414.37        20.40          0.55        29.82
 FeK        6.97         9.90          9.77         0.70
```

Fig. 6. EDX spectrum for leather surface covered with Fe-N-TiO_2 NPs

Figure 7 shows the photocatalytic effect of Fe-N-TiO_2 nanoparticles over leather samples stained with ball pen ink and MB, before and after exposure to Vis and UV irradiation for up to 60 hours. It can be observed that the blue line and MB stain become colourless after irradiation with Vis light after 20 hours.

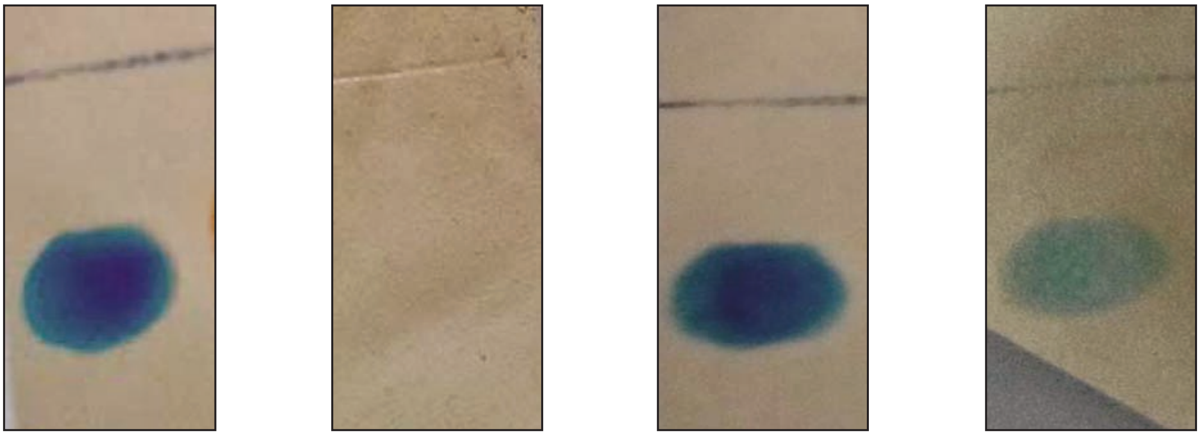
From the figure 8 it can be seen that the control sample (leather covered with film forming polymers with microsize TiO_2 commercial pigments) does not have photocatalytic properties even after 120 hours of exposure.

The stains of Orange II on leather surface with higher resistance to decomposition due to the benzensulphonate stable structure as compared to heterocyclic structure of methylene blue proved to be decomposed under visible light after 360 hours of exposure (figure 9a) and was stable under UV exposure (figure 9c).

Doping of TiO_2NPs with iron proved to enhance the efficiency of self-cleaning properties under visible light against OII, MB and ball pen ink stains.

CONCLUSIONS

The new doped nanoparticles with improved photocatalytic properties for leather finishing with self-cleaning properties were developed. TiO_2 dispersions



a *b* *c* *d*

Fig. 7. Model stains degradation (MB and ball pen ink) onto a treated leather surface with nano Fe-N-$TiO_{2,}$ under **Vis light**: ***a*** – initially; ***b*** – after 20 h of exposure and under **UV light**: ***c*** – initially; ***d*** – after 60 h of exposure

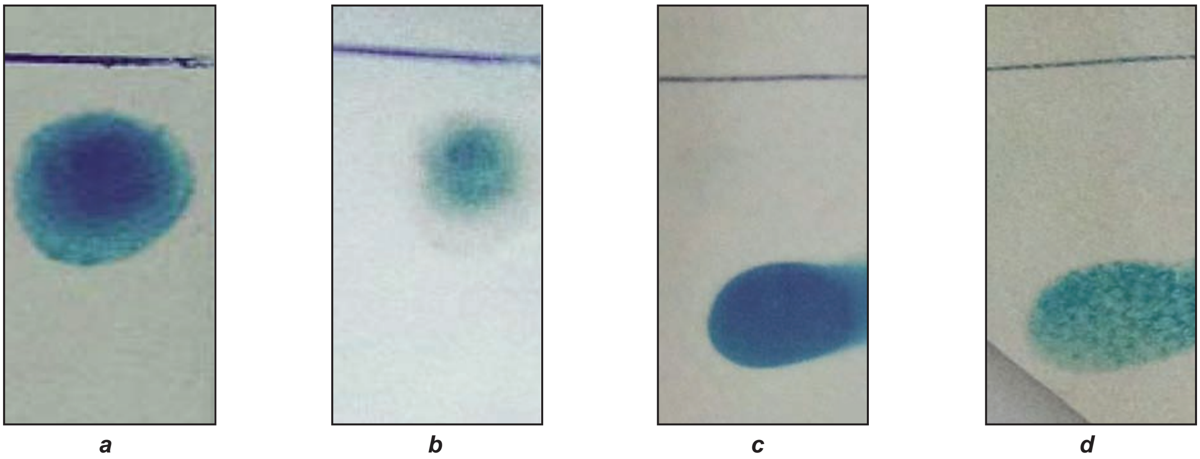


Fig. 8. Model stains (MB and ball pen ink) degradation onto untreated leather under **Vis light**: ***a*** – initially; ***b*** – after 120 h of exposure and under **UV light**: ***c*** – initially; ***d*** – after 120 h of exposure

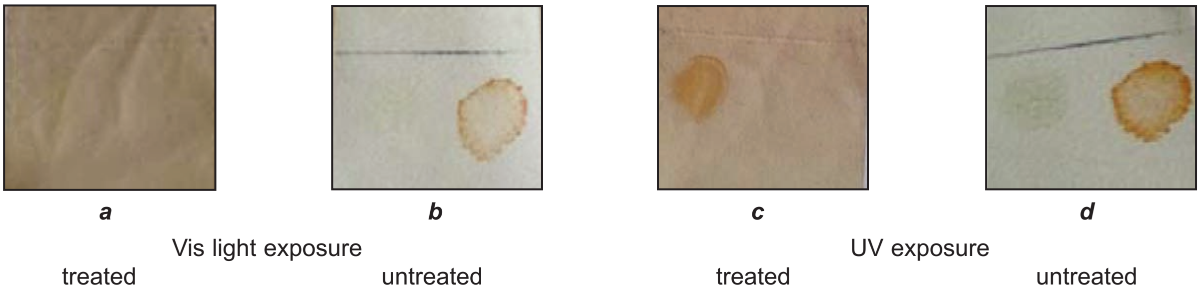


Fig. 9. Model stains (OII) degradation onto treated leather with nano Fe-N-TiO_2 (***a, c***) and untreated leather surface (***b, d***) after 360 h of **Vis** exposure (***a, b***) and **UV** exposure (***c, d***)

characterized by dynamic light scattering technique indicate that these are very stable and well dispersed. The shifted photocatalytic activity in visible domain, as function of the type of doping element, was evidenced by diffuse reflectance spectra due to the type of doping element. The Fe-N-TiO_2 nanoparticles showed organic soil photocatalytic decomposition activity on finished leather surface exposed for 20 hours to visible light. Contact angle measurements on treated leather surface showed the increase of hydrophobic properties with effect on soil repellency. The self-cleaning properties against ball pen ink, methylene blue and Orange II stains on leather surface exposed to UV-Vis radiation were demonstrated. The high interest for anti-soiling light colour leathers for upholstery and garment items opens a high area of application for photocatalytic nanoparticles with self-cleaning properties.

Acknowledgements

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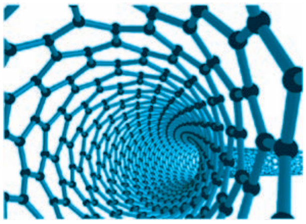

Research for accomplishing multifunctional textiles with plasma technology

LILIOARA SURDU
IOAN SURDU
IOAN RAZVAN RADULESCU

REZUMAT – ABSTRACT

Cercetări pentru realizarea de textile multifuncţionale prin tehnologia cu plasmă

Tratamentele în plasmă reprezintă soluții moderne utilizate în ultimii ani pentru a produce noi funcționalități materialelor textile. În această lucrare sunt prezentate efectele tratamentului în plasma de Hexafluoropropenă cu durată variabilă de tratare (de 0,10, 20, 30 și 90 minute) asupra proprietăților fizico-mecanice ale unui material textil țesut, tip CORTINA din 100% bumbac. Au fost analizate trei proprietăți fizico-mecanice ale materialelor textile crude și tratate în plasmă, respectiv: forța de rupere pe direcția urzelii, forța de rupere pe direcția bătăturii și rezistența la abraziune. Scopul studiului a fost de a determina durata optimă de tratare în plasmă a țesăturii CORTINA la care se obține valoarea maximă a forței de rupere, respectiv a rezistenței la abraziune. Determinarea duratei optime s-a făcut prin prelucrarea statistică și modelarea matematică a datelor experimentale, aplicându-se testele ANOVA şi posthoc Duncan pentru evidențierea efectului tratamentului în plasmă și stabilirea grupelor omogene în cazul fiecărei proprietăți testate pe epruvete individuale. Reprezentarea grafică a valorilor medii măsurate, respectiv a celor estimate prin regresie neliniară, oferă o imagine sintetică clară privind valoarea maximă pentru fiecare dintre cele trei proprietăți testate. Calculele analitice și reprezentările grafice au evidențiat faptul că o durată de 30–50 minute a tratamentului cu plasmă aplicat țesăturii de tip CORTINA produce îmbunătăţirea semnificativă a proprietăților fizico-mecanice analizate (de 0,53–0,61% pentru forța de rupere pe direcția urzelii și a bătăturii, respectiv de 4,03–5,53 % pentru rezistența la abraziune).

Cuvinte-cheie: ţesătură, adăposturi de urgenţă, plasmă, textile multifuncţionale

Research for accomplishing multifunctional textiles with plasma technology

Plasma treatments represent a modern solution for rendering new functionalities to textile materials. This paper presents the effect of Hexafluoropropane plasma treatment with variable process time (of 0, 10, 20, 0 and 90 minutes) upon the physical-mechanical properties of a 100% cotton woven fabric type CORTINA. Three physical-mechanical properties of the raw and plasma treated materials were analysed: the tensile strength on warp direction, the tensile strength on weft direction and the abrasion resistance. The aim of the study was to determine the optimal plasma treatment process time of the CORTINA woven fabric for the maximum value of the tensile strength and the abrasion resistance. The determination of the optimal process time was performed by means of statistical processing and mathematical modelling of the experimental data, with application of the tests ANOVA and posthoc Duncan, for evidencing the plasma treatment effect and establishing the homogenous groups in case of each property tested on individual samples. The graphical representation of the measured mean values and the values estimated by non-linear regression offer a clear synthetic image regarding the maximal value for each of the three tested properties. Analytical computation and graphical representation have evidenced that a process time of approx. 30-50 minutes of the plasma treatment applied to the fabric CORTINA produces the most significant improvement of the analysed physical-mechanical properties (cca. 0.53–0.61 % for the tensile strength on warp and weft direction and 4.03–5.53 % for the abrasion resistance).

Keywords: woven fabric, emergency shelters, plasma, multifunctional textiles

INTRODUCTION

Technical textiles are a field envisaged by the European strategy, and consist of textile materials with technical applications, in fields like: medicine, industry, safety, agriculture etc. This category of new high-added value textile products comprises the properties of textile materials (light-weightiness, flexibility, resistance) and offers adequate solutions for the specific technical purpose [1–5].

The treatment in plasma represents an innovative, alternative solution for the modification of the functional properties of the textile materials. The surface of the material is modified at microscopic level, during a dry process, which is eco-friendly and cost effective. This process is possible without auxiliary mechanical operations or application of chemical reactive substances. This technique is suitable for the modification of the surface properties, without the modification of the volume properties of textile materials. The resulting materials acquire new functionalities after these treatments, which allow new solutions for production and design and even the development of new applications [6–12].

The treatment in plasma represents also a solution for reducing the consumption of the three resources (chemical substances, water and energy).

The plasma technology is adequate for the modification of the chemical structure as well as of the topography of the surface of the textile material. The performed research in this field has shown that the special treatments performed by means of polymer grafting and coating improve the surface aspect and the tensile strength of the finished product [12–20].

The principle of plasma treatment consists in introducing the textile material in a highly reactive medium, which has as consequence the following effects:

- Structural modifications of the surface (cracks, roughness, micro craters);
- Compositional modifications through generation of radicals conducting to polymer grafting on surfaces.

The results of the plasma treatment are depending on the following treatment conditions:

- The installation and the characteristics of the generated plasma, the type of the plasma generator, the frequency of the generator, the plasma chamber, the type of plasma (primary or secondary), the treatment pressure, the composition of the plasma gases and the power.
- The treated material: structure, composition and presentation form.
- The type of treatment at low pressure or atmospheric pressure.
- The treatment time.

The experimental research based on plasma technology has been performed for evidencing the structural modifications and the properties of the textile material's surfaces. The tests have proved that plasma treatments can present the following actions:

- Improve the bonding of the deposited substances with the textile fibres and/or the adhesiveness capacity of the surfaces;
- Improve the anti-microbial properties of the surfaces;
- Increase the mechanical resistance of the textile material by means of reticulation;
- Modify the hydrophilic and the hydrophobic characteristics of the surfaces related to the necessities etc.

Hence, exceptional properties can be obtained by means of plasma treatment of textile materials. INCDTP-Bucharest has a plasma treatment laboratory and a CD 400 Roll-to-roll low-pressure plasma installation from Euro plasma Belgium (figure 1).

Plasma treatments on textile materials render various new properties, such as: hydrophobic/hydrophilic, anti-static, anti-felting, anti-bacterial etc. The previous research performed within INCDTP with plasma treatments tackled the following issues:

- performing of hydrophobic textile materials from cotton and polyester, destined for surgical gowns;
- performing of fabrics with anti-microbial character destined for textile medical articles (bandages, dressings);
- performing of wettability treatments of the fabrics for subsequent finishing.

The plasma treatment parameters are:

- treatment duration;
- type of gas: [e.g. Oxygen, Argon, Nitrogen, Hexafluoropropane];
- power: [20–300 W];
- pressure: 20 mTorr;
- frequency range of the generator: kHz/MHz.

Fig. 1. Laboratory plasma equipment in INCDTP

Related to the selected parameters, the plasma treatment renders a specific functionality to the textile surface.

The aim of the present research was to obtain the optimal plasma treatment duration for best values of the physical-mechanical tests: tensile strength on warp and weft direction, abrasion resistance, in order to highlight the improved mechanical resistance of the fabric. Mathematical modelling of the physical-mechanical properties on the treated samples was performed, in order to determine the optimal treatment duration.

EXPERIMENTAL WORK

Materials used

The plasma treatments were performed on a special fabric, destined for technical applications, especially shelters for emergency situations. INCDTP-Bucharest has research results in the field of technical fabrics for emergency shelters. It has recently received the Gold medal at the "Salon international des inventions" – Genève 2016, Switzerland. The awarded fabric relates to a poly-functional fabric for the production of shelters for campaign. The fabric has several functionalities due to special finishing treatments, characterized by a compact structure able to provide an adequate microclimate in the shelter for people and meets the conditions of mechanical resistance to dynamic stress, at working temperatures of up to 150°C.

Out of this fabric, the following end product is obtained (figure 2).

The fabric is named CORTINA: it is a 100% cotton woven fabric with weight of fabric between 400 and 600 g/m^2. The justification for the selection of the raw materials consists in its following specific characteristics for the cotton yarns:

Fig. 2. Shelter for emergency situations

- large variety of types and linear density;
- low cost;
- good process-ability;
- may be subjected for finishing of the surface by immersion, coating, special finishing;
- absorption and releasing of humidity, generating the effect of auto-water-proofing in case of the long-drawn contact with water;
- capable of processing on gripper or shuttle weaving machines, specific for many industrial companies.

The yarn properties are presented in table 1.

Table 2 shows the initial values for physical-chemical characteristics of cotton fabric type CORTINA.

The 100% cotton fabric type CORTINA was processed with Hexafluoropropane, at a power of 50 W,

Table 1

Yarn type	**Nm20/4 Cotton 100%**	**Nm10/2 Cotton 100%**	**Standard method**
Parameter/ measurement unit	**V1 Warp**	**V1 Weft**	
Yarn count: dtex (den) Tex Nm	 – 100,57x2 9,94/2	 – 51,4x4 19,5/4	SR EN ISO 2060
Breaking force (N)	24,47	30,06	SR EN 2062
Breaking elongation (%)	8,31	10,69	SR EN 2062
Twist (T/m)	339	562	SR EN ISO 2061
Yarn Twisting (T/m)	311	306	SR EN ISO 2061
Diameter (mm)	0,875	0,762	SR EN ISO 13152

Table 2

Characteristics			**Value**	**Reference documents**
Mass per unit area		g/m^2	400 ± 5 %	SR EN 12127/2003
Fibrous composition		%	Cotton 100%	SR ISO1833/95
Yarn Density	Warp	(thread/10 cm)	350 ± 5	SR EN 1049-2/2000 Method A
	Weft		195 ± 3	
Woven fabric thickness		mm	1.24	SR EN ISO 5084/2001
Width		cm	150 ± 3	SR EN 1773/2002
Linear Density of the yarns	Warp	tex (Nm)	37.4x2 (26.7/2)	SR EN ISO 2060:2010 Method B
	Weft	tex (Nm)	50.0x3 (20/3)	
Breaking force	Warp	N	1225.45±15%	SR EN ISO 13934-1/2002
	Weft		1264.95±15 %	
Breaking elongation	Warp	%	16.41	
	Weft		11.67	
Permeability to air		(l/m^2/s)	97.22	SR EN ISO 9237
Water permeability		%	31.9	SR 9005/1979
Abrasion resistance of fabrics		No. abrasion cycle	35047±30%	SR EN ISO 12947-2/2002
Dimensional modifications to washing 60°C	Warp	%	–3.7	SR EN ISO 5077/2008
	Weft		–3.0	
Dimensional modifications to hot air 210°C	Warp	%	–3.7	INCDTP METHOD
	Weft		–2.3	
Thermic conductivity		W/mK	0.0414	INCDTP METHOD
Thermic resistance		m$^2\cdot$ K/W	0.0233	SR EN 31092 (ISO 11092)
Resistance to water-vapour		m$^2\cdot$ Pa/W	4.85	SR EN 31092 (ISO 11092)

a pressure of 20 mTorr, frequency range of kHz, at different treatment duration.

The poly-functional woven fabric destined for the temporary shelters for people, has a structure with two warps and one weft form 100% cotton yarns, with the linear density of the yarns between Tex (Nm) = 50×2 (20/2) and 29,4×3 (34/3), Tex (Nm) = 50×3 (20/3) and 100×3 (10/3), and the torsion of the simple yarn between 400 and 600 tors/m, the twist of the yarn between 200–350 tors/m, a density of the warp of 27–39 yarns/cm. The fabric has double warps and different aspects and properties on the two sides. It has a ratio of 10 warp yarns for the upper side, 20 warp yarns for the bottom side and 4 warp yarns for consolidation. The woven fabric has several functionalities obtained by special treatments for boiling, dying, exhausting with organic-phosphoric products for fireproofing and Tetra-fluoro-ethylene products for hydrophobic character, applied by classical finishing treatments or plasma treatments in Hexafluoropropane.

The weight of fabric is between 400 g/m^2 and 600 g/m^2.

RESULTS AND DISCUSSION

Several properties were tested and mathematically modelled on the plasma treated CORTINA fabric:

A. Warp breaking force.
B. Weft breaking force.
C. Abrasion resistance.

The purpose is to obtain the optimal value for the plasma treatment duration.

A. Warp breaking force

For the evaluation of the maximal warp breaking force of the 100% cotton fabrics type CORTINA, there were extracted and analysed 20 samples from each untreated respectively treated in Hexafluoropropane plasma for different times (10, 20, 30 and 90 min) cotton fabrics. The average, the standard deviation and the coefficient of variation were estimated by descriptive analysis with a confidence interval of 95% and are showed in table 3.

The ANOVA test with one factor was used, in order to evaluate the influence of the plasma treatment on the maximal warp breaking force for the 100% cotton fabrics and the results are showed in table 4. The test evidenced that there are significant differences between the averages of the 5 groups of experimental data (p < 0,001). This fact demonstrates the influence of plasma treatment and its duration upon the maximal warp breaking force of the 100% cotton fabrics.

By applying the posthoc Duncan test, it was evidenced the existence of a single homogenous subset of the averages, showed in table 5.

It could observed from the analysis of the test results, that the highest value of the warp breaking force for the 100% cotton fabrics treated in Hexafluoropropane plasma is for the 30 minutes treatment time, while the lowest value is for the untreated (control) sample.

Table 3

Indicator	Control sample warp, N	Sample treated in plasma for 10 minutes, warp, N	Sample treated in plasma for 20 minutes, warp, N	Sample treated in plasma for 30 minutes, warp, N	Sample treated in plasma for 90 minutes, warp, N
Average	1225.45±1.38	1227.50±1.35	1229.80±1.42	1232.00±1.58	1229.80±1.56
STDEV	2.95	2.87	3.04	3.37	3.33
CV	0.24%	0.23%	0.25%	0.27%	0.27%

Table 4

ANOVA		Sum of Squares	df	Mean Square	F	Sig.
Warp breaking force	Between groups	501.840	4	125.460	12.894	.000
	Within groups	924.350	95	9.730		
	Total	1426.190	99			

Table 5

Warp breaking force	Treatment time [min]	N	Subset for alpha = .05			
		1	2	3	4	5
Duncan	0	20	1225.450			
	10	20		1227.500		
	20	20			***1229.800***	
	90	20			***1229.800***	
	30	20				1232.000
	Sig.		1.000	1.000	1.000	1.000

B. Weft breaking force

In the table 6 there are presented the results of the tests for the determination of the maximal tensile strength on weft direction, in Newton, according to the standard ISO 13934-1 on the 100% cotton fabric type CORTINA.

For the evaluation of the maximal weft breaking force for the 100% cotton fabrics type CORTINA, there were extracted 20 specimens from each sample, resulting from the untreated and plasma treated samples (10, 20, 30 and 90 min).

The average, the standard deviation and the coefficient of variation were estimated by descriptive analysis with a confidence interval of 95%.

The ANOVA test with one factor was used, in order to evaluate the influence of the plasma treatment on the maximal weft breaking force for the 100% cotton fabrics. The test evidenced that there are significant differences between the averages of the 5 groups of experimental data ($p<0,001$), showed in table 7. This fact demonstrates the influence of plasma treatment and its duration upon the maximal weft breaking force for the 100% cotton fabrics.

By applying the posthoc Duncan test, it could be evidenced the existence of two homogenous subsets of averages, showed in table 8.

It could observed from the analysis of the test results that the highest value of the weft breaking force for the 100% cotton fabrics treated in Hexafluoropropane plasma is for the 30 minutes treatment time, while the lowest value is for the untreated (control) sample.

C. Abrasion resistance

In table 9 there are presented the results of the tests for abrasion resistance, in number of abrasion cycles, according to ISO 12947-2 for the 100% cotton fabric type CORTINA.

In order to evaluate the abrasion resistance for the 100% cotton fabrics type CORTINA, there were extracted 20 specimens from each sample, resulting from the untreated and plasma treated samples (10, 20, 30 and 90 min).

Table 6

Indicator	Control sample weft, N	Sample treated in plasma for 10 minutes, weft, N	Sample treated in plasma for 20 minutes, weft, N	Sample treated in plasma for 30 minutes, weft, N	Sample treated in plasma for 90 minutes, weft, N
Average	1264.95±1.41	1267.70±1.18	1269.65±1.32	1271.65±1.25	1268.85±1.43
STDEV	3.02	2.52	2.81	2.66	3.05
CV	0.24%	0.20%	0.22%	0.21%	0.24%

Table 7

ANOVA		Sum of Squares	df	Mean Square	F	Sig.
Weft breaking force	Between groups	491.840	4	122.960	15.476	.000
	Within groups	754.800	95	7.945		
	Total	1246.640	99			

Table 8

Weft breaking force	Time exposure	N	Subset for alpha = .05			
		1	2	3	4	**5**
Duncan(a)	0	20	1264.950			
	10	20		***1267.700***		
	90	20		***1268.850***	***1268.850***	
	20	20			***1269.650***	
	30	20				1271.650
	Sig.		1.000	.200	.372	1.000

Table 9

Indicator	Control sample [abrasion cycles]	Sample treated in plasma for 10 minutes, [abrasion cycles]	Sample treated in plasma for 20 minutes, [abrasion cycles]	Sample treated in plasma for 30 minutes, [abrasion cycles]	Sample treated in plasma for 90 minutes, [abrasion cycles]
Average	35048±42	35028±93	35501±288	36461±161	35457±128
STDEV	90	198	616	345	273
CV	0,26%	0,57%	1,74%	0,95%	0,77%

Table 10

ANOVA		Sum of Squares	df	Mean Square	F	Sig.
Abrasion resistance	Between groups	27063427.460	4	6765856.865	54.537	.000
	Within groups	11785646.500	95	124059.437		
	Total	38849073.960	99			

Table 11

Abrasion resistance	Treatment time [min]	N	Subset for alpha = .05		
		1	2	3	5
Duncan(a)	0	20	***35028.0***		
	10	20	***35047.7***		
	90	20		***35457.4***	
	20	20		***35500.8***	
	30	20			36461.3
	Sig.		.860	.698	1.000

Table 12

Crt. no.	Characteristics	Coefficients				R^2
		a	*b*	*c*	*d*	
1.	Tensile strength warp	7.277	-0.459×10^{-3}	–13.874	1.962	0.998
2.	Tensile strength weft	7.143	-0.385×10^{-3}	–11.186	0.920	0.996
3.	Abrasion resistance	12.775	-6.460×10^{-3}	–198.401	47.761	0.963

The average, the standard deviation and the coefficient of variation were estimated by descriptive analysis with a confidence interval of 95%.

The ANOVA test with one factor was used, in order to evaluate the influence of the plasma treatment on the abrasion resistance for the 100% cotton fabrics. The test evidenced that there are significant differences between the averages of the 5 groups of experimental data ($p < 0{,}001$), showed in the table 10. This fact demonstrates the influence of plasma treatment and its duration upon the abrasion resistance of the 100% cotton fabrics.

By applying the posthoc Duncan test, it could be evidenced the existence of two homogenous subsets of averages, showed in the table 11.

It could observed from the analysis of the test results, that the greatest value of the abrasion resistance for the 100% cotton fabrics treated in Hexafluoropropane plasma is for the 30 minutes treatment, while the smallest value is for the 10 minutes treatment.

MATHEMATICAL MODELLING

Mathematical modelling of test results was performed with the application for linear regression of the IBM SPSS Statistics software [20].

From the graphics of the mean values measured for the untreated and plasma treated (10, 20, 30 and 90 minutes) woven fabric (100% cotton type CORTINA), it results a sigmoid, non-linear shape of the curve for the estimation of the tensile strength on warp and weft direction as well as for the abrasion resistance. From the family of sigmoid curve shapes, the best fit with experimental data measured in relation to the tendency of real variation of the modelled physical dimensions, can be performed with an exponential equation 1, of the form:

$$y = e^{\left(a + b \times t_x^1 + \frac{c}{t_x^1} + \frac{d}{(t_x - 100)^2}\right)} \qquad (1)$$

with: $t_x = (t_{tp} + 120)$ and the values of the coefficients determined by application of IBM SPSS Statistics and the procedure of non-linear regression, as follows (table 12).

The dynamics of measured and estimated values (based on the non-linear regression model) for warp breaking force of the 100% cotton fabric type CORTINA, 400 g/m^2, treated in Hexaflouropropane ($C_3H_2F_6$) plasma for different times (10, 20, 30 and 90 min), is showed in figure 3.

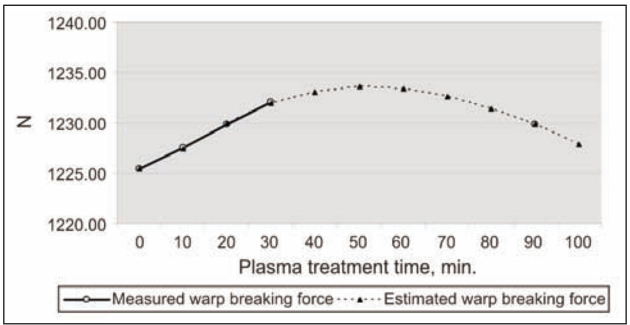

Fig. 3. Graphical representation of the regression which estimates the dynamics of warp breaking force for 100% cotton fabric, 400 g/m^2, treated in hexaflouropropane ($C_3H_2F_6$) plasma

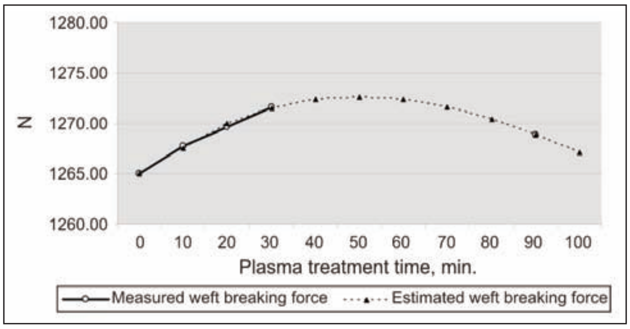

Fig. 4. Graphical representation of the regression which estimates the dynamics of weft breaking force for 100% cotton fabric, 400 g/m^2, treated in hexaflouropropane ($C_3H_2F_6$) plasma

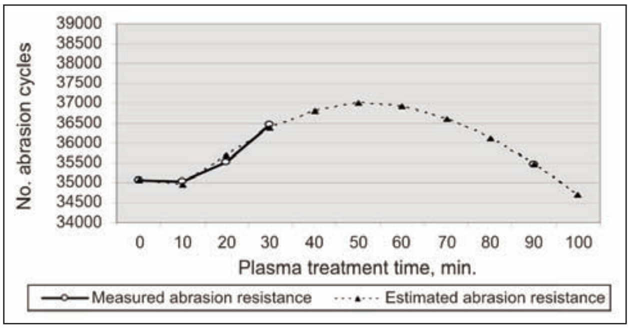

Fig. 5. Graphical representation of the regression which estimates the dynamics of abrasion resistance for 100% cotton fabric, 400 g/m^2, treated in hexaflouropropane ($C_3H_2F_6$) plasma

By analysing the results it can be observed that the highest value of warp breaking force of 100% cotton fabric type CORTINA, 400 g/m^2, was 1233.54 N for samples treated in Hexaflouropropane ($C_3H_2F_6$) plasma for 50 minutes and the lowest value was 1225.47 N for untreated samples. From the dynamics of estimated breaking force it was also noticed that by treating samples in plasma for more than 120 minutes, the maximum warp breaking force decreases below 1222.98 N, respectively the corresponding value of untreated samples. Most likely this is due to damage of structural surface fabrics caused by its bombing with reactive plasma particles. In conclusion it is recommended that the functionalization of 100% cotton fabrics, to be made by treating it with Hexafluoropropane plasma, for about 30–50 minutes. By using this treatment the average value of warp breaking force for 100% cotton fabric type CORTINA, 400 g/m^2 increase about 0.53% compared to the control (untreated) samples.

The dynamics of measured and estimated values (based on the non-linear regression model) for warp breaking force of the 100% cotton fabric type CORTINA, 400 g/m^2, treated in Hexaflouropropane ($C_3H_2F_6$) plasma for different times (10, 20, 30 and 90 min), is showed in figure 4.

By analysing the results it can be observed that the highest value of weft breaking force of 100% cotton fabric type CORTINA, 400 g/m^2, was 1272.65 N for samples treated in Hexaflouropropane ($C_3H_2F_6$) plasma for 50 minutes and the lowest value was 1264.95 N for untreated samples. From the dynamics of estimated breaking force it was also noticed that by treating samples in plasma for more than 120 minutes, the maximum weft breaking force decreases below 1262.62 N, respectively the corresponding value of untreated samples. Most likely this is due to damage of structural surface fabrics caused by its bombing with reactive plasma particles. In conclusion it is recommended that the functionalization of 100% cotton fabrics, to be made by treating it with Hexafluoropropane plasma, for about 30-50 minutes. By using this treatment the average value of warp breaking force for 100% cotton fabric type CORTINA, 400 g/m^2 increase about 0.53% compared to the control (untreated) samples.

The dynamics of measured and estimated values (based on the non-linear regression model) for abrasion resistance of the 100% cotton fabric type CORTINA, 400 g/m^2, treated in Hexaflouropropane ($C_3H_2F_6$) plasma for different times (10, 20, 30 and 90 min), is showed in figure 5.

By analysing the results it can be observed that the highest value of abrasion resistance of 100% cotton fabric type CORTINA, 400 g/m^2, was 36,986 abrasion cycles for samples treated in Hexaflouropropane ($C_3H_2F_6$) plasma for 50 minutes and the lowest value was 35090 abrasion cycles for samples treated in plasma for 10 min. From the dynamics of estimated abrasion resistance it was also noticed that by treating samples in plasma for more than 100 minutes, the maximum abrasion resistance decreases below 35048 abrasion cycles, respectively the corresponding value of untreated samples. Most likely this is due to damage of structural surface fabrics caused by its bombing with reactive plasma particles. In conclusion it is recommended that the functionalization of 100% cotton fabrics, to be made by treating it with Hexafluoropropane plasma, for about 30–50 minutes. By using this treatment the average value of abrasion resistance for 100% cotton fabric type CORTINA, increase about 1.29% compared to the control (untreated) samples.

CONCLUSIONS

The aim of the paper was to improve the mechanical resistance of the treated fabric and to obtain optimal plasma process duration.

Thus, the 100% cotton fabric type CORTINA was chosen for plasma treatment. Several treated and untreated samples (20 samples) were analysed for three investigation tests: warp breaking force, weft breaking force and abrasion resistance.

The fabric has several functionalities due to special finishing treatments, characterized by a compact structure able to provide an adequate microclimate in the shelter for people and meets the conditions of mechanical resistance to dynamic stress, at working temperatures of up to 150°C.

Statistical tests on these samples were performed for each investigation test. The mean values were analysed and graphical curves were proposed, estimating the evolution of the values. Based on these graphical curves, the maximal values for each investigation test were determined. The optimal plasma process duration was determined related to the maximal values of the investigation tests. It resulted an optimal process duration of about 30 minutes for best mechanical properties of the fabric.

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Interval type 2-fuzzy TOPSIS and fuzzy TOPSIS method in supplier selection in garment industry

AYTAÇ YILDIZ

REZUMAT – ABSTRACT

Metoda fuzzy TOPSIS Interval tip 2 şi metoda fuzzy TOPSIS în selectarea furnizorului din industria de confecţii

Selectarea furnizorului necesită luarea în considerare a mai multor factori și poate fi privită ca o problemă de luare a deciziilor cu criterii multiple (MCDM). Tehnica Fuzzy pentru preferința comenzii în mod similar cu Soluția Ideală (TOPSIS) este una dintre cele mai frecvent utilizate metode în rezolvarea diverselor probleme MCDM. În ultimul timp, TOPSIS a fost combinat cu seturile fuzzy interval tip 2. Seturile fuzzy interval tip 2 includ mai multe incertitudini decât seturile fuzzy de tip 1. Acestea asigură flexibilitate pentru a indica incertitudinea și neclaritatea din lumea reală. Prin urmare, în acest studiu, metodele fuzzy TOPSIS interval tip 2 și metodele fuzzy TOPSIS au fost folosite pentru selectarea celui mai bun furnizor, la firma producătoare de confecții "X", care operează în Turcia. La finalul acestui studiu, selecția celor mai buni furnizori se realizează prin compararea rezultatelor obținute din ambele metode. Rezultatul a evidenţiat că furnizorul "A3" este cel mai bun furnizor, dintre alternative, conform rezultatelor obținute din comparare, deoarece selectarea furnizorului "A3" va aduce avantaje semnificative întreprinderii în ceea ce privește atât beneficiile, cât și costurile.

Cuvinte-cheie: luarea deciziilor în funcţie de criterii multiple, metoda fuzzy TOPSIS interval tip 2, fuzzy TOPSIS, industria de confecții, selectarea furnizorului

Interval type 2-fuzzy TOPSIS and fuzzy TOPSIS method in supplier selection in garment industry

Supplier selection requires the consideration of multiple factors, and it can be viewed as a multi criteria decision making (MCDM) problem. Fuzzy Technique for Order Preference by Similarly to Ideal Solution (TOPSIS) is one of the most frequently used methods in solving several MCDM problems. Lately, fuzzy TOPSIS has been combined with interval type-2 fuzzy sets. Interval type-2 fuzzy sets include more uncertainties than type-1 fuzzy sets. They ensure us flexibility to indicate the uncertainty and the fuzziness of the real world. Therefore, in this study, interval type 2-Fuzzy TOPSIS and fuzzy TOPSIS methods were used for selection of the best supplier, at "X" garment firm operating in Turkey. At the end of this study, the best supplier selection is made through comparing the results obtained from both two methods. As a result, "A3" supplier has become the best supplier among alternatives according to the results obtained from comparing because the selection of the "A3" supplier will contribute significantly to the enterprise in point of both benefit and cost.

Keywords: multi criteria decision making, interval type 2-fuzzy TOPSIS, fuzzy TOPSIS, garment industry, supplier selection

INTRODUCTION

In the recent years, to establish an effective chain of supply has become one of the prerequisites for the survival of the companies in an environment of increasing competition with the effect of the globalization. Indispensable condition of establishing an effective chain of supply is the proper selection of the partner companies in the supply chain [1].

The selection of the supplier is a strategic and operational task that is very important for maintaining the development of partnership in the supply chain [2]. Determining the companies to do business together has put forward the problem of evaluation and selection of the supplier regarding the notion of the supply chain administration [1]. The selection of the supplier is one of the most important functions which are to be done by the buying determiners that aim to preserve the long term financial capacity and the competitiveness of the company. The selection of the most favourable supplier will increase the institutional competitiveness while decreasing the unit cost substantially. Because the suppliers are important participants in the supply chain and they affect the price and quality of the final product that's provided to the market [3, 4]. The supplier selection is accepted as a five-staged process by most of the purchasing departments, which is formed by feeling the need to a new supplier, determining and formulation of the criteria of decision, preselection, final supplier selection, and observing the chosen suppliers [3–5]. The quality of the final selection is closely related to the quality of all these stages of supplier selection [5]. To lead a long term partnership with the suppliers and to select a limited number but reliable ones are two of the most important decisions of the supply chain administration. The selection of the right supplier, because of this, depends on a long list of qualitative and quantitative factors [6]. Traditionally, such methods as groups of decision, supplier scoring or supplier evaluations to choose from the candidate suppliers list are chosen and applied in this process [3, 4].

The issue of supplier selection can be regarded as an issue with multiple criteria because of many quantitative and non-quantitative data it has in itself [1, 4, 6]. Various numbers of models and methods have been developed by the managements to be benefited in solving the problem of supplier selection after the increase in the importance of the supplier selection [6]. In solution of supplier selection problem fuzzy MCDM techniques are used such as quality function deployment (QFD) methodology, fuzzy analytic hierarchy process (AHP), TOPSIS, fuzzy analytic network process (ANP), fuzzy multi-criteria optimization and compromise solution, genetic algorithm (GA), PROMETHEE, grey relational analysis (GRA), and data envelopment analysis (DEA) [5]. A number of the studies performed on the selection of the supplier in literature are summarised as below.

A two-staged method formed by the DEA and the fuzzy TOPSIS methods according to the criteria of cost, quality, delivery and profile was used for the supplier selection in the textile sector of Taiwan in Chen's study [7], Yayla et al. [8], used the fuzzy TOPSIS method for the supplier selection in textile sector by using such as criteria quality, delivery time, cost, flexibility, geographical location. Ayyıldız and Demirel used the generalised Choquet Integral method for the supplier selection according to a number of various criteria like the price, geographical position, quality, financial status, flexibility, production capacity, the place in the textile sector, former performance, fulfilling the customer needs, logistics, delivery performance, packing, cooperation, service and support, closeness of the relations, and resolution of disagreements [9]. Özkök and Tiryaki applied the MLSSP (multi-objective linear supplier selection problem with multiple-item) method by using the criteria of quality, net cost, and service for the supplier selection [10]. In recent years, in solution of supplier selection problem there are used MCDM techniques that Rajesh and Malliga [11] AHP-QFD, Nazari-Shirkouhi et al. [12] fuzzy multi-objective linear programming, Rouyendegh and Saputro [13] fuzzy TOPSIS-multi choice goal programming (GP), Kar [14] AHP-fuzzy GP, Chen and Wu [15] modified failure mode and effect analysis, Ware et al. [16] mixed integer nonlinear program (MINLP), Rezaei et al. [17] fuzzy AHP, Jadidi et al. [18] multi-objective optimization, Dargi et al. [19] fuzzy ANP, Junior et al. [20] fuzzy TOPSIS-fuzzy AHP, Dobos and Vörösmarty [21] DEA, Safa et al. [22] TOPSIS.

The purpose of MCDM is to select a best competitor from a sequence of alternatives by means of assessing multi criteria of the alternatives [23, 24]. In recent years, some methods have been put forward for using fuzzy multi criteria decision-making based on type-1 fuzzy sets [24]. Uncertainty about the best alternative choice significantly affects the decision making process. The general structure of fuzzy reasoning lets usage much of this uncertainty and fuzzy systems operate type-1 fuzzy sets (FSs). When an asset is uncertain, it is difficult to define its accurate value, and of course a type-1 fuzzy set makes more sense than a traditional set. However, it is not reasonable to use an accurate membership function for something uncertain, so in this case what we need is another type of fuzzy sets, those which are able to operate these uncertainties, called type-2 fuzzy sets (T2FSs). The level of uncertainty in a system can be reduced by using type-2 fuzzy sets because this offers better ability to operate linguistic uncertainties by modelling ambiguity [25]. Type-2 fuzzy sets were introduced by Zadeh [26] as an extension of type-1 FSs. T2FSs are characterized with type-2 membership functions which are fuzzy themselves [27–29]. The membership functions of T2FSs are three dimensional and contain a step of uncertainty, it is the new third dimension of T2FSs and the step of uncertainty that ensures flexibility that makes it possible to directly model and operate uncertainties [29]. Type-2 fuzzy logic is shown in figure 1.

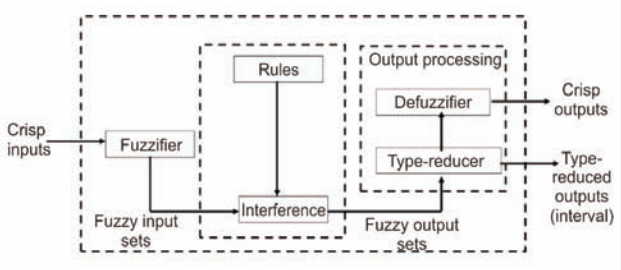

Fig. 1. Type-2 fuzzy logic system structure

Type-1 fuzzy sets are directly incapable to model such uncertainties, whereas, because their membership functions are fuzzy unlike in type-1 fuzzy sets, type-2 fuzzy sets are capable for such modelling [30]. T2FSs are more capable than ordinary fuzzy sets in utilization of defective and defective information in real world problems. Researchers use interval type-2 fuzzy sets (IT2FSs) to deal with the difficulties in establishing and handling the secondary membership functions. The notion of IT2FSs is described by an interval-valued membership function [27]. In IT2FSs, the upper and lower fuzzy membership functions of the type-2 fuzzy set were provided from the type-1 fuzzy membership function [31]. Some studies relating to interval type-2 fuzzy sets are shown below. Abdullah and Zamri [32] applied a decision making method to rank the causes linked to road accidents. Four criteria and five alternatives were used to be analysed using the interval type-2 fuzzy TOPSIS. The method suggests that ‘speeding’ is the main cause contributed to road accidents. Chen and Lee [24], in their study offered an interval type-2 fuzzy TOPSIS method to operate the fuzzy multi criteria decision making problems based on interval type-2 fuzzy sets. Nurnadiah and Lazim [31], in this paper used the IT2FS concept in the evaluation process to assess the attribute weight based on the credibility of data. Lee and Chen [33] used a method for fuzzy multi criteria group decision-making based on ranking values and the arithmetic operations of interval type-2 fuzzy sets. Wu and Mendel [34] used interval type-2 fuzzy sets for using fuzzy multi criteria decision-making problems. Chen and Wang [35] have used a new method for fuzzy multi criteria decision making based on interval type-2 fuzzy sets. Chen [36] used an ELECTRE based outranking method for multiple criteria decision making based on interval type-2 fuzzy sets. Celik et al. [37] is proposed an interval type-2 fuzzy MCDM method based on TOPSIS and GRA. Dymova Dymova et al. [38] proposed an interval type-2 fuzzy extension of the TOPSIS method realized with the use of a-cuts representation of the interval type-2 fuzzy values. Liao [39] presented two interval type 2 fuzzy multi-criteria decision making methods for material selection. Abdullah and Otheman [40] used interval type-2 fuzzy TOPSIS method for supplier selection.

The aim of this study is to make a selection of the best suppliers using interval type-2 fuzzy TOPSIS and fuzzy TOPSIS methods. In the study it is explained in detail that interval type-2 fuzzy TOPSIS method is little used in the literature.

FUZZY TOPSIS METHOD BASED ON INTERVAL TYPE-2 FUZZY SETS

Lee and Chen [32] introduced the notion of ranking values of trapezoidal interval type-2 fuzzy sets. Let $\tilde{\tilde{A}}_i$ be an interval type-2 fuzzy set shown in figure 2, where:

$$\tilde{\tilde{A}}_i = \left(\tilde{A}_i^U . \tilde{A}_i^L\right) = \left(\left(a_{i1}^U, a_{i2}^U, a_{i3}^U, a_{i4}^U; H_1(\tilde{A}_i^U), H_1(\tilde{A}_i^L)\right)\right)$$

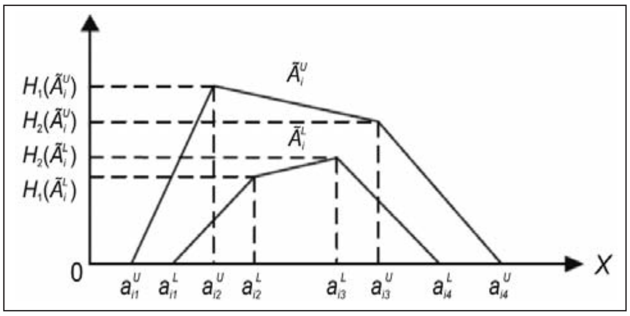

Fig. 2. A trapezoidal interval type-2 fuzzy number [33]

The ranking value $Rank\ (\tilde{\tilde{A}}_i)$ of the trapezoidal interval type-2 fuzzy set $\tilde{\tilde{A}}_i$ is identified as follows [33]:

$$Rank\ (\tilde{\tilde{A}}_i) = M_1(\tilde{A}_i^U) + M_1(\tilde{A}_i^L) + M_2(\tilde{A}_i^U) + M_2(\tilde{A}_i^L) + + M_3(\tilde{A}_i^U) + M_3(\tilde{A}_i^L) - \frac{1}{4}\Big(S_1(\tilde{A}_i^U) + S_1(\tilde{A}_i^L) + S_2(\tilde{A}_i^U) + + S_2(\tilde{A}_i^L) + S_3(\tilde{A}_i^U) + S_3(\tilde{A}_i^L) + S_4(\tilde{A}_i^U) + S_4(\tilde{A}_i^L)\Big) + + H_1(\tilde{A}_i^U) + H_1(\tilde{A}_i^L) + H_2(\tilde{A}_i^U) + H_2(\tilde{A}_i^L) \tag{1}$$

Where $M_p(\tilde{A}_i^j)$ indicates the average of the elements a_{ip}^j and $a_{i(p+1)}^j$, $M_p(\tilde{A}_i^j) = (a_{ip}^j + a_{i(p+1)}^j)/2$, $1 \leq p \leq 3$, $S_q(\tilde{A}_i^j)$ indicates the standard deviation of the elements a_{iq}^j and $a_{i(q+1)}^j$,

$$S_q(\tilde{A}_i^j) = \sqrt{\frac{1}{2}\sum_q^{q+1}\left(a_{ik}^j\ \frac{1}{2}\sum_{k=q}^{q+1} a_{ik}^j\right)^2}, 1 \leq q \leq 3$$

$S_4(\tilde{A}_i^j)$ indicates the standard deviation of the elements a_{i1}^j, a_{i2}^j, a_{i3}^j, a_{i4}^j

$$S_4(\tilde{A}_i^j) = \sqrt{\frac{1}{4}\sum_{k=1}^{4}\left(a_{ik}^j\ \frac{1}{4}\sum_{k=1}^{4} a_{ik}^j\right)^2}$$

$H_p(\tilde{A}_i^j)$ indicates the membership value of the element $a_{i(p+1)}^j$ in the trapezoidal membership function $\tilde{A}_i^j$, $1 \leq p \leq 2$, $j \in \{U, L\}$ and $1 \leq i \leq n$.

In eq. (1), the aggregate of: $M_1(\tilde{A}_i^U)$, $M_1(\tilde{A}_i^L)$, $M_2(\tilde{A}_i^U)$, $M_2(\tilde{A}_i^L)$, $M_3(\tilde{A}_i^U)$, $M_3(\tilde{A}_i^L)$, $H_1(\tilde{A}_i^U)$, $H_1(\tilde{A}_i^L)$, $H_2(\tilde{A}_i^U)$ and $H_2(\tilde{A}_i^L)$ is called the basic ranking score, where we decrease the average of $S_1(\tilde{A}_i^U)$, $S_1(\tilde{A}_i^L)$, $S_2(\tilde{A}_i^U)$, $S_2(\tilde{A}_i^L)$, $S_3(\tilde{A}_i^U)$, $S_3(\tilde{A}_i^L)$, $S_4(\tilde{A}_i^U)$ and $S_4(\tilde{A}_i^L)$ from the basic ranking score to give the distribute interval type-2 fuzzy set, where $1 \leq i \leq n$.

Assuming that there is a set X of alternatives, where $X = \{x_1, x_1, ..., x_n\}$ and assuming that there is a set F of criteria, where $F = \{f_1, f_1, ..., f_n\}$. Assuming that there are k decision-makers D_1, D_2,... and D_k. The set F of criteria can be divided into two sets F_1 and F_2, where F_1 indicates the set of benefit criteria, F_2 indicates the set of cost criteria, $F_1 \cap F_2 = \varphi$, and $F_1 \cup F_2 = F$ TOPSIS method for fuzzy multi criteria decision-making problems based on interval type-2 fuzzy sets method is presented as follows:

Step 1: Establish the decision matrix Y_p of the pth decision-maker and establish the average decision matrix $\overline{Y}$, respectively, shown as follows:

$$Y_p = (\tilde{\tilde{f}}_{ij}^p)_{m \times n} = \begin{bmatrix} f_1 & \tilde{\tilde{f}}_{11}^p & \tilde{\tilde{f}}_{12}^p \dots & \tilde{\tilde{f}}_{1n}^p \\ f_2 & \tilde{\tilde{f}}_{21}^p & \tilde{\tilde{f}}_{22}^p \dots & \tilde{\tilde{f}}_{2n}^p \\ \vdots & \vdots & \vdots & \vdots \\ f_m & \tilde{\tilde{f}}_{m1}^p & \tilde{\tilde{f}}_{m2}^p \dots & \tilde{\tilde{f}}_{mn}^p \end{bmatrix} \tag{2}$$

$$\overline{Y} = (\tilde{\tilde{f}}_{ij})_{m \times n}\ , \tag{3}$$

where $(\tilde{\tilde{f}}_{ij}) = \left(\dfrac{\tilde{\tilde{f}}_{ij}^1 \oplus \tilde{\tilde{f}}_{ij}^2 \oplus \dots \oplus \tilde{\tilde{f}}_{ij}^k}{k}\right)$, $\tilde{\tilde{f}}_{ij}$ is an interval type-2 fuzzy set, $1 \leq i \leq m$, $1 \leq j \leq n$, $1 \leq p \leq k$, and k indicates the number of decision-makers.

Step 2: Establish the weighting matrix W_p of the criteria of the pth decision-maker and establish the average weighting matrix $\overline{W}$, respectively, shown as follows:

$$W_p = \left(\tilde{\tilde{w}}_i^p\right)_{1 \times m} = \left[\tilde{\tilde{w}}_1^p\ \ \tilde{\tilde{w}}_2^p \cdots \tilde{\tilde{w}}_m^p\right], \tag{4}$$

$$\overline{W} = (\tilde{\tilde{w}}_i)_{1 \times m}\ , \tag{5}$$

where $(\tilde{\tilde{w}}_i) = \left(\dfrac{\tilde{\tilde{w}}_i^1 \oplus \tilde{\tilde{w}}_i^2 \oplus \dots \oplus \tilde{\tilde{w}}_i^k}{k}\right)$, $\tilde{\tilde{w}}_i$ is an interval type-2 fuzzy set, $1 \leq i \leq m$, $1 \leq p \leq k$, and k indicates the number of decision-makers.

Step 3: Establish the weighted decision matrix $\overline{Y}_w$,

$$\overline{Y}_w = (\overset{\approx}{v}_{ij})_{m\times n} = \begin{array}{c} f_1 \\ f_2 \\ \vdots \\ f_m \end{array} \begin{bmatrix} \overset{\approx}{v}_{11} & \overset{\approx}{v}_{12} & \cdots & \overset{\approx}{v}_{1n} \\ \overset{\approx}{v}_{21} & \overset{\approx}{v}_{22} & \cdots & \overset{\approx}{v}_{2n} \\ \vdots & \vdots & & \vdots \\ \overset{\approx}{v}_{m1} & \overset{\approx}{v}_{m2} & \cdots & \overset{\approx}{v}_{mn} \end{bmatrix} \quad (6)$$

where $\overset{\approx}{v}_{ij} = \overset{\approx}{f}_{ij}$, $1 \le i \le m$, and $1 \le j \le n$.

Step 4: With eq. (1), compute the ranking value $Rank\,(\overset{\approx}{v}_{ij})$ of the interval type-2 fuzzy set $\overset{\approx}{v}_{ij}$ where $1 \le j \le n$. Establish the ranking weighted decision matrix $\overline{Y}^*_w$,

$$\overline{Y}^*_w = Rank\,(\overset{\approx}{v}_{11})_{m\times n}, \quad (7)$$

where $1 \le i \le m$ and $1 \le j \le n$.

Step 5: Determine the PIS $x^+ = (v_1^+, v_2^+, \ldots, v_m^+)$ and the NIS $x^- = (v_1^-, v_2^-, \ldots, v_m^-)$, where

$$v_i^+ = \begin{cases} \max\limits_{1\le j\le n} \{rank(\overset{\approx}{v}_{ij})\}, & \text{if } f_i \in F_1 \\ \min\limits_{1\le j\le n} \{rank(\overset{\approx}{v}_{ij})\}, & \text{if } f_i \in F_2 \end{cases} \quad (8)$$

and

$$v_i^- = \begin{cases} \min\limits_{1\le j\le n} \{rank(\overset{\approx}{v}_{ij})\}, & \text{if } f_i \in F_1 \\ \max\limits_{1\le j\le n} \{rank(\overset{\approx}{v}_{ij})\}, & \text{if } f_i \in F_2 \end{cases} \quad (9)$$

where F_1 indicates the set of benefit criteria, F_2 indicates the set of cost criteria, and $1 \le i \le m$.

Step 6: Compute the distance $d^+(x_j)$ between each alternative x_j and the PIS x^+, shown as follows:

$$d^+(x_j) = \sqrt{\sum_{i=1}^{m} (Rank(\overset{\approx}{v}_{ij}) - v_i^+)^2} \quad (10)$$

where $1 \le j \le n$. Compute the distance $d^-(x_j)$ between each alternative x_j and the NIS x^-, shown as follows:

$$d^-(x_j) = \sqrt{\sum_{i=1}^{m} (Rank(\overset{\approx}{v}_{ij}) - v_i^-)^2} \quad (11)$$

where $1 \le j \le n$.

Step 7: Compute the closeness coefficient $C(x_j)$ of x_j with respect to the PIS x^+, shown as follows:

$$C(x_j) = \frac{d^-(x_j)}{d^-(x_j) + d^+(x_j)}, \quad (12)$$

where $1 \le j \le n$.

Step 8: Rank the values of $C(x_j)$ in a decreasing range, where $1 \le j \le n$. The larger the value of $C(x_j)$, the higher the preference of the alternative x_j, where $1 \le j \le n$.

THE CASE STUDY

It is clear that, to be successful in today's competitive environment is mainly related to strategic thinking. The success only in the inner processes of the management would be a limited one. The perception that a pieced solution without evaluating the process as a whole would never be adequate is growing up more and more. Even the advantages to be got at the production stage aren't shared among the management, supplier, distributer and the customer they won't be affective. Much management who sees the success mainly in this integration is seen to be heading for the supply chain. The way to the success in such a competitive environment can be achieved by reflecting the inner synergy created in the management to the whole process of supply chain. Because of its formation, the garment industry is also cooperating with a large web of suppliers. As the main supplier of the garment industry, the textile industry is one of the most powerful manufacturing sectors of Turkey with its share of the national income, the employment it provides, and a higher potential of export. For this cause the selection of appropriate suppliers in a sector where there is a strict competition is a tough matter of decision for the garment manufacturers. From this point, determining the criteria and their importance on the decision making process, and along with this the selection of the most appropriate supplier in a scientific manner will affect the garment manufacturers' strength of a quality based competitiveness. However, to do business with inappropriate suppliers will bring the loss of resources and opportunities. Because of this, we did a selection of appropriate supplier for a prominent garment company of Turkey. This company has approximately 2,437 employees, a daily production capacity of 55 tons of knitting, 60 tons of colouring, 100,000 meters of printing, 150,000 pieces of garment and 100,000 pieces of household textiles in an area of 325,000 square meters total. With these specs, being one of the four companies in the world to do all the processes like producing from yarn to finished product at the same time, it is also among the biggest companies in Turkey to create employment regarding its capacity to create employment.

The firm exports all of its production out of three alternative suppliers was performed through interval type-2 Fuzzy TOPSIS and fuzzy TOPSIS method. In the solution of the problem, selection criteria determined as quality, delivery time, cost, flexibility, geographical location were defined in line with literature review and the opinions of the firm’s three decision makers. Decision making group was composed of three experts working in the managerial level in the firm. Supplier selection problem hierarchy suggested by these experts is shown in the figure 3.

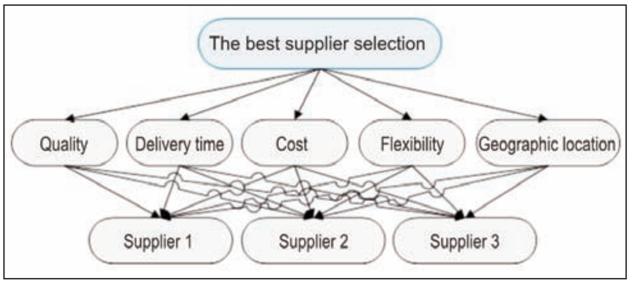

Fig. 3. Hierarchy of the supplier selection problem

In figure 3, the criteria used in the selection of suppliers are briefly described below.

Quality: As much as being the responsibility of the manufacturer, the quality is also the responsibility of the supplier who provides semi products, components and tools to the manufacturer. The production capability of the supplier also determines the quality of the finished product [41]. The deficiency in the quality or performance of the purchased product can create shortages in production and orders bring out time and cost burden like the repairment or replacing of the product and by this way it can cause loss of customers. A supplier who is selected because of the cost-effectiveness can lose the advantage of price which is the cause of selection because of the deficiency of performance. The sub-factors like the quality inspection the supplier applies for cooperation, its possession of quality standards, or the rate of fault in its production are regarded as part of the criteria of quality [42].

Delivery time: Not being able to deliver the product on time because of the uncertainties in the customer demands has made the on time delivery an important issue [43]. The capability of the supplier to conform to the predefined delivery program is an important criterion on choosing the supplier. The supplier's delivery of the order on time is important for a management for not to experience delays in its businesses [42]. A faster delivery of the products and a shorter delivery chain affect the delivery time substantially [44].

Cost: In choosing a service or a good to be purchased, the cost that will be taken over by the management for these purchases is one of the points that are taken care of mostly; the maximization of the profit is not possible without the minimization of the

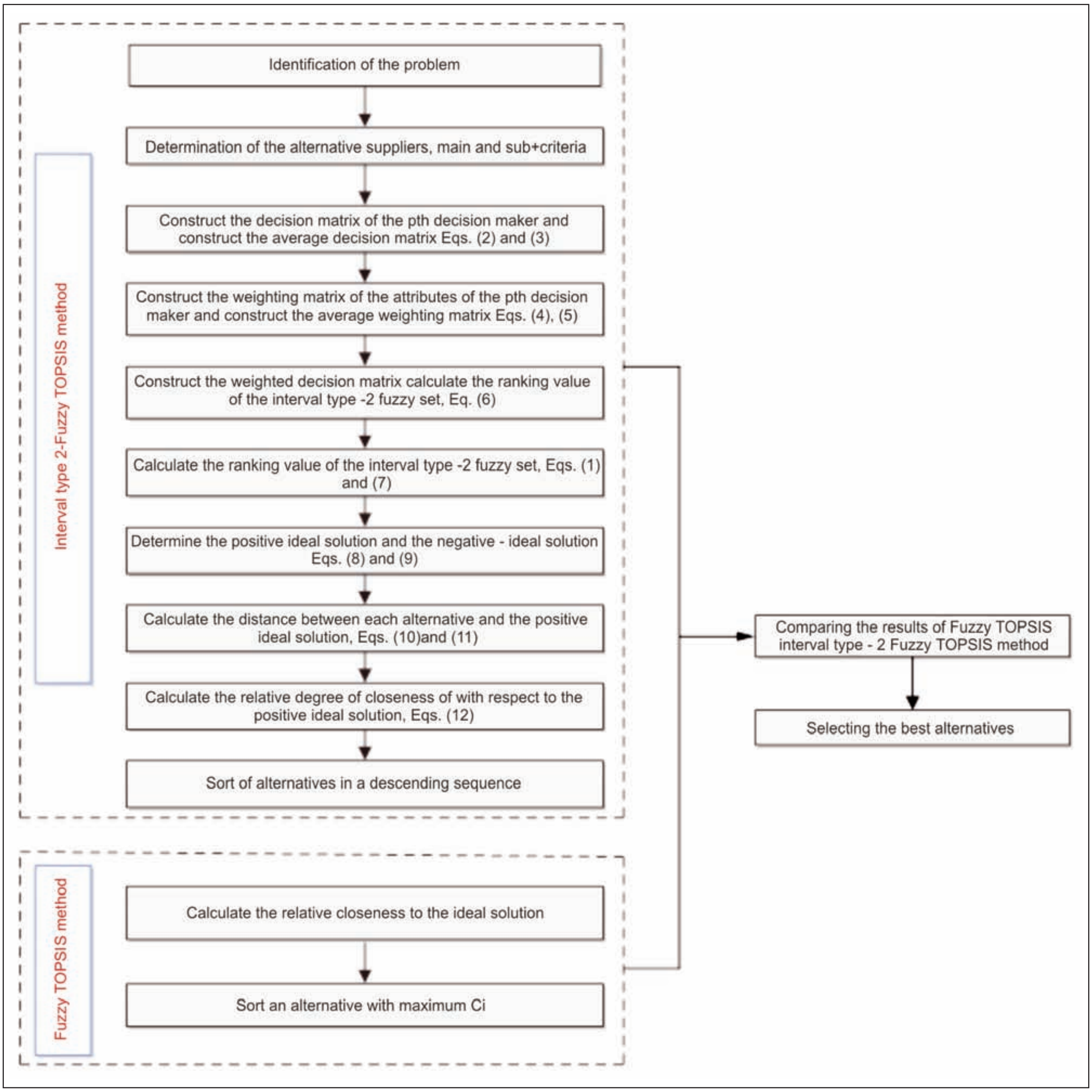

Fig. 4. Supplier selection problem flowchart

cost [42]. The cost of purchasing can change according to the amount that is being supplied. In case there is discount according to the amount, the relation of price to the amount of supply should be taken into consideration. When doing business with an international partner, on the other hand, the costs of customs and shipment are added to the problem of the selection of a supplier as new criteria. The manufacturer companies tend to supply the goods that they use for their products in minimum prices as possible. For this cause the companies have to find the lowest cost resources of supply by which they can minimize their production related costs [45].

Flexibility: The flexibility in administration and working means answering the changes demanded by the customers and the business environment, or reacting the changing conditions efficiently. Flexibility has a multi-dimensioned nature including the dimensions like supplying materials, machine operations, automation, work, route, product, new design, volume, enlargement, program, production, and market [46]. To answer the demands of changes in the design of the product, the changes in the combination of the products, and the changes in the volume of the product are the positive characteristics for the supplier [42]. Flexibility is also defined as the capability to change the rate of the production fast and effectively, the competence of the production systems in overcoming the changes in the shape and design of the products, and the capability to provide the market with new kinds of products faster [46].

Geographical location: Geographical location is important factor in supplier selection, as it impacts delivery time, transportation, and logistics costs. Some organisations require their suppliers to be located within a certain distance from their factory. Geographical location of the supplier's country and the transportation route require careful analysis in order to eliminate the risk of corruption in the supply chain [47].

After the identification of the supplier selection criteria, flowchart generated for Supplier selection is shown in figure 4.

Table 1

Linguistic terms	Interval type-2 fuzzy sets
Very Low (VL)	((0, 0, 0, 0.1, 1, 1), (0, 0, 0, 0.05, 0.9, 0.9))
Low (L)	((0, 0.1, 0.1, 0.3; 1, 1), (0.05, 0.1, 0.1, 0.2; 0.9, 0.9))
Medium Low (ML)	((0.1, 0.3, 0.3, 0.5; 1, 1), (0.2, 0.3, 0.3, 0.4; 0.9, 0.9))
Medium (M)	((0.3, 0.5, 0.5, 0.7; 1, 1), (0.4, 0.5, 0.5, 0.6; 0.9, 0.9))
Medium High (MH)	((0.5, 0.7, 0.7, 0.9; 1, 1), (0.6, 0.7, 0.7, 0.8; 0.9, 0.9))
High (H)	((0.7, 0.9, 0.9, 1; 1, 1), (0.8, 0.9, 0.9, 0.95; 0.9, 0.9))
Very High (VH)	((0.9, 1, 1, 1; 1, 1), (0.95, 1, 1, 1; 0.9, 0.9))

Table 2

Criteria	Decision-makers		
	DM1	DM2	DM3
Quality	VH	VH	H
Delivery Time	H	H	MH
Cost	VH	VH	H
Flexibility	M	MH	M
Geographic Location	M	ML	ML

Table 3

Criteria	Alternatives	Decision-makers		
		DM1	DM2	DM3
Quality	A1	MH	H	MH
	A2	M	H	L
	A3	MH	MH	VH
Delivery Time	A1	H	VH	MH
	A2	H	ML	MH
	A3	VH	H	MH
Cost	A1	MH	ML	H
	A2	H	M	VH
	A3	M	H	M
Flexibility	A1	MH	VH	L
	A2	H	MH	L
	A3	VH	H	L
Geographic location	A1	L	L	ML
	A2	VL	L	L
	A3	L	ML	L

Supplier selection interval using type 2-fuzzy TOPSIS

Table 1 shows the linguistic terms and their corresponding interval type-2 fuzzy sets. Assume that there are three decision-makers DM1, DM2 and DM3 to evaluate suppliers and assume that there are three alternatives Supplier 1 (A1), Supplier 2 (A2), Supplier 3 (A3) and five criteria "Quality", "Delivery time", "Cost", "Flexibility", "Geographic Location". Let X be the set of alternatives, where $X = \{A1, A2, A3\}$, and let F be the set of criteria, where $F = \{Quality, Delivery\ time, Cost, Flexibility, Geographic\ location\}$. Assume that the three decision-makers DM1, DM2, DM3 use the linguistic terms shown in table 1 to represent the weights of the five criteria, separately, as shown in table 2. In table 2, four benefit criteria are considered, including "Quality", "Delivery time", "Flexibility and "Geographic location" one cost criteria is considered, "Cost". Assume that the three decision-makers DM1, DM2, DM3 use the linguistic terms shown in table 1 to represent the evaluating values of the alternatives according to different criteria, respectively, as shown in table 3.

Step 1: Based on table 1 and eq. (2), we can construct the decision matrices Y_1, Y_2 and Y_3 of the alternatives A1, A2 and A3, respectively, where,

$$Y_1 = \begin{array}{l} \textit{Quality} \\ \textit{Delivery time} \\ \textit{Cost} \\ \textit{Flexibility} \\ \textit{Geographic location} \end{array} \begin{bmatrix} MH & M & MH \\ H & H & VH \\ MH & H & M \\ MH & H & VH \\ L & VL & L \end{bmatrix}$$

$$Y_2 = \begin{array}{l} \textit{Quality} \\ \textit{Delivery time} \\ \textit{Cost} \\ \textit{Flexibility} \\ \textit{Geographic location} \end{array} \begin{bmatrix} H & H & MH \\ VH & ML & H \\ ML & M & H \\ VH & MH & H \\ L & L & ML \end{bmatrix}$$

$$Y_3 = \begin{array}{l} \textit{Quality} \\ \textit{Delivery time} \\ \textit{Cost} \\ \textit{Flexibility} \\ \textit{Geographic location} \end{array} \begin{bmatrix} MH & V & VH \\ MH & MH & MH \\ H & VH & M \\ L & L & L \\ ML & L & L \end{bmatrix}$$

Based on table 1 and eq. (3), we can obtain the average decision matrix $\overline{Y}$, shown as follows:

$$\overline{Y} = \begin{array}{l} \textit{Quality} \\ \textit{Delivery time} \\ \textit{Cost} \\ \textit{Flexibility} \\ \textit{Geographic location} \end{array} \begin{bmatrix} \tilde{\tilde{f}}_{11} & \tilde{\tilde{f}}_{12} & \tilde{\tilde{f}}_{13} \\ \tilde{\tilde{f}}_{21} & \tilde{\tilde{f}}_{22} & \tilde{\tilde{f}}_{23} \\ \tilde{\tilde{f}}_{31} & \tilde{\tilde{f}}_{32} & \tilde{\tilde{f}}_{33} \\ \tilde{\tilde{f}}_{41} & \tilde{\tilde{f}}_{42} & \tilde{\tilde{f}}_{43} \\ \tilde{\tilde{f}}_{51} & \tilde{\tilde{f}}_{52} & \tilde{\tilde{f}}_{53} \end{bmatrix}$$

where

$\tilde{\tilde{f}}_{11} = ((0.57,0.77,0.77,0.93,1,1),(0.67,0.77,0.77,0.85,0.9,0.9))$

$\tilde{\tilde{f}}_{12} = ((0.33,0.5,0.5,0.67,1,1),(0.42,0.5,0.5,0.58,0.9,0.9))$

$\tilde{\tilde{f}}_{13} = ((0.63,0.8,0.8,0.93,1,1),(0.72,0.8,0.8,0.87,0.9,0.9))$

$\tilde{\tilde{f}}_{21} = ((0.7,0.87,0.87,0.97,1,1),(0.78,0.87,0.87,0.92,0.9,0.9))$

$\tilde{\tilde{f}}_{22} = ((0.43,0.63,0.63,0.8,1,1),(0.53,0.63,0.63,0.72,0.9,0.9))$

$\tilde{\tilde{f}}_{23} = ((0.7,0.87,0.87,0.97,1,1),(0.78,0.87,0.87,0.92,0.9,0.9))$

$\tilde{\tilde{f}}_{31} = ((0.43,0.63,0.63,0.8,1,1),(0.53,0.63,0.63,0.72,0.9,0.9))$

$\tilde{\tilde{f}}_{32} = ((0.63,0.8,0.8,0.9,1,1),(0.72,0.8,0.8,0.85,0.9,0.9))$

$\tilde{\tilde{f}}_{33} = ((0.43,0.63,0.63,0.8,1,1),(0.53,0.63,0.63,0.72,0.9,0.9))$

$\tilde{\tilde{f}}_{41} = ((0.47,0.6,0.6,0.73,1,1),(0.53,0.6,0.6,0.67,0.9,0.9))$

$\tilde{\tilde{f}}_{42} = ((0.4,0.57,0.57,0.73,1,1),(0.48,0.57,0.57,0.65,0.9,0.9))$

$\tilde{\tilde{f}}_{43} = ((0.53,0.67,0.67,0.77,1,1),(0.6,0.67,0.67,0.72,0.9,0.9))$

$\tilde{\tilde{f}}_{51} = ((0.03,0.17,0.17,0.37,1,1),(0.1,0.17,0.17,0.27,0.9,0.9))$

$\tilde{\tilde{f}}_{52} = ((0,0.07,0.07,0.23,1,1),(0.03,0.07,0.07,0.15,0.9,0.9))$

$\tilde{\tilde{f}}_{53} = ((0.03,0.17,0.17,0.37,1,1),(0.1,0.17,0.17,0.27,0.9,0.9))$

Step 2: Based on table 1 and eq. (4), we can obtain the weighting matrices W_1, W_2 and W_3, where

$W_1 = [VH \quad H \quad VH \quad M \quad M]$,

$W_2 = [VH \quad H \quad VH \quad MH \quad ML]$,

$W_3 = [H \quad MH \quad H \quad M \quad ML]$

With eq. (5), we can obtain the average weighting matrix $\overline{W}$:

$$\overline{W} = \left[\tilde{\tilde{w}}_1 \quad \tilde{\tilde{w}}_2 \quad \tilde{\tilde{w}}_3 \quad \tilde{\tilde{w}}_4 \quad \tilde{\tilde{w}}_5\right]$$

where

$\tilde{\tilde{w}}_1 = ((0.83,0.97,0.97,1,1,1),(0.9,0.97,0.97,0.98,0.9,0.9))$

$\tilde{\tilde{w}}_2 = ((0.63,0.83,0.83,0.97,1,1),(0.73,0.83,0.83,0.9,0.9,0.9))$

$\tilde{\tilde{w}}_3 = ((0.83,0.97,0.97,1,1,1),(0.9,0.97,0.97,0.98,0.9,0.9))$

$\tilde{\tilde{w}}_4 = ((0.37,0.57,0.57,0.77,1,1),(0.47,0.57,0.57,0.67,0.9,0.9))$

$\tilde{\tilde{w}}_5 = ((0.17,0.37,0.37,0.57,1,1),(0.27,0.37,0.37,0.47,0.9,0.9))$

Step 3: With eq. (6), we can obtain the weighted decision matrix $\overline{Y}_w$:

$$\overline{Y}_w = \begin{array}{l} \textit{Quality} \\ \textit{Delivery time} \\ \textit{Cost} \\ \textit{Flexibility} \\ \textit{Geographic location} \end{array} \begin{bmatrix} \tilde{\tilde{v}}_{11} & \tilde{\tilde{v}}_{12} & \tilde{\tilde{v}}_{13} \\ \tilde{\tilde{v}}_{21} & \tilde{\tilde{v}}_{22} & \tilde{\tilde{v}}_{23} \\ \tilde{\tilde{v}}_{31} & \tilde{\tilde{v}}_{32} & \tilde{\tilde{v}}_{33} \\ \tilde{\tilde{v}}_{41} & \tilde{\tilde{v}}_{42} & \tilde{\tilde{v}}_{43} \\ \tilde{\tilde{v}}_{51} & \tilde{\tilde{v}}_{52} & \tilde{\tilde{v}}_{53} \end{bmatrix}$$

Where

$\tilde{\tilde{v}}_{11} = ((0.47,0.74,0.74,0.93,1,1),(0.6,0.74,0.74,0.84,0.9,0.9))$

$\tilde{\tilde{v}}_{12} = ((0.28,0.48,0.48,0.67,1,1),(0.38,0.48,0.48,0.57,0.9,0.9))$

$\tilde{\tilde{v}}_{13} = ((0.53,0.77,0.77,0.93,1,1),(0.65,0.77,0.77,0.85,0.9,0.9))$

$\tilde{\tilde{v}}_{21} = ((0.44,0.72,0.72,0.93,1,1),(0.57,0.72,0.72,0.83,0.9,0.9))$

$\tilde{\tilde{v}}_{22} = ((0.27,0.53,0.53,0.77,1,1),(0.39,0.53,0.53,0.65,0.9,0.9))$

$\tilde{\tilde{v}}_{23} = ((0.44,0.72,0.72,0.93,1,1),(0.57,0.72,0.72,0.83,0.9,0.9))$

$\tilde{\tilde{v}}_{31} = ((0.36,0.61,0.61,0.8,1,1),(0.48,0.61,0.61,0.7,0.9,0.9))$

$\tilde{\tilde{v}}_{32} = ((0.53,0.77,0.77,0.9,1,1),(0.65,0.77,0.77,0.84,0.9,0.9))$

$\tilde{\tilde{v}}_{33} = ((0.36,0.61,0.61,0.8,1,1),(0.48,0.61,0.61,0.7,0.9,0.9))$

$\tilde{\tilde{v}}_{41} = ((0.17,0.34,0.34,0.56,1,1),(0.25,0.34,0.34,0.44,0.9,0.9))$

$\tilde{\tilde{v}}_{42} = ((0.15,0.32,0.32,0.56,1,1),(0.23,0.32,0.32,0.43,0.9,0.9))$

$\tilde{\tilde{v}}_{43} = ((0.2,0.38,0.38,0.59,1,1),(0.28,0.38,0.38,0.48,0.9,0.9))$

$\tilde{\tilde{v}}_{51} = ((0.01,0.06,0.06,0.21,1,1),(0.03,0.06,0.06,0.12,0.9,0.9))$

$\tilde{\tilde{v}}_{52} = ((0,0.02,0.02,0.13,1,1),(0.01,0.02,0.02,0.07,0.9,0.9))$

$\tilde{\tilde{v}}_{53} = ((0.01,0.06,0.06,0.21,1,1),(0.03,0.06,0.06,0.12,0.9,0.9))$

Step 4: With eq. (1), the ranking values $Rank\,(\tilde{\tilde{v}}_{ij})$ of the interval type-2 fuzzy set $\tilde{\tilde{v}}_{ij}$ can be computed, where $1 \le i \le 5$ and $1 \le j \le 3$

$$\begin{aligned} Rank\,(\tilde{\tilde{v}}_{11}) &= M_1(\tilde{v}_{11}^U) + M_1(\tilde{v}_{11}^L) + M_2(\tilde{v}_{11}^U) + M_2(\tilde{v}_{11}^L) + \\ &+ M_3(\tilde{v}_{11}^U) + M_3(\tilde{v}_{11}^L) - \frac{1}{4}\Big(S_1(\tilde{v}_{11}^U) + S_1(\tilde{v}_{11}^L) + S_2(\tilde{v}_{11}^U) + \\ &+ S_2(\tilde{v}_{11}^L) + S_3(\tilde{v}_{11}^U) + S_3(\tilde{v}_{11}^L) + S_4(\tilde{v}_{11}^U) + S_4(\tilde{v}_{11}^L)\Big) + \\ &+ H_1(\tilde{v}_{11}^U) + H_1(\tilde{v}_{11}^L) + H_2(\tilde{v}_{11}^U) + H_2(\tilde{v}_{11}^L) = \\ &= 0.61 + 0.67 + 0.74 + 0.74 + 0.84 + 0.79 - \\ &- \frac{1}{4}\Big(0.13 + 0.07 + 0 + 0 + 0.096 + 0.047 + \\ &+ 0.16 + 0.084\Big) + 1 + 0.9 + 1 + 0.9 = 8.04 \end{aligned}$$

In the same way, we can get

$Rank\,(\tilde{\tilde{v}}_{12}) = 6.55$, $\quad Rank\,(\tilde{\tilde{v}}_{13}) = 8.24$,

$Rank\,(\tilde{\tilde{v}}_{21}) = 7.92$, $\quad Rank\,(\tilde{\tilde{v}}_{22}) = 6.79$,

$Rank\,(\tilde{\tilde{v}}_{23}) = 7.92$, $\quad Rank\,(\tilde{\tilde{v}}_{31}) = 7.28$,

$$Rank(\tilde{\tilde{v}}_{32}) = 8.23, \quad Rank(\tilde{\tilde{v}}_{33}) = 7.28,$$

$$Rank(\tilde{\tilde{v}}_{41}) = 5.75, \quad Rank(\tilde{\tilde{v}}_{42}) = 5.64,$$

$$Rank(\tilde{\tilde{v}}_{43}) = 5.96, \quad Rank(\tilde{\tilde{v}}_{51}) = 4.16,$$

$$Rank(\tilde{\tilde{v}}_{52}) = 3.96, \quad Rank(\tilde{\tilde{v}}_{53}) = 4.16.$$

With eq. (7), we can construct the ranking weighted decision matrix $\overline{Y}_w^*$:

$$\overline{Y}_w^* = \begin{array}{l} \text{Quality} \\ \text{Delivery time} \\ \text{Cost} \\ \text{Flexibility} \\ \text{Geographic location} \end{array} \begin{bmatrix} Rank(\tilde{\tilde{v}}_{11}) & Rank(\tilde{\tilde{v}}_{12}) & Rank(\tilde{\tilde{v}}_{13}) \\ Rank(\tilde{\tilde{v}}_{21}) & Rank(\tilde{\tilde{v}}_{22}) & Rank(\tilde{\tilde{v}}_{23}) \\ Rank(\tilde{\tilde{v}}_{31}) & Rank(\tilde{\tilde{v}}_{32}) & Rank(\tilde{\tilde{v}}_{33}) \\ Rank(\tilde{\tilde{v}}_{41}) & Rank(\tilde{\tilde{v}}_{42}) & Rank(\tilde{\tilde{v}}_{43}) \\ Rank(\tilde{\tilde{v}}_{51}) & Rank(\tilde{\tilde{v}}_{52}) & Rank(\tilde{\tilde{v}}_{53}) \end{bmatrix}$$

$$\overline{Y}_w^* = \begin{array}{l} \text{Quality} \\ \text{Delivery time} \\ \text{Cost} \\ \text{Flexibility} \\ \text{Geographic location} \end{array} \begin{bmatrix} 8.04 & 6.55 & 8.24 \\ 7.92 & 6.79 & 7.92 \\ 7.28 & 8.23 & 7.28 \\ 5.75 & 5.64 & 5.96 \\ 4.16 & 3.96 & 4.16 \end{bmatrix}$$

Step 5: With eq. (8) and (9), we can obtain the positive ideal solution (PIS) x^+ and the negative ideal solution (NIS) x^-, respectively, where

$$\begin{aligned} x^+ &= (v_1^+, v_2^+, \ldots, v_m^+) = \\ &= \Big(\max\big(Rank(\tilde{\tilde{v}}_{11}), Rank(\tilde{\tilde{v}}_{12}), Rank(\tilde{\tilde{v}}_{13})\big), \\ &\quad \max\big(Rank(\tilde{\tilde{v}}_{21}), Rank(\tilde{\tilde{v}}_{22}), Rank(\tilde{\tilde{v}}_{23})\big), \\ &\quad \min\big(Rank(\tilde{\tilde{v}}_{31}), Rank(\tilde{\tilde{v}}_{32}), Rank(\tilde{\tilde{v}}_{33})\big), \\ &\quad \max\big(Rank(\tilde{\tilde{v}}_{41}), Rank(\tilde{\tilde{v}}_{42}), Rank(\tilde{\tilde{v}}_{43})\big), \\ &\quad \max\big(Rank(\tilde{\tilde{v}}_{51}), Rank(\tilde{\tilde{v}}_{52}), Rank(\tilde{\tilde{v}}_{53})\big)\Big) = \\ &= (8.24, 7.92, 7.28, 5.96, 4.16). \end{aligned}$$

$$\begin{aligned} x^- &= (v_1^-, v_2^-, \ldots, v_m^-) = \\ &= \Big(\min\big(Rank(\tilde{\tilde{v}}_{11}), Rank(\tilde{\tilde{v}}_{12}), Rank(\tilde{\tilde{v}}_{13})\big), \\ &\quad \min\big(Rank(\tilde{\tilde{v}}_{21}), Rank(\tilde{\tilde{v}}_{22}), Rank(\tilde{\tilde{v}}_{23})\big), \\ &\quad \max\big(Rank(\tilde{\tilde{v}}_{31}), Rank(\tilde{\tilde{v}}_{32}), Rank(\tilde{\tilde{v}}_{33})\big), \\ &\quad \min\big(Rank(\tilde{\tilde{v}}_{41}), Rank(\tilde{\tilde{v}}_{42}), Rank(\tilde{\tilde{v}}_{43})\big), \\ &\quad \min\big(Rank(\tilde{\tilde{v}}_{51}), Rank(\tilde{\tilde{v}}_{52}), Rank(\tilde{\tilde{v}}_{53})\big)\Big) = \\ &= (6.55, 6.79, 8.23, 5.64, 3.96). \end{aligned}$$

Step 6: With eq. (10) and (11), we can compute the distance $d^+(x_j)$ between each alternative x_j and the PIS x^+ and we can compute the distance $d^-(x_j)$ between each alternative x_j and the NIS x^-, respectively, where $1 \leq j \leq 3$

$$d^+(x_1) = \sqrt{\sum_{i=1}^{5}\big(Rank(\tilde{\tilde{v}}_{i1}) - v_i^+\big)^2} = 0.292$$

$$d^-(x_1) = \sqrt{\sum_{i=1}^{5}\big(Rank(\tilde{\tilde{v}}_{i1}) - v_i^-\big)^2} = 2.10$$

$$d^+(x_2) = \sqrt{\sum_{i=1}^{5}\big(Rank(\tilde{\tilde{v}}_{i2}) - v_i^+\big)^2} = 2.27$$

$$d^-(x_2) = \sqrt{\sum_{i=1}^{5}\big(Rank(\tilde{\tilde{v}}_{i2}) - v_i^-\big)^2} = 0$$

$$d^+(x_3) = \sqrt{\sum_{i=1}^{5}\big(Rank(\tilde{\tilde{v}}_{i3}) - v_i^+\big)^2} = 0$$

$$d^-(x_3) = \sqrt{\sum_{i=1}^{5}\big(Rank(\tilde{\tilde{v}}_{i3}) - v_i^-\big)^2} = 2.27$$

Step 7: With eq. (12), we can compute the closeness coefficient $C(x_j)$ of each alternative x_j with respect to the PIS x^+, where $1 \leq j \leq 3$

$$C(x_1) = \frac{d^-(x_1)}{d^-(x_1) + d^+(x_1)} = \frac{2.10}{2.10 + 0.29} = 0.878$$

$$C(x_2) = \frac{d^-(x_2)}{d^-(x_2) + d^+(x_2)} = \frac{0}{0 + 2.27} = 0$$

$$C(x_3) = \frac{d^-(x_3)}{d^-(x_3) + d^+(x_3)} = \frac{2.27}{2.27 + 0} = 1.00$$

Step 8: Because $C(x_3) > C(x_1) > C(x_2)$, the preferred rank of the alternatives A1, A2 and A3 is: A3 > A1 > > A2. That is, the best alternative among A1, A2 and A3 is A3.

Supplier selection using fuzzy TOPSIS

At this section for the same supplier selection problem, the necessary calculations were made for solution of the problem by using the steps of Chen's [48] fuzzy TOPSIS method algorithm. In this section, due to just the result table obtained by using of the method will be given, for detailed information regarding to the algorithmic steps and sample applications of the fuzzy TOPSIS method; studies of Zouggari and Benyoucef [3], Yayla et al. [8], Chen [48], Chen et al. [49], Boran et al. [50], Jolai et al. [51], Yildiz and Ergul [52] and Yukseloglu et al. [53] can be reviewed. The results obtained from fuzzy TOPSIS are demonstrated in table 4.

Table 4

d_i^+, d_i^- AND C_i VALUES

	A1	A2	A3
d_i^+	2.32	2.63	2.32
d_i^-	3.57	2.96	3.25
C_i	0.61	0.53	0.58

According to closeness coefficient, alternative supplier firms were ranked as A1 > A3 > A2 from the most convenient to the least convenient. According to this result, it will be appropriate for "X" garment firm to select A1 supplier as it has the highest closeness coefficient value.

After determination the results obtained from the two methods, comparing the results of fuzzy TOPSIS-Interval type 2 fuzzy TOPSIS method are shown in table 5.

Table 5

	Fuzzy TOPSIS		Interval Type-2 TOPSIS	
Supplier	*Closeness Coefficient*	*Ranking*	*Closeness Coefficient*	*Ranking*
A1	0.61	1	0.878	2
A2	0.53	3	0	3
A3	0.58	2	1.00	1

Table 6

	Degree of sensitivity	Ability in uncertainty modelling	Simplicity
Ordinary TOPSIS	Low	Low	High
Fuzzy TOPSIS	Medium	Medium	Medium
Type 2 Fuzzy TOPSIS	High	High	Low

In table 5, when these results obtained from both two methods are considered, the “A1” supplier has become the best supplier among alternatives according to the results obtained from fuzzy TOPSIS method, while the “A3” supplier has been according to the interval type 2-fuzzy TOPSIS method.

Even if it creates an uncertainty on selection of the best supplier due to unlike emergence of the best suppliers according to the each two methods, as can be seen in table 6, being high of the type-2 fuzzy TOPSIS method’s for both ability uncertainties modelling and degree of sensitivity, increases the chance of the “A3” supplier, which ranks first in the interval type 2 fuzzy TOPSIS method, for to be selected as the best supplier.

Accordingly, the selection of the “A3” supplier will contribute significantly to the enterprise in point of both benefit and cost.

CONCLUSIONS

In this study, the interval type 2-fuzzy TOPSIS and fuzzy TOPSIS methods were used in order to select the best one among the three alternative fabric suppliers by “X” garment firm operating in Turkey. The interval type 2 fuzzy TOPSIS method was included in this study with a larger basis relatively. The five selection criteria at the supplier selection model created in line with the opinions of expert persons and the literature review were evaluated according to the linguistic assessment scale present in the algorithms of both methods by three expert persons who are working in the firm. The results obtained from the study were compared according to the suppliers’ closeness coefficients and the suppliers’ rankings were turned out unlike at both methods and it was alleged that it would have been more appropriate to take into account of the results obtained from this method for selection of the best supplier due to having high ability in uncertainties modelling and degree of sensitivity of the type 2-fuzzy TOPSIS method.

This study provide us with a beneficial way to operate fuzzy multi criteria decision-making problems in a more flexible and more intelligent manner due to the fact that it uses interval type-2 fuzzy sets rather than traditional type-1 fuzzy sets to represent the evaluating values and the weights of criteria.

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Effect of sewing speed on the physical properties of firefighter sewing threads

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REZUMAT – ABSTRACT

Efectul vitezei de coasere asupra proprietăţilor fizice ale firelor de cusut ale echipamentului de protecţie pentru pompieri

Acest articol investighează experimental unele proprietăți fizice ale firelor de cusut speciale, ignifuge la diferite viteze de coasere. Firele aramidice sunt utilizate în mod obişnuit pentru coaserea echipamentului pentru pompieri datorită rezistenței mari și a temperaturii ridicate de topire. Sunt folosite 3 tipuri de fire aramidice cu diferite densități liniare, pentru coaserea la diferite viteze situate între 2000 şi 4000 imp./min. Temperatura acului este măsurată la diferite viteze de coasere, iar proprietăţile de rezistenţă la tracţiune ale firelor sunt măsurate înainte și, respectiv, după procesul de coasere. Rezultatele arată că frecarea și abraziunea în timpul procesului de coasere determină o pierdere semnificativă a proprietăților de tracțiune ale firelor, iar temperatura acului se ridică la aproape 300 °C la 4000 imp./min. Imaginile cu microscopul electronic cu baleiaj, înainte și după procesul de coasere, nu evidenţiază prezenţa unor zone de topire, ci doar o deteriorare semnificativă a firului. De asemenea, s-a constatat că viteza mașinii de 2000 imp./min este ideală pentru coaserea echipamentului pentru pompieri datorită proprietăților de tracțiune mai mari și nivelului de productivitate.

Cuvinte-cheie: Kevlar, temperatura acului, Nomex, coasere

Effect of sewing speed on the physical properties of firefighter sewing threads

This article experimentally investigates various physical properties of special fire retardant sewing threads under different sewing speeds. The aramid threads are common for sewing the fire-fighter clothing due to high strength and high melting temperature. 3 types of aramid threads with different linear densities are used for sewing at different speed of 2000 to 4000 r/min. The needle temperature is measured at different speeds of sewing and tensile properties of threads are measured before and after the sewing process respectively. The results shows that the friction and abrasion during the sewing process causes a significant loss to the tensile properties of the threads and needle temperature rises to nearly 300 °C at 4000 r/min of machine speed. The Scanning electron microscope images are taken before and after the sewing process and show no melting spots but significant damage to the yarn. It is also found that machine speed of 2000r/min is ideal for sewing firefighter clothing for higher tensile properties and production.

Keywords: Kevlar, needle temperature, Nomex, sewing

INTRODUCTION

Aramid and other high-strength fiber are extensively used in protective clothing; Kevlar and Nomex registered trademark of DuPont and are widely used for the flame resistant clothing due to high melting points and excellent durability [1–2]. DuPont reported only the tensile properties of filament yarn [3–4]; it is necessary to determine the properties of these Aramid threads after sewing process, where the thread goes under high needle temperature and abrasion and friction during the high-speed sewing process.

During sewing at high speed, the needle thread is subjected to repeat tensile stresses, bending, pressure torsion, wearing and heat. These forces act on the sewing thread repeatedly and the thread has to pass through needle eye, fabric and the bobbin case mechanism 50–80 times before becoming part of the seam [5]. The rubbing at the top of needle eye can cause local abrasion and cutting of the thread. In early research work reported 60% reduction in thread strength after sewing [6–7]. Later a number of researcher observed that there could be 30–40% strength reduction in the cotton thread after sewing [8]. In a recent research on the tensile properties of mercerized cotton thread, nearly 30% strength reduction is reported [9]. Furthermore closer estimation of the seam strength was also possible after considering the loss in sewing thread strength [10–11]. A number of researchers also study the dynamic loading of the sewing thread during high-speed sewing process [12]. The mechanical performance of threads is governed by the properties of constituent fibers and their arrangement. In the course of tensile loading the tension induced by applied strain is transferred to the fibers through the interfacial shear stress, which leads to substantial changes in the yarn structure and fiber mechanical properties [13]. The friction, bending, and compression during the sewing process cause damage/pull-out of surface fibers resulting in a loss in mechanical properties. Heating of the needle cause synthetic fibers to soften or melt, leaving a weakened thread after sewing. Depending on the sewing conditions, maximum needle temperatures range from 100°C–300°C [14]. This high temperature weakens the thread, since thread tensile strength is a function of temperature, resulting in decreased production [15]. The majorities of these loadings are cyclic by nature and therefore cause the fiber fatigue [16].

EXPERIMENTAL PART

Material and method

The fire retardant sewing threads are obtained from company COATS and basic properties like tenacity and breaking extension are measured on the Instron Tensile Tester as per ASTM standard D2256, with a gauge length of 250 mm. The sewing thread tensile strength is measured before the sewing process and then seam is stitched at different speeds for a continuous 10 seconds; the thread is then pulled from the seam precisely by cutting the bobbin thread and each measurement is performed 10 times.

The coefficient of friction is an important factor to understand the effect of abrasion during the sewing process. Thread to metal coefficient of friction is measured for all threads with instrument CTT-LH401 (Company Lawson-Hemphill) according to standard ASTM D-3108 for 100m/min and contact angle of 180°.

The needle temperature is measured at different sewing speeds by inserted thermocouple method [17].

The thread properties are shown in table 1.

Table 1

Serial Number	Material	Thread Count (tex)	Ply	Twist Angle
1A	Spun-Kevlar	40	2	Z/S
1B	Spun-Kevlar	70	3	Z/S
2A	Long staple spun-Meta Aramid	40	3	Z/S
2B	Long staple spun-Meta Aramid	70	3	Z/S
3A	Meta Aramid	40	3	Z/S
3B	Meta Aramid	70	3	Z/S

RESULTS AND DISCUSSIONS

Firstly the metal to thread coefficient of friction for all threads with instrument CTT-LH401 (Company Lawson-Hemphill) according to standard ASTM D-3108 for 100 m/min and contact angle of 180°. Table 2 shows the result of coefficient of friction for the used threads.

The result shows that the Kevlar has the lowest coefficient of friction followed by Meta-Aramid. The long staple Meta-Aramid causes a marginal change in the coefficient of friction compared to staple Meta-Aramid. The lower coefficient of friction is always better for the sewing threads as low abrasion and friction causes less heat and wear of the thread surface.

The threads are used for sewing the 4 layer of 320 g/m^2 denim fabric with different sewing speeds. The stitched thread is pulled from the seam precisely by cutting the bobbin thread and ASTM standard 2256 measures the tensile strength of the thread on tensile tester.

Table 2

Serial number	Coefficient of friction [μ]
1A	0.25
1B	0.28
2A	0.38
2B	0.38
3A	0.4
3B	0.4

Table 3

Threads	Sewing speed [r/min]		
	2000 r/min	2000 r/min	2000 r/min
	Needle temperature [°C] (standard deviation)		
1A	170 (±4)	228 (±4.5)	280 (±5.2)
1B	192 (±6.5)	251.5 (±6.5)	305 (±4)
2A	185 (±6)	238 (±2.5)	295 (±6.5)
2B	196 (±3.5)	258.5 (±7)	315 (±5)
3A	188 (±3.2)	241 (±3.3)	298 (±7)
3B	199 (±6)	263 (±2.7)	321 (±3)

Figure 1 shows the breaking strength of sewing threads (before and after sewing at different speeds) Figure 1 shows that the tensile strength is decreasing dramatically at higher sewing speeds. The damage could be because of the abrasion or the needle heat. The needle temperature was also measured after 10 seconds of continuous sewing at different sewing speed by inserted thermocouple method as shown in table 3.

Table 3 shows that the needle temperature rises with the higher thread count and sewing speed, the highest temperature is observed for the Meta-Aramid thread which is also due to higher coefficient of friction. Anyhow the temperature at 3000 r/min is nearly or above 300°C; which is much above the glass transition temperature of the threads' material but lower than the melting point.

Figure 2 shows that the needle temperature rises linearly with the sewing speeds and tensile strength of threads decreases linearly.

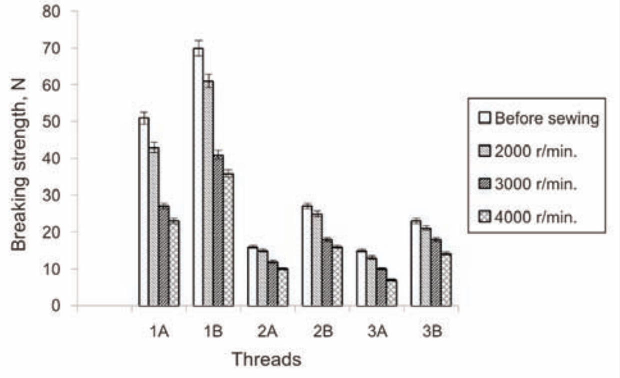

Fig. 1. Breaking strength of sewing threads

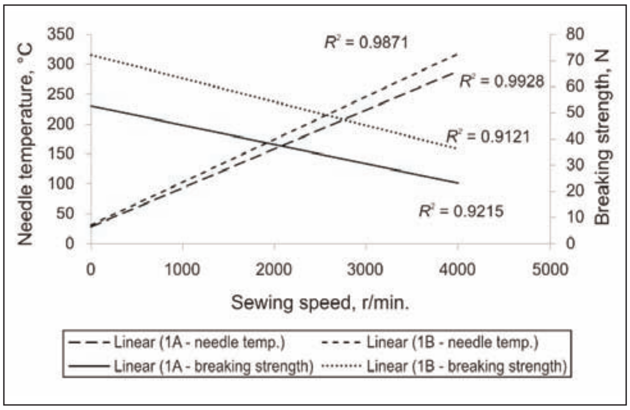

Fig. 2. Needle temperature and breaking strength of threads

To see the effect of abrasion and friction during the sewing process the threads sewed at different speeds are examined under the electron microscope for comparison. The samples are obtained before and after sewing and coated with Platinum under plasma sputtering and examined at 100&50 times magnification.

The SEM images of the Kevlar thread clearly shows that the yarn surface is badly damaged at higher speed and causes a dramatic decrease of the tensile strength, but no burn or melted sport is observed on the thread surface as Kevlar has higher melting temperature as compared to needle temperature but still this needle temperature is higher than the glass transition temperature of the Kevlar and may have

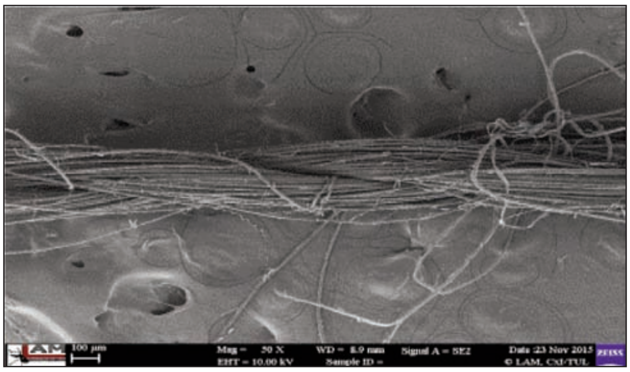

Fig. 3. SEM image of sample 1A before sewing

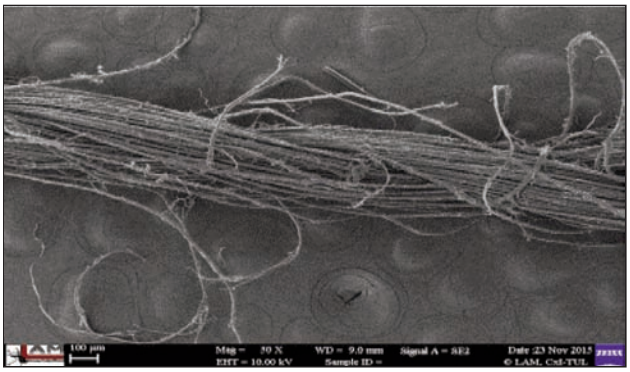

Fig. 4. SEM image of sample 1A after sewing (4000 r/min)

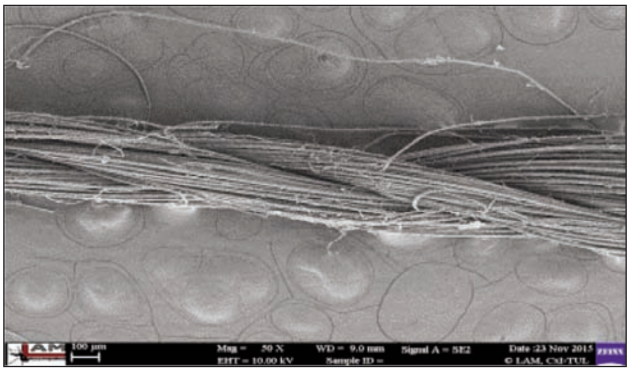

Fig. 5. SEM image of sample 1B before sewing

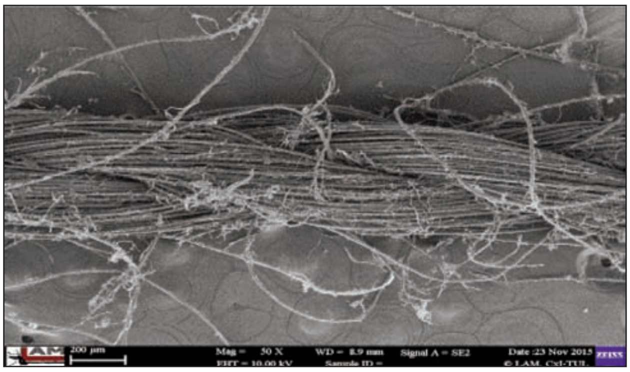

Fig. 6. SEM image of sample 1B after sewing (4000 r/min)

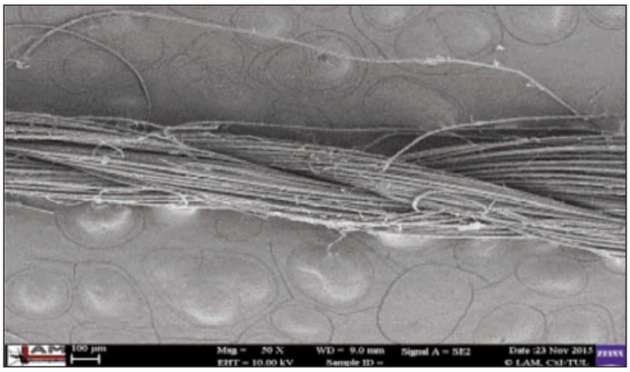

Fig. 7. SEM image of sample 2A before sewing

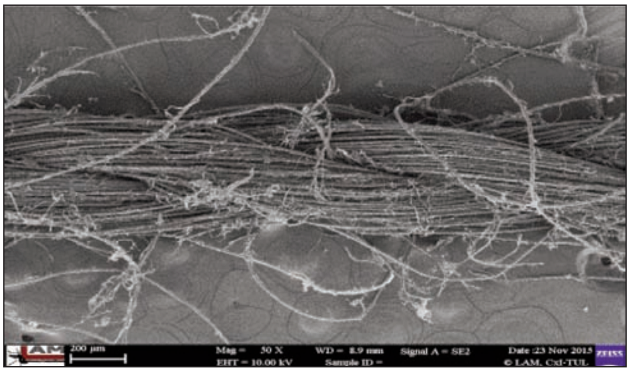

Fig. 8. SEM image of sample 2A after sewing (4000 r/min)

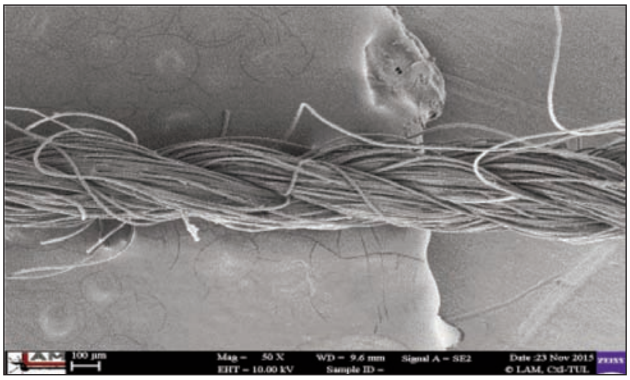

Fig. 9. SEM image of sample 3B before sewing

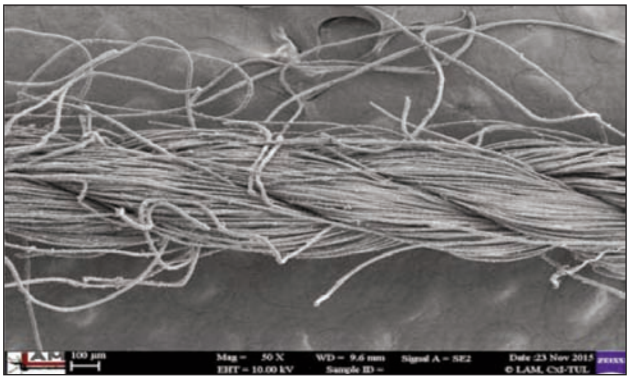

Fig. 10. SEM image of sample 3B after sewing (4000 r/min)

causes the internal changes in the Kevlar structure. The images of sample 2A are shown before and after the sewing process under SEM in figure 7 and figure 8.

The selected samples presented here show the effect of sewing speed on the Meta-Aramid, the surface damage is visible but comparatively less as compared to the Kevlar thread. Even though the coefficient of friction of Kevlar thread is less as compared to the Meta-Aramid but visible friction damage is much higher, which can be because of forward and backward motion of the thread in the needle eye and may have caused the Kevlar fibers to open and get more damage.

CONCLUSIONS

The following is concluded from this research:

The tensile strength of the sewing threads significantly decreases with the speed of the machine. The abrasion and friction during high-speed sewing are causing yarn and fiber breakage. There is no melting spots observed on the sewing thread after sewing process but the needle temperature reaches 300°C; which is much higher than the glass transition temperature of Kevlar and Meta-aramid fibers.

The Electron microscope images shows the effect of sewing speed on the sewing thread; the damage is more for the Kevlar and least for the long-staple meta aramid fibers. The sewing speed should not be higher than 2000 r/min when working with aramid threads, as there is significant tensile strength loss at higher speeds.

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Revealed comparative advantage and competitiveness in Romanian Textile and Clothing Industry

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REZUMAT – ABSTRACT

Avantaj comparativ aparent și competitivitate în industria de textile şi de confecţii din România

Această lucrare investighează competitivitatea industriei textile şi de îmbrăcăminte din România în relaţie cu piaţa mondială, care utilizează trei indicatori ai avantajului comparativ aparent din perioada 1990–2014. Toți indicatorii evidenţiază faptul că România a avut un avantaj comparativ și o competitivitate a produselor de îmbrăcăminte în creștere până în 2003 și în declin constant după acest an. În conformitate cu RCA Balasa din anul 2003, produsele textile au, în general, un avantaj comparativ. Utilizarea altor doi indicatori conduce la alte concluzii: produsele textile prezintă un dezavantaj comparativ pe întreaga perioadă analizată. Indicii RCA, în ciuda limitelor, oferă un ghid util pentru estimarea avantajului comparativ, oferă o perspectivă mai cuprinzătoare în ceea ce priveşte competitivitatea industriei de textile-confecţii din România și implicațiile acesteia asupra comerțului din economia mondială.

Cuvinte-cheie: avantaj comparativ, industria de textile și de îmbrăcăminte, avantaj competitiv aparent, Indicele Balassa, Indicele Lafay

Revealed comparative advantage and competitiveness in Romanian Textile and Clothing Industry

This paper investigates the competitiveness of Romanian textile and clothing Industry in relation to that of the Global Market employing three indices of revealed comparative advantage for the period 1990 to 2014. All indices underline the fact that Romania had a comparative advantage and competitiveness of the clothing products growing up till in 2003 and declining steadily after this year. According to RCA Balasa since 2003 the textile products have a competitive advantage. The use of the other two indicators leads to other conclusions: textile products have comparative disadvantage during the entire analysed period. RCA indices, despite their limitations, provide a useful guide to underlying comparative advantage and offer a further insight into the competitiveness of Romanian textile and clothing industries and the implications for trade in the global economy.

Keywords: comparative advantage, Textile and Clothing Industry, Revealed Competitive Advantage, Balassa Index, Lafay Index

INTRODUCTION

The textile and clothing sectors are key sectors for economic enhancement and its development strategy and maintaining the competitiveness of this sector should be part of Romania's economic priorities. Furthermore, the clothing industry is a branch with significant tradition, ranking third place in Romania’s exports and fourth place in the EU clothing exports [1]. As part of a general restructuring trend, observed in most of Western countries, to deal with sharp competitive pressures and increased market volatility, firms in vulnerable sectors like textile and clothing manufacturing sought after ways of reform their production processes, rationalizing their supply chain and inventory management, downsizing, and seeking improved flexibility. In order to understand the causes of the recent situation and to try to speculate on the future of the textile industry, it is relevant to know the comparative advantage which contributed to Romania’s competitiveness.

SCOPE AND OBJECTIVE OF THE RESEARCH

Given the importance of Romanian textile and clothing industries in the Romanian economy, this study has the object of scientific research – export specialization and export competitiveness in the textile and clothing sector. The main aim of this paper is to present the analysis of indices of comparative advantage of commodities of Romanian textile and clothing industries during 1990–2014.

Research tasks of the paper are to analyze the conceptions and main issues of measuring export competitiveness on the basis of contemporary theories, to perform the analysis of modified RCA (revealed comparative advantage) indexes of textile and clothing industries of Romanian during 1990–2014. In the last few years, it has been noted a renewed interest in empirical works on the sources of comparative advantages develop competitiveness indexes [2, 3, 4]. The choice of the right index depends on many circumstances; our opinion is that in the current context of increasing intra-industry trade, a careful assessment of international comparative advantages requires to take into consideration not only exports but also imports. Thus, we used the Revealed Comparative Advantage Index (RCA) of Balassa (1965), the Vollrath’s revealed competitiveness (RC) and a (simplified) version of the index suggested by Lafay

(1992), which uses commodity-specific net exports as a measure of comparative advantage [5–7]. The analysis is based on the annual time series data on textile and clothing exports and imports, obtained from the World Trade Organization.

Methodology and methods

The methods of the scientific research that we have used in the paper included: summarizing of literature and scientific analysis, mathematical calculations, comparative analysis of statistic indexes.

The paper is organized in four parts: the first part analyses the stand of exports and imports of textiles and clothing in the Romanian trade. The second part outlines the theoretical basis of the RCA index and also provides a brief literature survey. Alternative measures of RCA indices, a comparison and our approach are presented in the third part. The final part draws some conclusions based on the findings. Romanian RCA* indices are analyzed over the study period.

TRADE DEVELOPMENTS IN THE ROMANIAN TEXTILE AND CLOTHING INDUSTRY

This chapter analyzes the evolution of the Romanian textile and clothing trade and shows the position held by Romania's total trade products of these industries.

The Romanian textile and clothing industry has a venerable history and tradition of supplying good quality products and despite increasingly ferocious global competition and significant relocation of manufacturing to low-wage countries it continues to represent one of Romanian's major industrial sectors [9].

Romania exports clothing products totalling $ 3626.65 million (in 2014) decreasing compared to 2003–2008 when its exports peaked in 2004 export value totalling $ 4780.87 million. Analysing the evolution of imports of clothing it can be noted a tendency for their annual increase of $ 26 million in 1990 to a peak recorded in 2008 namely $ 1274.40 million. However the trade balance of trade in textiles remained positive throughout the period analyzed.

The decrease of clothing exports is best reflected in the share of total Romanian exports, which is currently only 5.20 % (in 2014) dropping from 24.42 % in 2001 (figure 2). Regarding the share of garment imports in total Romanian statistical data show that they have not changed so much throughout the analyzed period – the highest increase being registered with the change of political regime in Romania in 1990 (figure 3).

Analysis of the evolution of textile imports highlights similar trend with export of clothing. This evolution is given mainly by the type of garment production in Romania – lohn system which works as outward processing trade (OPT). Evolution of textile exports is mainly due to the significant reduction of productions in the textile industry. The growth from last few years is determined by manufacturing products with high added value-like technical textiles. These evolutions of exports and imports of textiles are reflected in a negative trade balance of this sector (figure 4).

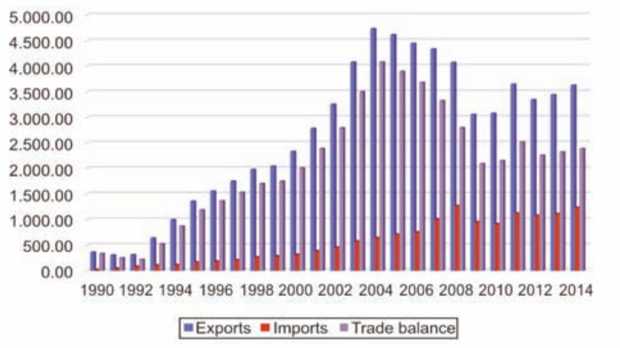


Fig. 1. Evolution of Romanian clothing trade, during 1990–2014 (*Source: INSSE*)

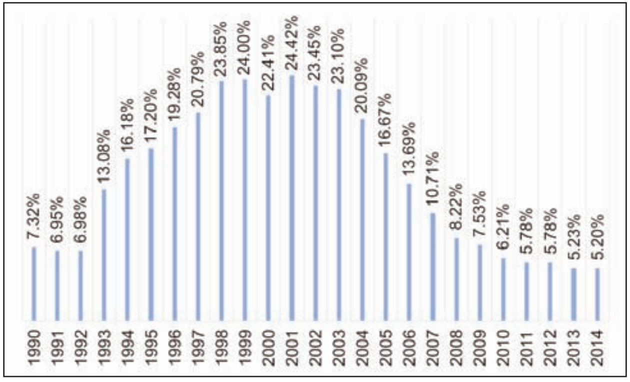


Fig. 2. The percentage held by clothing exports in total Romanian exports (*Source: Calculated by the authors according to the INSSE dates*)

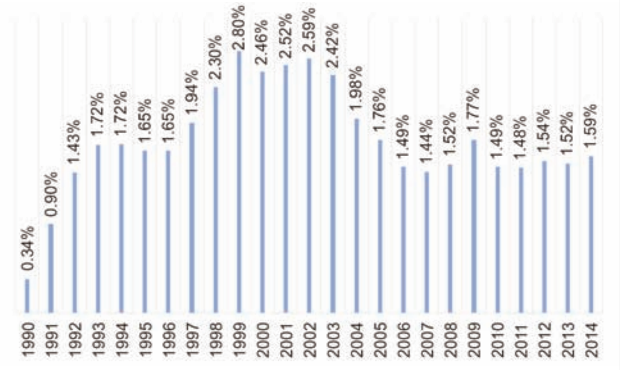


Fig. 3. The percentage held by clothing imports in total Romanian imports (*Source: Calculated by the authors according to the INSSE dates*)

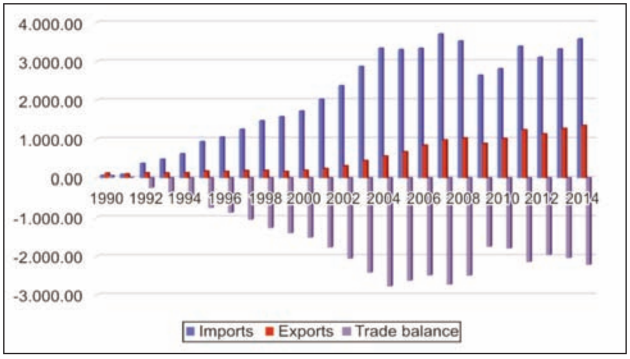


Fig. 4. Evolution of Romanian textiles trade, during 1990–2014 (*Source: INSSE*)

Table 1

Activity	Average number of employees (thou persons)		
	Total	Men	Women
Industry – total	1296	741	555
Manufacturing	1064	562	502
Manufacture of wearing apparel	145	20	125
Manufacture of textiles	30	8	22

Source: INSSE

Despite significant decreases of Romanian exports of clothing, this industry remains a major employer, the number of employees in this area representing 16.44 % of total employment in manufacturing and 11.18 % of all employees in the Romanian industry. The number of employees in the textile industry is much lower, 30,000 employees – representing 2.81 % of total employment in manufacturing (table 1)

LITERATURE REVIEW AND RECENT DEVELOPMENTS

Measuring Revealed Comparative Advantage

The evolution of trade specialization is a phenomenon that reflects structural changes in the entire economic system of a country and needs time, since the comparative advantages in trade cannot be achieved in a short time, especially since they are structurally by definition. The concept of comparative advantage is grounded in conventional trade theory and is widely used in modern economic literature to evaluate the patterns of trade and specialization of countries in commodities which have a competitive advantage. There are many ways to measure comparative advantages [10], but the most used in the empirical work is still the index of revealed comparative advantage (RCA) by Balassa (1965) and various modifications thereof.

The index of revealed comparative advantage, first formulated by Bela Balassa (1965) suggested that country's revealed comparative advantage in the trade of a certain industry is assessed by the share of that industry in the country's total exports relative to the industry's share in total world exports of manufactures [5]. Actually, the Balassa index (RCA) gives an indication of the industries in which a particular country may have a comparative advantage in. A country is said to have a revealed comparative advantage in a good when the share of that good in the country's exports is bigger than the corresponding share of world export of that good in total world exports. In essence, the RCA is really a measure of specialization. Balassa outlined that it is difficult to measure competitiveness due to the lack of comprehensive data on factor costs, so the most widely accepted indirect approach is the revealed comparative advantage (RCA) index, which reveals the comparative advantage of a nation from its past trade data. The export results could be used to reveal the comparative advantage of a particular country in the absence of comprehensive data on factor costs. The pattern of commodity exports reflects relative costs as well as differences in non-price factors that can be expected to determine the structure of export.

$$RCA_{ij} = (X_{ij} / X_{it}) / (X_{nj} / X_{nt}) \qquad (1)$$

Where: X is exports, i is the country, j is the commodity/industry, n is the world or a set of countries, and t is all product groups.

As per Balassa if RCA index is greater than 1 (RCA > > 1) indicates that the country has a comparative advantage in the commodity/ industry and has a comparative disadvantage when RCA < 1.

In a later work, Balassa (1986) restricted his analysis to manufactured goods only, as distortions in primary products, such as subsidies, quotas and special arrangements would not reflect the real comparative advantage. RCA index represents post trade relative prices and a prevailing factor as well as product market distortions.

Since first proposed by *Balassa* (1965), the definition of RCA has been revised and modified such that a plethora of measures now exists. Some specifications aim to measure RCA at the global level (eg. *Vollrath,* 1991), others at a regional or sub-global level (as in Balassa's original specification), whilst some restrict the analysis to bilateral trade between just two countries or trading partners.

There is some criticism of this method of analysis of competitiveness. The Balassa Index has been criticized for its poor empirical distribution characteristics and for taking only the exports into consideration while ignoring the imports [11–12]. Another objection is the fact that if the country has a "comparative disadvantage" the index ranges from zero to one, whereas if it has a "comparative advantage", the index ranges from one to infinity [13].

RCA was modified by Vollrath (1991) in order to avoid double counting between pairs of countries. *He* offered three alternative specifications of revealed comparative advantage. One of these measures is the relative trade advantage *(*RTA), which accounts for imports as well as exports. It is calculated as the difference between relative export advantage (RXA), which calculates the ratio of a country's export share of a commodity in the international market to the country's export share of all other commodities, and its counterpart, relative import advantage (RMA).

$$RXA_{ij} = (X_{ij} / X_{it}) / (Xn_j / Xn_t) \qquad (2)$$

The relative import advantage (RMA) index is similar tothe RXA, but relates to imports (M) rather than exports:

$$RMA_{ij} = (M_{ij} / M_{it}) / (Mn_j / Mn_t) \qquad (3)$$

In this case, an RMA index of less than 1 indicates revealed comparative advantage and thus higher competitiveness.

$$RTA_{ij} = RXA_{ij} – RMA_{ij} \qquad (4)$$

Vollrath's second measure is simply the logarithm of the relative export advantage (ln RXA); and his third measure is *revealed competitiveness* (RC), defined as:

$$RC_{ij} = \ln (RXA_{ij}) - \ln (RMA_{ij}) \qquad (5)$$

When RXA and RMA are compared in logarithmic form, they are symmetric at the origin. According to Vollrath a positive value of the three measures, RTA, ln RXA and RC,indices reveals a comparative advantage, while a negative value reveals a comparative disadvantage. The index also has some limitations: it is sensitive to very small values of exports and imports.

Another attempt to overcome the empirical weakness of the Balassa index was made from G. Lafay (1992), an index that combines together trade and production variables. The Lafay Index is a measure of country's trade specialisation with regard to a specific product. The existence of a comparative advantage is revealed if the index assumes positive values, whereas negative values show de-specialization. The greater the absolute values, the higher the degree of specialization/de-specialization [6].

This indicator is based on evaluating normalized trade balance of the country *i* in a particular product *j*. Normalized trade balance is measured as a ratio of the trade balance for the product to the total value of trade, i.e.:

$$LFI_j^i = 100 \cdot \frac{x_j^i - m_j^i}{x_j^i + m_j^i} - \frac{\sum_{j=1}^{N} (x_j^i - m_j^i)}{\sum_{j=1}^{N} (x_j^i + m_j^i)} \cdot \frac{x_j^i + m_j^i}{\sum_{j=1}^{N} (x_j^i + m_j^i)} \qquad (6)$$

Where: x_j^i is the country's (*i*) export on this particular product (*j*) and m_j^i is the import.

A positive value for the index for product *j* indicates a country's comparative advantage and high level of specialization on the associated product. A negative value, quite the reverse indicates a comparative disadvantage and low degree of specialization in that product. Given this definition, Lafay Indices maintains symmetry across all products in the country and the sum of for all sectors of a given country must be zero. This specialization index of a product *j* in country *i* is thus related to the deviation of the product normalized trade balance and the country's overall trade balance and its share of trade.

Although RCA indexes are relative measures, so results should be treated with caution and with understanding of their limitations, an analysis of revealed comparative advantage of the industrial sector is helpful in analyzing structural change in export specialization.

Recent studies of competitiveness of textiles and clothing sector

The RCA index and other modified indices have been widely used in cross country and product specific comparisons to assess competitiveness.

Havrila and Gunawardana (2003) analysed Australia's comparative advantage and competitiveness in textile and clothing (TAC) industries using Balassa's revealed comparative advantage index and Vollrath's measures of competitiveness. The analysis based on Balassa's indices shows that Australia has a strong comparative disadvantage in textiles and clothing as aggregate commodity groups, but there is comparative advantage in sub-categories of some special textile products [14]. The analysis based on Vollrath's indices shows that Australia is not competitive in the world market with respect to aggregate commodity groups of textiles and clothing. Different from our work, they used the Grubel-Loyd index of intra-industry trade.

Małgorzata Koszewska evaluated the competitiveness of Polish protective clothing manufacturers based on Balassa and Vollrath's indicators. The analysis is built on statistical data from 1999–2004 according to the appropriate codes of the Eurostat Combined Nomenclature [15].

Turkey's comparative advantage and competitiveness in the textile and clothing industries are analysed also by employing Balassa's revealed comparative advantage index and Vollrath's indices of competitive advantage for the period of 1988–2008 in the enlarged EU market [16]. Erkan studied the international competitiveness in Turkey's export of textile and apparel sector between the years 1993–2009. In this context, Balassa Index, Vollrath Index and Export-Import Rate Index were calculated with reference to 59 product group of textile sector and 37 product group of apparel sector [17].

Karaalp and Yilmaz analysed the comparative advantage of four countries in the world: Bangladesh, China, Germany and Turkey with respect to the US and the EU-15 textiles and clothing markets by using Balassa's revealed comparative advantage index for the period 2000–2010 [18].

Using Balassa index Lalit (2013) calculated revealed comparative advantage of export performance of clothing sector of India and Bangladesh [19] and Shahab and Mahmood (2013) estimated RCA of leather industry and leather products of Pakistan, China, India and Iran, for the period of 2002 to 2009 [20].

With the help of Balassa's index the comparative advantage and intra-industry trade of the major exporter countries of Eastern Europe in the global textile and clothing markets was analyzed for the period 2002–2013. Czech Republic, Hungary, Poland, Romania and Turkey are the top five exporters among Eastern European countries in the global textile market. The results have shown that Turkey is the only one among the countries selected to have comparative advantage in the global textile market and Romania joins Turkey in comparative advantage in the world's clothing market. The comparative advantage of all the countries selected in the global clothing market presents a stronger decline compared to that in textiles [21].

APPLICATION OF BALASSA'S, VOLLRATH'S AND LAFAY INDICES TO ROMANIAN TEXTILES AND CLOTHING

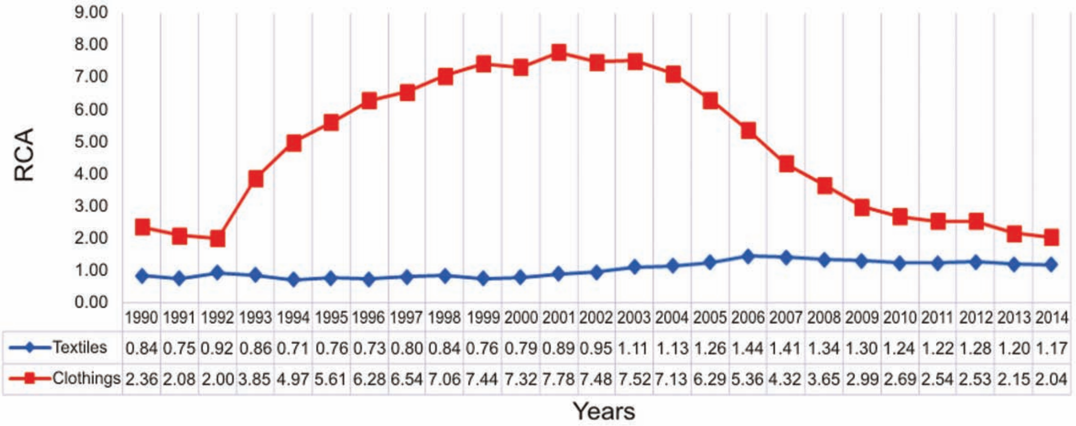
In this section comparative advantage in the textiles and clothing industries was calculated using indexes of developed by Balassa, Vollrath and Lafay. The period of our study begins with the start of the Romanian economy transition from a centrally planned to a market economy. Starting with this date privatization of companies begins in the two sectors analyzed, leading all firms that currently to be privately owned. Because the clothing industry is more labour-intensive compared to the textile sector, which is relatively capital-intensive these sectors develop in different directions namely while in the garment sector, private investment significantly increased, investment in the textile sector are insignificant in the early 90s, most of them made by state [22].

Geographical locations of the Romanian country, cheap and qualified workforce are just some of the factors that favoured increased lohn production of clothing and implicit export growth. Termination of the Agreement on Textiles and Clothing (ATC) and all restrictions thereunder on January 1, 2005 it was reflected in the evolution of Romanian trade with clothing products – who was continuously decreasing after this date. The price of Chinese products has determined increased imports of clothing from this country and has exerted a pressure on domestic firms in this industry because the increase in imports of clothing was not only in Romania but worldwide which has determined reduced orders from the main partners of Romania.

In this situation a lot of the Romania clothing companies have reoriented their business to the domestic market, a fact that led to the growth of Romanian textile imports. The global economic crisis that began in August 2008 has had an impact both on imports and on exports of Romanian clothing and textiles – that dropped in future years.

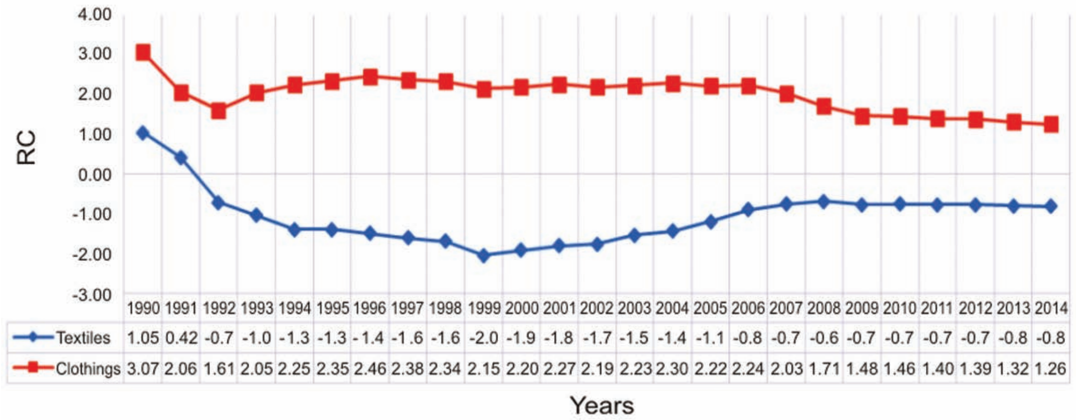
In this context according to Balassa the garment sector indices recorded annual increases competitiveness by 2004 and after this year a continuous decline of this indicator. Furthermore according to the Hinloopen proposed classification in the period 1994–2007 Romania has a strong competitiveness for the clothing sector comparing to other years when the competitiveness is moderate [23].

During the same analysed period we find that textiles are characterized by comparative disadvantages until 2002, after which the competitiveness has slightly increased but it remains at a low level.



	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014
Textiles	0.84	0.75	0.92	0.86	0.71	0.76	0.73	0.80	0.84	0.76	0.79	0.89	0.95	1.11	1.13	1.26	1.44	1.41	1.34	1.30	1.24	1.22	1.28	1.20	1.17
Clothings	2.36	2.08	2.00	3.85	4.97	5.61	6.28	6.54	7.06	7.44	7.32	7.78	7.48	7.52	7.13	6.29	5.36	4.32	3.65	2.99	2.69	2.54	2.53	2.15	2.04

Fig. 5. Evolution of Romanian RCA (Ballasa indices) for clothing and textiles in the period 1990–2014. Calculated by the authors according to the WTO dates



	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014
Textiles	1.05	0.42	-0.7	-1.0	-1.3	-1.3	- 1.4	-1 .6	-1.6	-2.0	-1.9	-1.8	-1.7	-1.5	-1.4	-1.1	-0.8	-0.7	-0.6	-0.7	-0.7	-0.7	-0.7	-0.8	-0.8
Clothings	3.07	2.06	1.61	2.05	2.25	2.35	2.46	2.38	2.34	2.15	2.20	2.27	2.19	2.23	2.30	2.22	2.24	2.03	1.71	1.48	1.46	1.40	1.39	1.32	1.26

Fig. 6. Evolution of Romanian RC (Volrath indices) for clothing and textiles in the period 1990–2014. Calculated by the authors according to the WTO dates

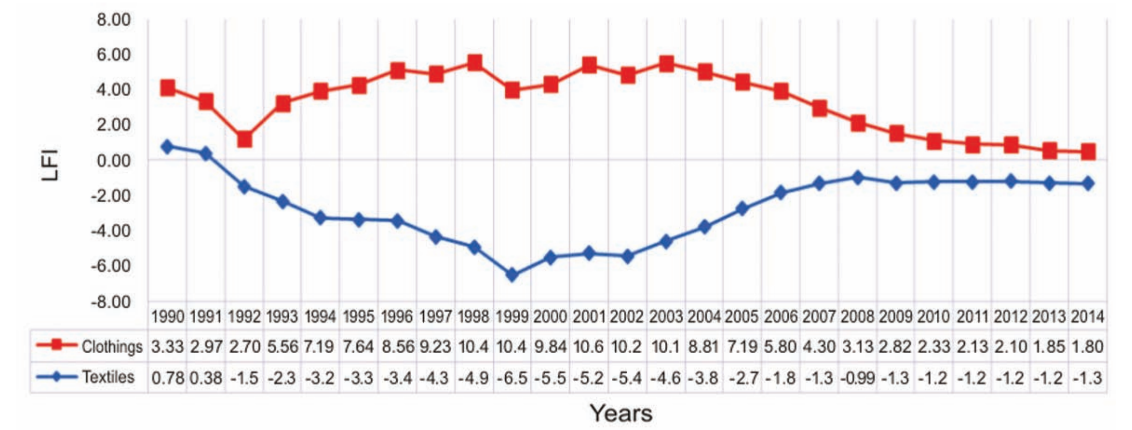


Fig. 7. Evolution of Romanian LFI (Lafay index) for clothing and textiles in the period 1990–2014. Calculated by the authors according to the WTO dates

Refocusing manufacturers of textiles for technical textiles and manufacturers of clothings toward domestic market the changes in recent years in these sectors can be explained.

According to the indicator proposed by Vollrath (Revealed Competitiveness) who includes the effects of imports and exports of the country, RC for clothings have positive values in all the analyzed period that is equivalent to a competitive advantage of the products made by this industry. Even though textiles have a competitive disadvantage in the analyzed period it can be observed a decrease in the last years – figure 6.

In the following part Lafay indicator was calculated. This indicator is based on evaluating normalized trade balance of the country in a particular product. As we have stated earlier a positive value for the Lafay's index indicates a country's comparative advantage and high level of specialization on the associated product and a negative value, on the contrary, indicates a comparative disadvantage and low degree of specialization in that product. Analyzing the obtained results, we found that Romania has comparative advantage and high level of specialization on clothing and has comparative disadvantage and low degree of specialization in textiles products.

CONCLUSIONS

The comparative advantage of Romania's competitiveness in the global market for textile and clothing products were analyzed by RCA – Balassa indices, RC – Vollrath indices and LFI – Lafay index, developed during 1990–2014.

According to all three calculated indices, Romania established increasing values of comparative advantage and Competitiveness of the clothing products until 2003. After that Romania continued to present comparative advantages of the clothing products, but the values of the indicators begin to decrease annually, with a minimum in 2014.

According to RCA Balasa since 2003 the textile products have a competitive advantage. The use of the other two indicators lead to other conclusions: textile products have comparative disadvantage on the entire analysed period.

Increasing competition from developing countries, the elimination of the last set of quotas of the Agreement on Textiles and Clothing (ATC) and all restrictions thereunder on January 1, 2005, financial crisis that started in 2008 and an increase in the exchange rate are the main factors for the evolution of comparative advantage and competitiveness of Romanian clothing and textiles product.

In this context, the Romanian Textile and Clothing Industry must take measures to increase labor productivity, to continue transition from outsourcing production at own brand and promote their brands on the international markets, to develop sectors that involved innovation, fashion knowledge with respect to environmental concerns and sustainability issues. Also Romania should capitalize more of its geographical position, membership of the EU market, infrastructure in garment sector, highly skilled workforce.

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Globalization as a factor of influence on the R&D activity and the case of the textile industry in Romania

MARGARETA STELA FLORESCU FLORENTINA IVANOV

REZUMAT – ABSTRACT

Globalizarea ca factor de influență asupra activității de C&D. Cazul industriei textile din România

Modelele teoretice de analiză a efectelor globalizării menționează faptul că transferul de cunoştinţe este favorizat de combinaţia dintre expertiza externă și cunoștințele existente pe plan intern. Un rezultat posibil este că dezvoltarea poate fi stimulată în mod indirect prin politici care să încurajeze transferul de cunoștințe între țări și regiuni prin intermediul investițiilor străine directe, parteneriatelor, mobilității și schimbului de informații. Prin acest articol se testează validitatea acestei ipoteze utilizând date din România. Mai precis, se evaluează dacă nivelul globalizării regiunilor macroeconomice din România are un impact semnificativ asupra activității de cercetare și dezvoltare a regiunilor respective. Ulterior, se analizează condițiile esenţiale ale acestui model prin ilustrarea cazului industriei textile şi de îmbrăcăminte din România. Rezultatele acestei analize pot servi drept factor de decizie pentru elaborarea politicilor de dezvoltare regională.

Cuvinte-cheie: transfer de cunoștinţe, transfer de competențe, C&D, mobilitate, industria textilă

Globalization as a factor of influence on the R&D activity and the case of the textile industry in Romania

The theoretical models analysing the globalisation effects suggest that knowledge transfer is fostered by the combination between foreign expertise and domestic knowledge. One possible outcome is that development can be stimulated in an indirect way by policies that encourage transfer of knowledge between countries and regions through FDI, partnerships, mobility and exchange of information. This article will test this hypothesis on data from Romania. More precisely, we will test if the level of globalisation of the Romanian macroeconomic regions has a significant impact over the R&D activity of the regions. Afterwards, we will analyse an essential condition of this model by illustrating the case of the Romanian textile and clothing industry. The results of the test would be a guiding factor in regional development policies.

Keywords: knowledge transfer, transfer of competencies, R&D, mobility, textile industry

INTRODUCTION

The EU Strategy 2020 mentions seven new modalities to raise growth and employment, among which creating an innovation-friendly environment was a top priority. From this point of view, the 2014 report of the European Commission illustrates a week position of Romania compared with the EU average [1]. Romania ranks the last one in the EU after the average public expenditure on R&D as percentage of GDP in 2007–2012, proving lack of real support from the public authorities for research and development activities. According to the 2015 innovation report, the Romanian population continues to be unaware of the value that the R&D sector has for enhancing competitiveness and securing high-quality jobs [2]. This article aims at rising the interest in R&D activities by underlining some of the factors influencing it.

FACTORS INFLUENCING R&D

In an open economy, foreign companies bring new products and strategies to the local economy. By adapting them to the domestic circumstances, they create new outputs, and engage in the innovation process. Ultimately, they test their limits, but also put pressure on the limits of the market. This is not a one-way transfer of knowledge and inspiration, but rather a mutual benefit [3].

The business sector is not the only means of innovation by pooling together diverse knowledge. International research projects, the inflow and outflow of students engaged in mobility programs and teachers' professional stages abroad are conductors of innovative ideas in the education and public research system. In the same time, the distribution of international publications and global access to data are all pathways for knowledge and ideas transfer and thus enablers of innovation in the private sector, with potential for entrepreneurship.

In this article, we will show that the internalization of the Romanian macroeconomic NUTS 2 regions relates to the R&D activities in the country. After studying the general model, we will look at the textile industry in particular. The textile industry in Romania received a high level of FDI, but the foreign presence brought a very low level of knowledge transfer, as most of the economic activities were conducted by an unfavourable type of contract, which came to be called by the name of "lohn". This type of economic contract stipulates that the entire innovation activity, market research, design, strategy and business model is created abroad and only the low-skilled fabrication process is manufactured in Romania. The materials used in the manufacture process are usually imported as well.

Estimators of the general model

Our analysis focuses on the eight macroeconomic regions of Romania. This general model is based on the article presented at the 8th International Management Conference "Management Challenges for Sustainable Development" in 2014 [4]. In order to assure the relevance of the results for the policymaking, we considered only data starting one year after the membership in the European Union. Delayed effects of membership are thus accounted for by leaving one year for adaptation. The R&D activity of the regions is estimated by the percentage of the total population engaged in R&D activities (*y*). The knowledge and innovation channels linking the domestic business sector to the external sector are estimated by the level of foreign direct investments (*x*1). The foreign knowledge inflow in the education system is estimated by the number of students and teachers engaged in mobility programs, reported to the regional population (*x*2).

The access to international information is estimated by the proportion of the population who used the internet at least once a week, including every day (*x*3). This last variable has a mixed composition, referring not only to the population using the internet for research or for obtaining ideas with the potential of becoming innovative products or strategies, but also to the use of internet for any other purposes. We expect actually that a significant part of the population with internet access uses it for other purposes, like socialisation, entertainment, networking, shopping etc. Indeed, the Eurostat regional information society statistics shows that 66% of the individuals who used the internet in one week's time also used it for networking purposes, like creating user profile, or using social media in 2011. A similar study performed in 2014 shows an average of 74%. This proves that most of the individuals use the internet for other purposes than research and this trend is getting stronger. The available information does not allow us to differentiate on the exact purpose. We are aware of the "noise" in the data, but in the same time, we do not delimitate what kind of knowledge transfer is useful for the innovative process or what kind of specific interactions with the external sector are influencing the R&D process. There are sufficient grounds to consider that some of the networking activities might actually support the R&D employment from a cognitive point of view, but this discussion is beyond the purpose of this article. Therefore, we will include this variable in our model under the doubt that it might not be very significant for our analysis.

We can summarize the discussion above in the following form:

$$y_{it} = \beta_0 + \beta_1 \cdot x_{1it} + \beta_2 \cdot x_{2it} + \beta_3 \cdot x_{3it} + u_{it} \qquad (1)$$

where $i = 1,\ldots,8$ is the individual dimension representing the number assigned to each macroeconomic region, $t = 2008,\ldots,2012$ is the time dimension and u – the error term [4].

Data collection

The data included in the model is calculated as an average for each of the eight NUTS2 macroeconomic regions of Romania. The period analyzed is 2008–2012, starting one year after the membership of Romania in the European Union in order to take into consideration lagged effects.

The data was collected and computed using databases from the Romanian National Institute of Statistics, Eurostat, the Romanian National Bank, the Romanian Ministry of Finance, UNCTAD and the National Office of Patents. The number of students and teachers engaged in mobility programs was computed by manually summing up the reported data from all universities located in each of the regions and include both teachers and students engaged in study, research or practice stages on a temporary basis.

Summary of data

Each variable includes one value for each year (from 2008 through 2012) for each macro-economic region (numbered from 1 to 8 as it follows: 1 – Nord-West, 2 – Center, 3 – Nord-East, 4 – South-East, 5 – South, 6 – Bucharest and Ilfov, 7 – South-West, 8 – West). There are 40 observations in total.

The independent variables in the model are the following:

- percentage of the population using the internet each week, based on data from Eurostat;
- total employees in R&D activities by NUTS 2 regions at the end of the year, retrieved from the National Institute of Statistics;
- foreign direct investments in each region per capita, in mil. Euros;
- number of students and teachers who fulfilled mobility stages, computed for each region as a percentage of the total population by adding the data reported by each university in the NUTS2 regions, based on statistics published by The Lifelong Learning Programme and UNCTAD. Based on this data, we computed the dependent variable rate of the students and teacher engaged in mobility programs;
- number of resident population in each region at the beginning of each year, based on data from Eurostat database. All the variables above are compared to the corresponding regional population in order to account for regional differences and to make the values comparable;

The dependent variable of our model is the percentage of the regional population working as employees in the R&D sector.

The steps followed for estimating our model were the ones described by Stănilă, Andreica and Cristescu [5]. After declaring the variables as panel dataset, with the identification variable A and the time variable B, the econometrics program recognized a strongly balanced panel (the same number of years for each region) with complete observations for each panel from 2008 through 2012, meaning 40 observations.

Taking in consideration our assumptions, we expected a positive sign for all the three explanatory variables included.

Linear panel data regression model

If we assume fixed effects, we impose time independent effects for each variable possibly correlated with the predictor variable. This means that we can control for unobserved heterogeneity of the regions, when this heterogeneity is constant over time and correlated with the R&D activity [6]. For choosing between fixed and random effects model, we conducted a Hausman test [7]. The test conducted on our data showed a statistically insignificant P-value of 0.6016, meaning that it is better to use fixed effects than random effects [8]. Indeed the Hausman test reported fixed effects model to be consistent under Ho and Ha and random effects model to be inconsistent under Ha and efficient only under Ho. Using the fixed effects model makes sense, as most of the differences between the regions are anyhow accounted for in our model by adjusting all variables by the population size.

Additional, we considered robust standard errors for taking in consideration heteroskedasticity problems. Conducting a serial correlation test and a test for group heteroskedasticity indicated that a robust estimation was more appropriate.

Results

The regression analysis with robust standard errors for the 8 clusters of data shows that all the three explanatory variables considered are statistically significant at 1%. They are also jointly significant. The overall fit of the model (R-sq) is very good, showing that the foreign direct investments (FDI), the mobility of students and teachers and the access to internet explain 97% of the R&D activities per capita in the Romanian macroeconomic regions after the adherence to the European Union, when controlling for the number of population in each region. This means that only 3% of the R&D employment variations are left unexplained. Even if this high goodness-of-fit shows that our model explains most of the R&D activity in Romania, we still have to treat with caution the practical significance of the exact percentage. Mainly, we can give a special attention to the fact that the number of individuals residing in each region in included in all the variables in our test. The size of the population in each region implicitly includes many other factors like the general regional economic conditions, climate, socio-economic development possibilities, ethnic situation, political stability etc.

The coefficients obtained are: β_0 = 0.1041217, β_1 = 1.426246, β_2 = 58.69103, β_3 = –0.002777.

We can rewrite our model equation as it follows:

$$y_{it} = 0.1 + 1.4 \cdot x_{1it} + 58.7 \cdot x_{2it}, \qquad (2)$$

where: y is the percentage of the regional population working as employees in the R&D sector, x_1 represents the number of students and teachers who fulfilled mobility stages divided by the region's population and x_2 is the FDI per capita.

From the three factors explaining the R&D activity, the FDI per capita has an important influence on the number of jobs in R&D sector (variable O). The results of our test show that 10,000 euros increase in FDI per capita will lead to an additional 0.59 % of the population working in the R&D activities, holding other factors fixed. The rate of the students and teacher engaged in mobility programs (variable L) has, as expected, a positive impact on the R&D employees: for the same level of FDI, double more students and teachers going abroad for short term professional stages will increase the R&D employment in the region by 1.4%. Using the internet with at least a weekly frequency has however a negative, but practically insignificant impact on the number of R&D jobs, given by the low coefficient of –.002777 (β_3).

This is related to our discussion from chapter 2.1, in which we explained that given the fact that internet seems to be used for other activities than information purposes, this variable might not have a significant economic impact on the R&D sector.

Finally, the intercept of 0.104 shows that there would be on average 0.104 % of the population working in the R&D sectors if the FDI would be zero and if there would not be any mobility at the university level.

Accounting for the economic crisis

The Hausmann test conducted showed random effects as a better choice than fixed effects, but we still assume that there are no time dependent effects. However, this assumption might not hold. The period we analysed included not only the beginning, but also the unfolding of the economic crisis. In a previous model, we analyzed the case when an unobserved variable like the economic conjuncture affects all the regions in a similar way, but differently in time [4]. On the grounds on this assumption, we conducted as well regression analysis with time fixed effects. This analysis took in consideration mainly the effects of the economic crisis during the studies period, but controlled as well for other time-dependent variations, like the economic cycle. The results are reported below (table 1), with the absorbing indicator set to "year" (variable B).

The results showed that, taking into account time dependent circumstances, the internet use failed to be a statistical significant variable (P-value for the variable "internet" is high). All other coefficients, except the constant term are statistically significant. The goodness-of-fit remains high (adjusted R-sq = 98%), with the variables included explaining most of the variance in the R&D employment. Otherwise, the coefficients of the foreign investments and mobility are only slightly different from the random effects model, which is a good support for our initial model. The interpretation of the coefficients show that, while leaving the economic conjecture to affect our data in an unobserved way, an increase by 1% in the FDI will create jobs in the R&D sector for an additional 0.57%

Table 1

TIME FIXED EFFECTS REGRESSION

Number of observations = 40
F(3, 32) = 529.14
Probability > F = 0.0000
R-squared = 0.9803
Adjusted R-squared = 0.9759

R&D employment	**Coefficients**	**Standard Error**	**t-value**	**P>|t|**	**[95% Conf. Interval]**	
internet	–.0019214	.0026672	–0.72	0.477	–.0073543	.0035116
mobility	1.539934	.4630871	3.33	0.002	.5966566	2.483212
FDI	57.16481	4.175887	13.69	0.000	48.6588	65.67081
constant	.0750489	.0799808	0.94	0.355	–.0878666	.2379645

Source: [4]

of the population of the region. Holding the FDI constant, a 10 % increase in the enrolment to mobility programs will have a positive impact on the R&D employment by 0.15%.

THE CASE OF THE TEXTILE INDUSTRY

The textile and clothing industry is of considerable size. However, in the face of intense global competition, the European textile and clothing companies are increasingly turning to research and innovation to ensure sustainable competitiveness and attractive working places. The European textile and clothing sector has always received substantial founding from the European research and innovation funding programmes such as the 7th Framework Programme, Competitiveness and Innovation Framework Programme (CIP) or the recent Horizon 2020. Collaboration and knowledge exchange is regarded as central for the development of this industry towards trends like more individual, adaptive and multi-functional clothing; sensor and actuator materials to increase the comfort and safety of the wearer; just-in-time production close to the customer in order to save time and lower the environmental impact of long transport distance [9].

Regarding the Romanian textile and clothing industry, including leather products, its situation is highly correlated with the European market. The other EU countries are the main trade partners for the Romanian textile industry, accounting for 92% of the imports and 84% of the exports (2008–2014 average). The trade balance is negative, on an unfavourable trend, reaching a negative balance of 560 mil. EUR in 2014, as the figure 1 shows.

From the point of view of resources flows with the external sector, statistical data shows that the Romanian textile industry, employing ca. 220,000 people in 2014 (INSSE), is among the most attractive ones for the FDI. In 2013, it attracted 17.8% of foreign direct investments directed to the Romanian industry, out of which 7.5% were directed to greenfield companies (BRD). However, these foreign investments do not support the development of the industry as we would expect. The share of R&D employment in the number of persons employed (%) and the total intra-mural R & D expenditure reported by the foreign controlled enterprises in the textile industry in Romania has been constantly 0 over the entire 2009–2013 period, showing that this industry remains mostly traditional in the production pattern [10]. The companies reported no R&D expenditure as part of the value added in the manufacture of textiles, wearing apparel, leather and related products either, meaning that the entire value added resides in the traditional production methods.

Fig. 1. Trade Balance for the Romanian Textile Industry

Therefore, the econometric model described in the chapters above cannot be applied in its statistics format to the Romanian textile sector for the time being. The R&D activity in this sector is too low to be suitable for an econometric analysis. However, this low level of R&D is not to be neglected, as it poses a limit to the general model. Our model shows the positive influence that knowledge transfer has on R&D activity, but the case of the textile industry also illustrates a vital condition for its viability: the foreign investments will not have a positive influence on the research development as long as they are directed only to low-skilled activities. Even though we cannot conduct a statistical analysis for this sector, economic data available can help us analyse the situation of the textile and clothing sector using descriptive methods and economic reasoning.

The unfavorable present situation of the R&D level in the Romanian textile industry is related to the fact that the Romanian textile and clothing production, striving to survive after losing the traditional markets in 1990s, developed mostly through unskilled or less qualified local labor force [11].

However, the increasing minimum wage and the general development of the expected working conditions in Romania represent a challenge to this type of business models. According to the statistics made by Economica.net in cooperation with ANOFM, the textile industry is among the top three employers in the country with free available positions [12]. Because of the low wages offered (usually the minimum wage for unqualified workers) and the demanding program, the industry has difficulties is covering its needs for human resources. In 2002 there were over 347.000 people working in textile fabrics in Romania. 12 years later, this industry employed only 137.000 people [13]. In the same time, the raising price of utilities, as well as unfavourable exchange rates has further hampered the advantage given by the business model based on low-cost labour force.

The textile industry in Romania is in a process of redesigning its competitive advantage. One possibility would be to follow the European strategy and concentrate on exclusive products, close to the customer, involving a high degree of innovation and technology, anticipating the market trends and incorporating our society's values of environment protection, social responsibility, human development and adaptive personal design. Another possibility would be to focus on unique products, hand-made, practicing the traditional production methods. This last model can though be vulnerable to price competition and would not be able to reach economies of scale, satisfy the needs for industrial or sportive clothing incorporating high technology or create attractive working places.

Anyhow, we have to consider the particularities of the Romanian textile industry. The Romanian companies in this sector of activity are traditionally acting more at local, regional or at best national level. These circumstances make it especially difficult to support R&D activities, to implement new technologies or to reach large scale reductions of costs. Therefore, expanding the possibilities of knowledge exchange through collaborative projects and involving the Romanian textile companies in R&D programs with public financing is vital for keeping the pace with changing customer expectations.

Actors on this market have already identified these opportunities of developing the national sector by joining partnerships for international R&D projects [14]. The lack of R&D activity of the private enterprises is partially compensated by public institutions, like the National Research & Development Institute for Textiles and Leather. This institute reported a high level of R&D projects reaching over 3,4 mil euros in 2012 and the same amount in 2013 in financing from international programmes like: Horizon 2020, ERA-NET, Eureka, Eurostars, Erasmus+, EEA Grants, JPI Cultural Heritage etc., programs in which the institute fulfilled the eligibility criteria and faced international competition for attracting financing. The cooperation between research centres and private enterprises in these projects ensures knowledge transfer and directs research towards the development and implementation of innovative solutions. In this way, the partners are combining their resources, generating solutions that are otherwise difficult to obtain for small enterprises. Such cooperation also increases the interest in continuing education, making the industrial sector to be more attractive for the labor force [15]. Nonetheless, more support from the public authorities is needed in order to assure the access of the SMEs to this type of financing and the implementation of performance management practices [16].

Taking in consideration these remarks, we see that the Romanian textile industry is an example of an economic sector in which the globalisation of the private sector was not accompanied by knowledge transfer. If the FDI and the mobility of the tertiary education persons where directly related to the level of R&D activity in the Romanian macro-economic regions, this cannot be verified for the private sector of the textile and clothing industry, as the foreign investments are directed only to low knowledge intensive activities. The public institutes, like the National Research & Development Institute for Textiles and Leather, which focused on knowledge intensive partnerships with international collaborators, experienced a high level of R&D and important research funds attracted from European programs. This brings us to a conclusion: the globalization level is positively correlated to the level of R&D, as long as it has a knowledge intensive component.

CONCLUDING REMARKS

It is often stated that opening the economy to the external sector has adverse effects, something that the specialists like to compare to immigration of the skilled labour force, instability of production, drain of resources, macroeconomic imbalances and others. In our opinion, these are all risks that need to be handled by firmly coordinated strategies and not avoided by retreating from the globalisation process. It is very hard and expensive to swim against the tide and try stopping the natural exchange of information and work force movement across the country borders, especially in the technological era. Instead, we can concentrate on the positive aspects of globalisation and take the most out of them. The flow of knowledge across countries is such a positive aspect, because it stimulates creativity and innovation in the confluence space where different perspectives and ideas meet each other. Students and teachers travelling abroad, foreign companies coming with new products and strategies on the domestic market and people accessing information from all around the world are the kind of factors that create diffusion of knowledge and transfer of competencies around the globe.

Our model showed a positive relationship between the level of globalization and the R&D activity. In selecting the estimators for the level of globalisation, we concentrated on foreign direct investments (FDI) for the business sector according to the IMF methodology [15], the number of students and teachers enrolled in exchange programs for the government sector and the rate of the population using internet for the private sector. The estimator for the level of R&D activity is the total number of employees activating in regional R&D activities. The model used for analysing the relationship between the variables is regression with panel data and time fixed effect, with R&D employees as the dependent variable. The results of both simple linear regression with robust estimation and time fixed effects suggest a significant positive relationship between the estimators used and the number of people employed in R&D sector.

Subsequently, we analysed the limits of this model by showing that this relationship is not valid in the cases in which the foreign investments were not accompanied by knowledge transfer. As an example of such a case, the textile industry in Romania, which benefited from FDI only for the use of low-cost labour force, had an almost not existing R&D activity in the private sector. In the same time, the public institutes from the same industry, which engaged in international partnerships involving knowledge transfer, were able to obtain important financing for research projects and had a significant R&D activity.

Our society can progress on the long term only by encouraging research on the limits and problems that we are facing and development of new solutions to our needs. As this article shows, research and development is positively correlated to higher openness of the economy. An active presence of foreign companies and mobility of personnel at the tertiary education level is likely to bring an infusion of knowledge from abroad and has a positive impact on R&D jobs creation. Opening to external influences in business and education sectors has therefore the potential of developing the country and creating high-quality jobs, without relying on the governmental R&D investments. However, if this openness is not guided towards knowledge transfer, its advantages are not obvious, as the example of the private textile industry shows.

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Biological synthesis of copper nanoparticles by using *Halomonas elongata* IBRC-M 10214

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REZUMAT – ABSTRACT

Sinteza biologică a nanoparticulelor de cupru prin utilizarea *Halomonas elongata* IBRC-M 10214

În ultimele decenii, proprietățile fizico-chimice unice și o gamă largă de aplicații ale nanoparticulelor de metal au atras atenţia multor domenii. Tehnica biosintezei și aplicarea microorganismelor au apărut ca o alternativă prietenoasă cu mediul, curată și cu beneficii la tehnicile fizico-chimice. În studiul de față, a fost investigată biosinteza nanoparticulelor de cupru (CuNPs), prin utilizarea supernatantului de culturi ale celulelor libere de Halomonas elongata IBRC-M 10214. Aceste nanoparticule de Cu au fost caracterizate prin spectroscopie UV-Vis, difracție de raze X (XRD) și spectroscopie în infraroşu cu transformată Fourier (FT-IR). S-a ilustrat faptul că dimensiunea medie a particulelor a fost de 56–73 nm, cu forme sferice. Factorii de influență cum ar fi: $CuSO_4$, glucoza și concentrațiile de NaCl au fost, de asemenea, studiate. În plus, activitatea antibacteriană a nanoparticulelor de cupru împotriva agenților patogeni rezistenți la multiple medicamente, cum ar fi Escherichia coli şi Staphylococcus aureus, a fost evaluată prin tehnica de difuzie în agar în calitate de culturi standard. Testul antibacterian al nanoparticulelor de Cu a prezentat o activitate antibacteriană față de tulpini bacteriene patogene de Escherichia coli cu 1 cm și Staphylococcus aureus cu 0,87 cm pentru zona maximă de inhibiţie. De asemenea, acoperirea cu nanoparticulele de Cu a probelor de textile a demonstrat că diametrele de inhibiţie zonale pentru E. coli și S. aureus au fost de 0,35 și respectiv 0,53. Pe baza acestui studiu, metoda "green" este ușoară și sigură pentru sinteza nanoparticulelor de Cu, cu o puritate relativă a nanoparticulelor obţinute. Cu toate acestea, rezultatele acestei cercetări ilustrează interacțiunea dintre concentrațiile de $CuSO_4$, glucoză și NaCl și efectele acestora asupra cantităţii sintezei nanoparticulelor de Cu.

Cuvinte-cheie: nanoparticule de cupru, Halomonas elongata, spectroscopie UV-Vis, XRD, FTIR, patogeni rezistenţi la multiple medicamente

Biological synthesis of copper nanoparticles by using *Halomonas elongata* IBRC-M 10214

In recent decades, unique physiochemical properties and a wide range of applications of metal nanoparticles have received particular attention in a wide range of fields. Biosynthesis technique and applying microorganisms, have emerged as an eco-friendly, clean and benefit alternative to physicochemical techniques. In the present study, the biosynthesis of copper nanoparticles (CuNPs) by using cell-free culture supernatant of Halomonas elongata IBRC-M 10214 was investigated. The CuNPs were characterized by UV-Vis spectroscopy, X-ray diffraction (XRD) and Fourier Transform Infra-red (FT-IR) spectroscopy. It was illustrated that the average particles size was 56–73 nm with spherical shapes. The influencing factors such as $CuSO_4$, glucose and NaCl concentrations were also studied. In addition, antibacterial activity of copper nanoparticles against multidrug resistant pathogens like Escherichia coli and Staphylococcus aureus was evaluated by the agar well diffusion technique as standard cultures. Antibacterial assay of Cu NPs showed antibacterial activity toward the pathogenic bacterial strains of Escherichia coli by 1 cm and Staphylococcus aureus by 0.87cm for maximum zone of inhibition. Also, Coating CuNPs onto textile samples showed inhibition zone diameters for E. coli and S. aureus were 0.35 and 0.53 respectively. Based on this study, green method is easy and safe for CuNPs synthesis with relative purity of the NPs obtained. However, the results of this research illustrate the concentrations interaction of $CuSO_4$, glucose and NaCl and their effects on amount of CuNPs synthesis.

Keywords: Copper nanoparticles, Halomonas elongata, UV-Vis spectroscopy, XRD, FTIR, multidrug resistant pathogens

INTRODUCTION

In order to specify the electrical, optical, magnetic and catalytic properties of metal nanoparticles, the synthesis and application of metal nanoparticles have obtained great attention comparing to bulk materials [1–5]. There are many physical and chemical methods for synthesis of inorganic nanoparticles that have toxic effects [6]. Moreover, biological synthetic strategies are cost-effective, eco-friendly and convenient to scale [7].

A wide range of biological sources such as bacteria, plant, fungi, yeasts, and algae can be applied for nanoparticles synthesis. Bacteria have obtained considerable importance for the synthesis of nanoparticles among all the biological systems [8]. Using of bacteria for nanoparticles synthesis has several advantages such as mild experimental conditions (temperature, pH, pressure, simple culturing, and extracellular production of nanoparticles), easy downstream processing as well as short generation time [9].

It is illustrated that when microorganisms are kept in contaminated metal environment, they evolve as mechanism to survive the condition by transforming toxic metal ions into their non-toxic forms such as metal sulfide/oxides [10]. *Halomonas elongata* can grow in high salt concentrations (more than 15% (wt/vol) NaCl) as extremely halophile bacteria [11]. Therefore, this property promotes us to evaluate metal nanoparticles synthesis by this bacterium. In addition, there are potential applications for these resulted metal forms. For instance, copper nanoparticles (CuNPs) can be utilized as antibacterial agent through growth hindering bacteria like *Escherichia coli* and *Staphylococcus aureus* [12,13]. On the basis of the pioneering studies, *Escherichia coli* ATCC 25922 and *Staphylococcus aureus* ATCC 45500 were used as multi drug resistant bacteria [14–16]. These bacteria are resistant to wide range of antibiotics including moxifloxacin (MFX), amoxicillin (AMX), cefixime (CFI), cefuroxime (CFX), imipenem (IMI), meropenem (MER), faropenem (FAR), and ertapenem (ERT) as well as clarithromycin (CLA) [17–18]. In this study, *Halomonas elongata* was used as biological source for synthesis of CuNPs; in addition, applied X-ray diffraction (XRD) and Fourier Transform Infra-red (FT-IR) spectroscopy were benefitted from for the characterization of Cu nanoparticles. Then, the antibacterial activity of these nanoparticles was evaluated.

EXPERIMENTAL WORK

Materials and Method

Taguchi methodology experimental design

All the combinatory experiments using the assigned parameter values were conducted with the aim of obtaining the final optimum conditions. The Qualitek-4 software was used to design and annualize Taguchi experiments [19]. Table 1 demonstrates considerable variable factors and their levels.

Halomonas Elongata Culturing Conditions

Copper resistant bacteria *Halomonas elongata* IBRC-M 10214 was obtained from bacterial archive of Razi University of Kermanshah and was cultivated in the basal medium contains (g/L) Glucose, 10; $MgSO_4$, 1.4; NaCl, 150; NH_4Cl, 2.3; K_2HPO_4, 0.6; $FeSO_4$, 0.001 and 10% (V/V) inoculum was transferred into 100 mL Erlenmeyer flasks and incubated in a shaker at 37 °C for one week. The growth medium was supplemented with various nutrients compositions by varying glucose and NaCl according to the details following the experiment design (table 2). Afterwards, 1.5 cc from each Erlenmeyer flask was centrifuged at 10,000 rpm for 5 minutes. According to the details following the experiment design, 1 cc of supernatant was poured test tube followed by treating with different concentrations of copper sulfate ($CuSO_4$) (table 2–5). Then, resulted solution was placed at 28 °C in a shaking incubator with set at 120 rpm [11].

CuNP Characterization

The formation of CuNPs was observed preliminary by visual color change. UV-Vis spectrophotometer, XRD, FT-IR were used for further characterization of CuNPs. Cu^{2+} reduction in the prepared mixtures was surveyed by UV-Vis absorption spectra analysis from 400 to 800 nm by UV-Vis spectrophotometer (Tomas, UV 331). Crystallographic information of the samples was gained from XRD. XRD patterns were evaluated through a (EQUNIOX 3000) operating at 30 kV and a

Table 1

Trail	$CuSO_4$ (mmol)	Glucose (mmol)	NaCl (mmol)	OD
1	1.4	0.28	8.3	0.519
2	2.8	0.28	9.7	0.673
3	5.6	0.28	11.1	0.868
4	2.8	0.39	8.3	0.904
5	5.6	0.39	9.7	0.935
6	1.4	0.39	11.1	0.620
7	5.6	0.56	8.3	0.912
8	1.4	0.56	9.7	0.849
9	2.8	0.56	11.1	0.694

Table 2

Factors	Level 1	Level 2	Level 3
Glucose	0.818	0.819	0.686
NaCl	0.727	0.819	0.778
$CuSO_4$	0.904	0.756	0.662

Table 3

Factors	Effects of factor pairs based on (SI)	Columns	Intensity of interactions (%)	Columns	Optimum conditions
1	NaCl × $CuSO_4$	2 × 3	67.42	1	[2,3]
2	Glucose × $CuSO_4$	1 × 3	15.62	2	[2,3]
3	Glucose × NaCL	1 × 2	14.78	3	[2,2]

Table 4

Factors	DOF (f)	Sum of Sqrs.	Variance	F-Ratio (F)	Pure Sum (S´)	Percent (%)
Glucose	2	0.035	0.017	0.788	0	0
NaCl	2	0.012	0.006	284/0	0	0
$CuSO_4$	2	0.089	0.044	014/2	045/0	815/24

Table 5

Factors	Level	Contribution
Glucose	2	0.044
NaCl	2	0.044
$CuSO_4$	3	0.13
Total contribution from all factors	-	0.217
Current grand average of performance	-	0.774
Expected result at optimum condition	-	0.992

current of 20 mA with Cu Kα radiation (λ = 1.54059 Å). Also, the 2ɵ scanning was of 30–80° at 2° min^{-1}. FT-IR measurements were done by (Germany, Bruker, Model: ALPHA) spectrophotometer. Nanoparticles morphology and size of dried CuNPs suspension were carried by SEM (Model XL30, Philips, Eindhoven) [20].

Antibacterial Effects

Escherichia coli ATCC 25922 and *Staphylococcus aureus* ATCC 45500 as multi-drug pathogens were applied for evaluation of the effect of antibacterial properties of CuNPs by modified ager well diffusion. Clinical pathogen bacteria were cultivated in Muller Hinton agar (MHA) plates; 5 mm diameter disks were prepared with the help of a sterilized steel cork borer. Afterwards, different concentrations of CuNPs (1.4, 2.8 and 5.6 mmol) were loaded in different disks, and after placing the disks on agar the plates were incubated at 37 °C for 48 hr [21].

Coating CuNPs onto textile samples

First, the samples had been washed in a washing machine followed by textile fabrics washing in a water bath at 60°C for 3 hours. Afterwards the samples were washed with cold deionized water and dried in and oven at 60°C overnight. Due to testing of antibacterial effect, 1.5 cm × 1.5 cm squares of clean samples were cut. The antibacterial activity of textile samples coated with CuNPs solution was investigated against *E.coli* as facultative gram negative and *S.aureus* as gram positive. After sterilization of nutrient broth agar solid cultures in an autoclave for 20 min at 120 °C, the cultures were prepared. The disk diffusion method was applied to assessment of antibacterial activity of the CuNPs [22].

RESULTS AND DISCUSSION

UV-Vis analysis of CuNP biosynthesis

When cell free supernatant of *H. elongata* was added to $CuSO_4$ solution and incubated for 48 h, the mixture's colour reaction altered from blue to light green (figure 1). This colour change illustrates the formation and oxidation of CuNPs. Shantkriti and Rani (2014) reported similar appearance of green colour solution with addition of $CuSO_4$ to a flask containing *Pseudomonas fluorescens* [7].

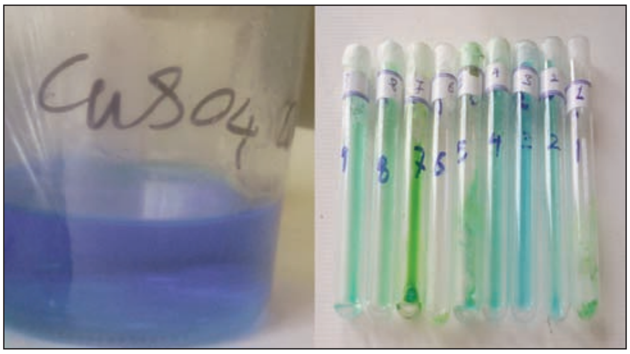

Fig. 1. Changes of reaction mixture's colour from blue (left) to light green (right) after supernatant addition

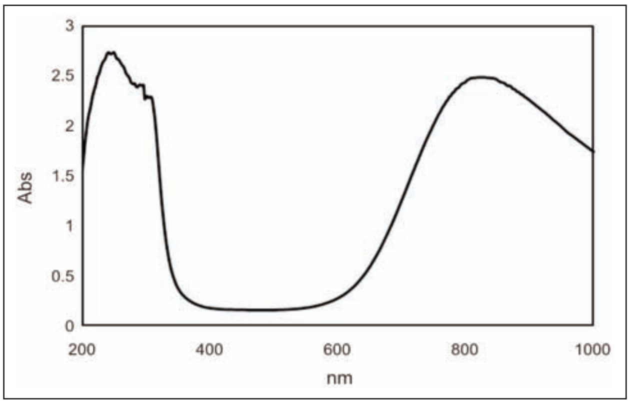

Fig. 2. UV-Vis absorption spectra of CuNPs produced by culture supernatant of *H. elongata*

Spectrum absorption of UV-Vis for this solution illustrated a distinct absorption peak in the region of 700–800 nm (figure 2). Based on special particles properties such as size, shape and capping agents, the precise position of SPR band may shift [23]. Also, copper NPs synthesized from Aspergillus species confirmed the presence of copper nanoparticles at 300 nm [24].

FT-IR analysis

FT-IR was performed in order to evaluate the molecular interactions between the CuNPs and the media. Figure 3 demonstrates a C=O vibration band at 1631 cm^{-1}. In addition, the spectrum illustrates at 3447 cm^{-1} to O-H stretching, 2109 cm^{-1} to C=C stretching, 1048 cm^{-1} to C-O stretching, 989 cm^{-1} to =C-H bending, 773 cm^{-1} to C-Cl stretching, 628 cm^{-1} to C-H bending, 561 cm^{-1} to C-Cl stretching and 416 cm^{-1} to C-Br stretching. The interaction between the Cu-NPs and media is the cause of the corresponding vibration bond, which demonstrates a reaction between Cu-NP surface and carbonyl and hydroxyl functional groups [25]. Similar results were reported by Roca et al. [26].

X-ray diffractometer

A high crystalline level of the CuNPs sample can be seen with diffraction angles of 45.19, 74.87 and

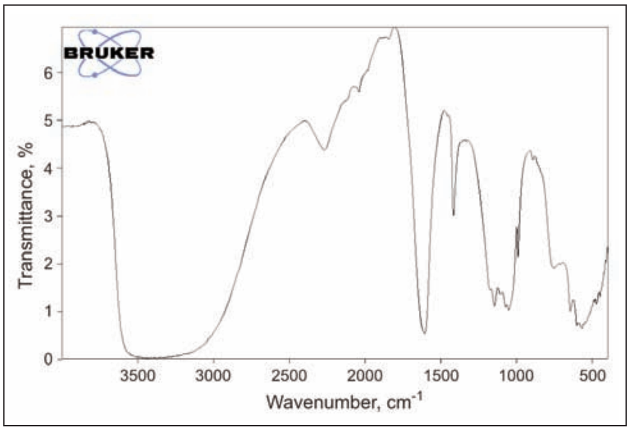

Fig. 3. FT-IR spectra of Cu-NPs synthesized by culture supernatant of *H. elongata*

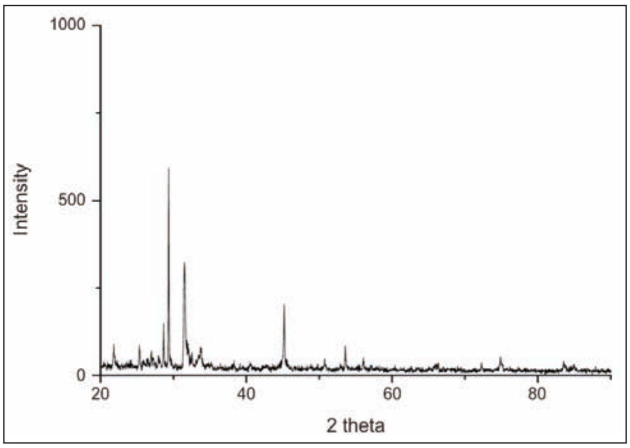

Fig. 4. X-ray diffraction pattern of CuNPs synthesized by *H. elongata* after 48 h

50.56, which corresponds to the characteristic face centered cubic (fcc) of copper lines indexed at (111), (200) and (220), respectively [23]. Impurities such as CuO and Cu_2O may be effective on absence of any noticeable peaks in pattern. The sizes of nanoparticles gained were estimated to be in the range of 56–73 nm using Debye-Scherer eq. (1):

$$d = \frac{k\lambda}{\beta \cos \theta} \tag{1}$$

where k, known as Scherer's constant, ranges from 0.9 to 1.0, λ is 1.5418 Å, which is the wavelength of the X-Ray radiation source, $\beta1/2$ is the width of the XRD peak at half height and θ is the Bragg angle (figure 4). However, XRD measurement revealed that the nanoparticles initially formed as colloids tend to grow and react with environment oxygen [27].

To obtain more morphology information, electron microscopy scanning was performed on the dried samples. The analysis of SEM demonstrated that Cu nanoparticles were formed as aggregates and had a variable morphology (figure 5). Therefore, SEM illustrated spherical shape of CuNPs. In similar way, Ashajyothi and co-workers reported size ranging from 20 to 90 nm with spherical shape [28]. CuO NPs prepared with *Pseudomonas Fluorescens* showed average particle size in 49 nm with spherical and hexagonal shapes [7].

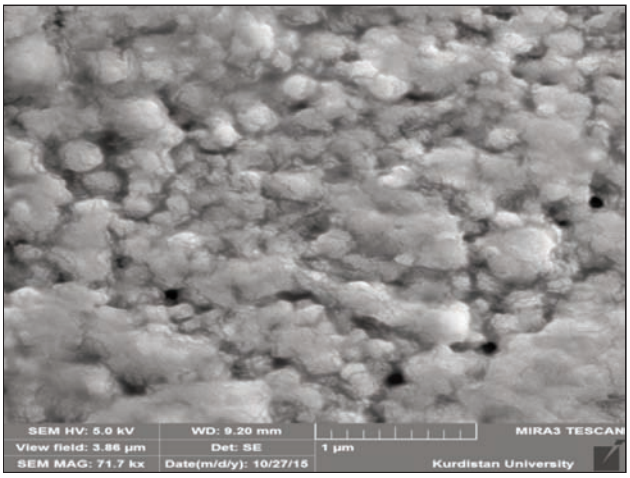

Fig. 5. SEM image of CuNPs

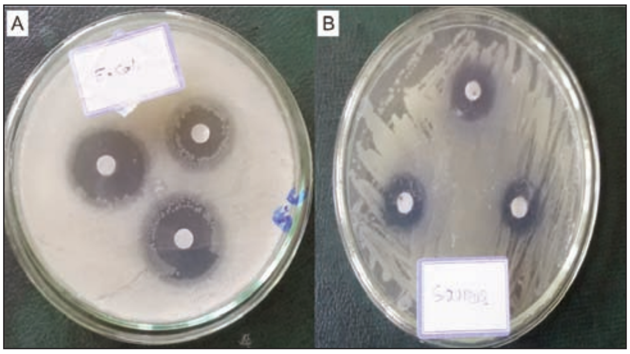

Fig. 6. Antibacterial in vitro effect of CuNPs on *E. coli* (A) *and S. aureus* (B) at trial number seven

Antibacterial Effects

Figure 6 illustrates the presence of antibacterial effect of the nanoparticles on the pathogen bacteria. Antibacterial assay of Cu NPs demonstrated antibacterial activity toward the pathogenic bacterial strains of *Escherichia coli* by 1 cm and *Staphylococcus aureus* by 0.87 cm for maximum zone of inhibition. These amounts of inhibition were for trial number seven with concentrations of 5.6 mmol, 8.3 mmol and 0.56 mmol for $CuSO_4$, NaCl and glucose respectively. There is a similar result regarding antibacterial properties of copper NPs on *S. aureus* [29]. Antimicrobial effects of silver and copper NPs against *E. coli* (four strains), *B. subtilis*, and *B. aureus* (three strains) have been reported by Ruparelia and coworkers [30]. Also, compared with antifungal activities, antibacterial activities were higher [31].

The better inhibitory effect observed in *E. coli* than in *S. aureus* is related to difference in the outer structure of these bacteria [32]. Gram positive bacteria such as *S. aureus* than to gram negative bacteria such as *E. coli* have a thick layer of peptidoglycan that prevents penetration of the Cu ions [33]. Bactericidal may be resulted from the attaching of copper ions (released by the nanoparticles) to the negatively charged bacterial cell [34].

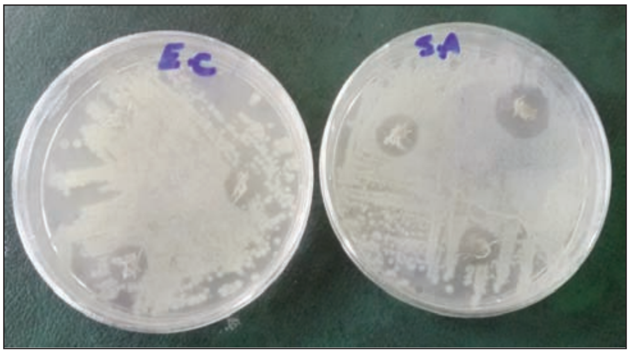

Fig. 7. Antibacterial effects of CuNPs@fiber against *E. coli* and *S. aureus*

Antibacterial activity of Cu NPs is not merely resulted from their release of metal ions but also can be related to their morphology, specifically their small size and higher surface area to volume ratio [7]. Similarly, antibacterial activity of Ag NPs has been reported on the same bacterial strains by Guzman et al [32].

Coating CuNPs onto textile samples

Figure 7 shows antibacterial activity of CuNPs@fiber on *E. coli* ATCC 25922 and *S. aureus* ATCC 43300 [34]. Antibacterial activity of fabric samples was evaluated in terms of inhibition zone formed on nutrient broth agar. The control samples, did not illustrated any antibacterial activity [35]. The Cu nanoparticles@fiber removed all the bacteria under and around them. Inhibition zone diameters for *E. coli* and *S. aureus* were 0.35 and 0.53 respectively. These diameters of IZ were for trial number seven with concentrations of 5.6 mmol, 8.3 mmol and 0.56 mmol for $CuSO_4$, NaCl and glucose respectively.

CONCLUSION

CuNPs with spherical shapes and average mean sizes in the range of 56–73 nm and an fcc crystal structure were synthesized in a green method by *Halomonas elongata*. UV-Vis, XRD, FT-IR were used to characterization of NPs. Based on this study, green method is easy and safe for CuNPs synthesis with relative purity of the NPs obtained. However, the results of this research demonstrate the concentrations interaction of $CuSO_4$, glucose and NaCl and their effects on amount of CuNPs biosynthesis. Also, a safe assessment of these NPs as *in vitro* and *in vivo* is required in future studies.

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