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RECONSIDERING HEMP AS AN INDUSTRIAL PLANT CULTIVATED IN ROMÂNIA

BY

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Abstract. Given the highly valuable properties of hemp fiber, at the present time in the world this plant has found use in many areas. Before 1990 the area planted with hemp in Romania had an important share of total technical crops. But hemp culture at present almost no longer exists. To this situation has also contributed the fact that Cannabis Sativa is confused with Cannabis Indica that contains a higher percentage of hallucinogenic substances. The paper presents some important mechanical properties of hemp yarns and recommendations for the returning of industrial hemp plant to the attention of growers and processors.

Key words: hemp, fibbers, yarn, properties, technical articles.

1. Introduction

Before 1989, Romania was among European countries with the largest areas planted with hemp and the third country in the world among the manufacturers of hemp (Ionescu-Muscel, 1990)

At the Lovrin Research Station were invested money, time and human labour to create performant varieties of industrial hemp. Here there was

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obtained the most competitive variety of hemp: Lovrin 110, with a production of 10,000 kilograms per hectare and a quantity of 1,100 kilograms of seed per hectare. Besides this variety was created a new one, Lovrin 111, which was resistant to the specific plant disease, drought and hail, (http://www.ziare.com/)

Data published by the Ministry of Agriculture and Rural Development (MARD) mentions, for 2010, an area of 23 hectares planted with hemp. The number of processing units has decreased considerably from 28 processing units existing in 1991 to two operating units.

According to European legislation, in Romania it can be grown only those hemp varieties that have tetrahydrocannabinol (THC) content which do not exceeding 0.2%. Eligible hemp varieties are Denise, Diana, Lovrin 110, Silvana and Zenit.

The products obtained from Cannabis sativa, regardless hemp species of which they were processed, were placed under the control of national law, by law no. 143/2000, and to the United Nations Conventions of 1961 and 1971. The reason is the presence of tetrahydrocannabinol in the chemical composition of hemp plant. By the Convention on Narcotic Drugs signed at New York in 1961, to which România joined, the international community has expressed strong determination to fight against all illicit operations related to cannabis plant, regardless the concentration of THC.

According to law no. 143/2000 and its implementing Regulation, on combating illicit drug trafficking and consumption, all cultures of plants containing narcotic or psychotropic substances should be authorized by the Ministry of Public Health, the Public Health Department.

In order to avoid all situations arising from ignorance of the law and in order to delimit the good faith grower from the one who deliberately circumvent legislation, population has a simple and legal instrument, that to obtain authorization for cultivation before the beginning of the sowing works (http://www.agromania.ro/). Hemp is one of the oldest plants cultivated in our country (over 2000 years), being used mainly for production of fibres used in clothing and technical articles. Hemp stems from local populations and wild hemp contain 10-12% fibres and the improved varieties contain 26-32%.

Fibres content is influenced by variety, climatic conditions and technology. Hemp fibres have a number of invaluable features such as resistance (tensile, torsion, friction, decay), extensibility (elastic and plastic), spinning capacity, length greater than that of sisal fibres, jute, manila or cotton, being used in the textile industry, manufacturing industry or the automotive industry. Hemp seed can be used to extract oil used directly in food and the manufacture of margarine. Raw oil is used to obtain varnish, paint, linoleum, soap and wax canvas. The seeds are widely used, directly or as concentrated feed, for poultry feed. Residues cakes left over from oil extraction are used alone or in feed concentrate for animals feed.

About 55% from the weight of the stem represent the woody part which contains over 50% cellulose. The woody wastes resulting from fibers extraction are used to obtain paper, noise insulating laminated boards, for the furniture industry, cellulosic man-made fibres.

Chaff resulting after threshing the hemp planted for seeds is a particularly valuable fertilizer: 10 tons of hemp chaff equals 40 tons of manure. Leaves and flowers are used in medicine.

Narcotic action is given by substances produced by the secretive fluff of the leaves from inflorescence, by the flowers coating and by the bracts that covers the seeds. The content of narcotic and hallucinogenic substances differs greatly from species to species. The highest content was found in Indian hemp – Cannabis indica with its two forms: sub narcotic and narcotic. This species can be found cultivated and uncultivated in: India, Iran, Turkey, Syria, and North Africa, Near and Middle East. The height of the plant varies from 1 to 1.5 m; it is strongly branched, with leaves with narrow leaflets and with large seeds.

Hemp for fibres and especially Cannabis Sativa L., which is also the variety cultivated in Romania and in general, in Europe, has a low content of hallucinogenic drugs (in most cases 0.2 to 0.3%). This hemp is cultivated and used for the production of fibres. It is characterized by plants with high waist, from 2.0 to 5.0 m, unbranched, leaves with large and long leaflets, and semi compact short inflorescences (especially female plants). This type of hemp is found in Bulgaria, Italy, Spain, Hungary, France and România.

Hemp plant, especially the Indian one, has the biosynthetic ability to produce from the acid canabigerolic – the cannabidiol acids (DCBD and CBD), tetrahydrocannabinol acid (ATHC) canabinol (CNB) and canabinolic acid (ACNB). ACBD and ATHC acid predominates in plants until flowering, then turns to the CBD, CNB and THC (tetrahydrocannabinol). This transformation occurs during the flowering season and the plant needs a daily average temperature over 32ºC, which can be done only in warm climates of the world and less in Europe and România.

Towards the end of vegetation, by drying, some of the THC is transformed into cannabinol (CNB), which is pharmacologically inactive. An important fact is that on the territory of România hemp was cultivated only for fibres and it was not used for drug even accidentally. Industrial hemp is confounding with the drug simply because of its name cannabis.

The European Union offers advantages for the production of textile plants (flax and hemp) and the government also approved a development program of textile crops (subsidies for seeds and production).

Crops sector and his textile plants (hemp, flax and cotton) represent a particular importance not only for the agricultural economy but also for the sustainable ecological agriculture. The hemp grown in România is specific for fibre production, arguments being age of cultures and the value of hemp varieties obtained at S.C.D.A. Lovrin and S.C.A. Secuieni (http://www.revista-ferma.ro/).

World surface planted with industrial hemp is more than 500,000 ha, until 1990, important cultivators countries were: Russia, Yugoslavia and România. Flax and hemp are considered priorities in The Ministry of Agriculture and Rural Development strategy due to the amounts awarded to support the two cultures for the expansion of the cultivated areas with this textile plants. In Figs. 1,…,3 it can be seen the evolution of hemp production during the interval from 2001 to 2008.

The number of processing units has decreased considerably (from 28 existing processing units in 1991) in 2010 remained in operation: S.C. Galir, Mangalia - Constanța County- with the processing fibres capacity of 640 tons /year; S.C. Carpic Carei - Satu Mare County - with the processing fibres capacity of 2000 tons/year.

Through the SAPARD program it was built and put into use a hemp seed processing unit in order to obtain oil and nutritional supplements, the flour protein meal obtained after pressing, and the shelled seed - used for food purposes. CANAH International is the first hemp oil producer in România, whose factory has a processing capacity of 1000 tons of seeds per year, the equivalent of 250,000 litres per year hemp oil and it is placed in Salonta, Bihor County.

The very favourable areas are characterized by a rainfall during the vegetation period of 300-550 mm and the average temperatures do not exceed 16 to 18ºC. These climatic conditions can be found on the following areas:

− The west country (Criş plains, Mureş, Timiş, Caraş);

− The valleys of Târnave, Mureş, Someş Valley, Olt, in Bârsa depression, Sfântu Gheorghe depression, in the valley of Siret and in the valley of Moldova.

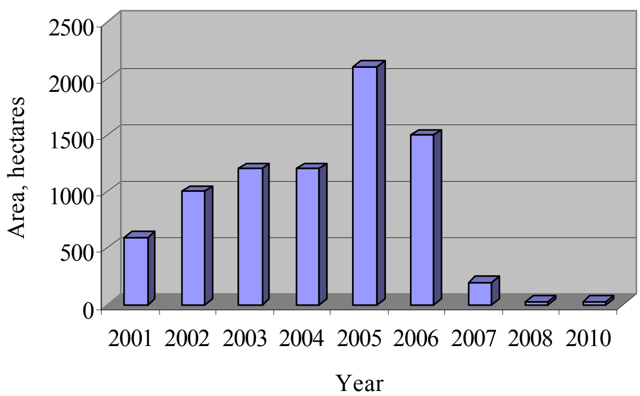

Fig. 1 − Areas cultivated with hemp during the 2001-2010 interval.

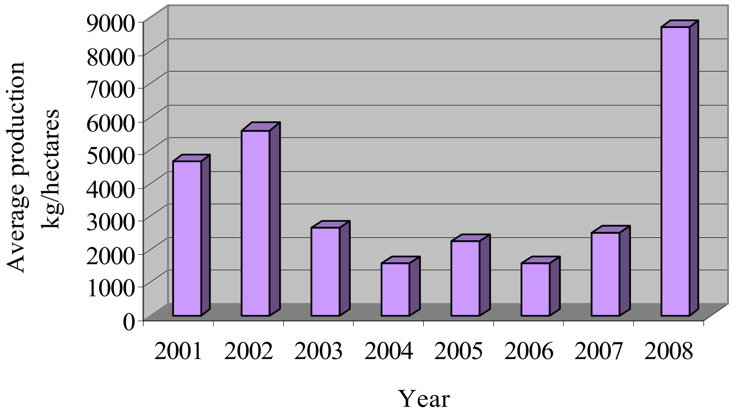

Fig. 2 − Hemp average production of stems during 2001-2008.

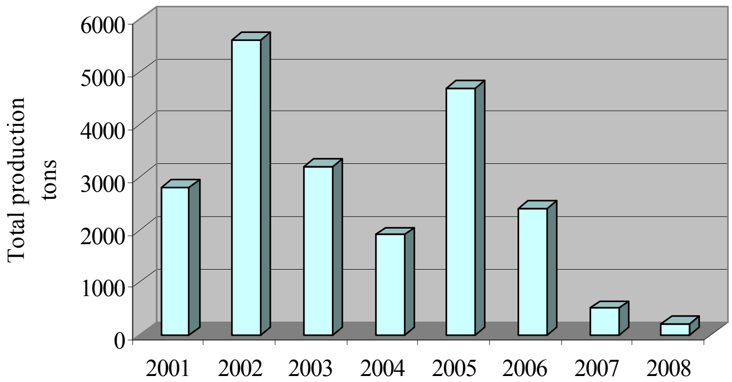

Fig. 3 − Hemp total production during 2001-2008.

Strategic elements in the textile plants domain considered by The Ministry of Agriculture and Rural Development (http://www.madr.ro/) are:

− orientation of the crops farmers in traditional areas to cultivation of hemp and flax;

− encouraging the association with the aim of reaching the rural development measures for new processing units;

− maintaining the financial support for industrial hemp and flax cultivation;

− support the textile plants for other uses: seed oil, bio-fuel, paper, etc.

To underline the mechanical qualities of the hemp yarns, the experiments present a comparison between hemp and glass yarns.

2. Materials and Methods

The hemp yarns which were investigated have 100 tex and 93.5 tex and there were spun from boiled roving and from bleached roving respectively. There were compared with glass yarn. The water absorption in hemp fibres modifies their mechanical properties and it is important to know the transfer mode of the fibres properties in the yarn characteristics.

For the serial testing in wet stage, the yarns were stressed after one minute moisten and their drying between two sheets of absorbent paper. Measuring of the yarns breaking strength was made according to ISO 2062 on TINIUS OLSEN H5K-T yarn tester (England) by automatic registering of the load–displacement curve. The initial length of the tested sample was 500 mm.

3. Results and Discussions

The hemp yarns have a good behaviour in wet stage. The breaking tenacity increases with 38% in case of yarn with the linear density of 100 tex, spun from boiled hemp roving, see Table 1. In the case of the bleached hemp yarn with the linear density of 93.5 tex, the breaking tenacity increases over 15% in wet stage, in comparison with the breaking tenacity of the same yarn testing in dry stage.

During the breaking process, irreversible displacements are taking place between fibres as well as between the macromolecular chains of the fibres compounds, the yarn supporting a higher load increase than the displacement increase (Figs. 4,…,6).

Table 1
Tenacity of Hemp Yarn Spun from Boiled Roving and Hemp Yarn Spun from Bleached Roving, in Dry and Wet Stage

Characteristics	Hemp yarn with the linear density of 100 tex, spun from boiled roving:		Hemp yarn with the linear density of 93.5 tex, spun from bleached roving:	
	tested in dry stage	tested in wet stage	tested in dry stage	tested in wet stage
Breaking strength, [cN]	642	915	1100	1881
Breaking tenacity, [cN/tex]	7.5	10.4	11.7	13.5
Change of tenacity in wet stage in comparison with dry stage, [%]	–	+38.6	–	+15.4
Degree of water permeation, [%]	–	50.6	–	76

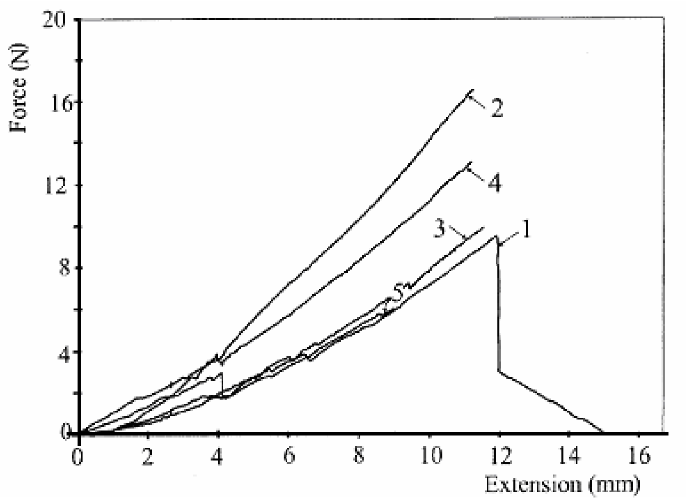

Fig. 4 − Force-extension curves of the hemp conditioned yarn 93.5 tex produced by wet spinning from bleached roving.

The spiral structure of cellulose macromolecular into the hemp fibres, changes its orientation by water absorption and increase of intermolecular forces causing the increase of the strength yarns tested in wet stage, see Fig. 5.

In the case of the studied hemp yarns all the fibres of the cross section are fixed together and take part with their tenacity into the yarn strength. This yarns deformation arises during the moment of application of the traction force. The elasticity range is missing, see Figs. 4,…,6.

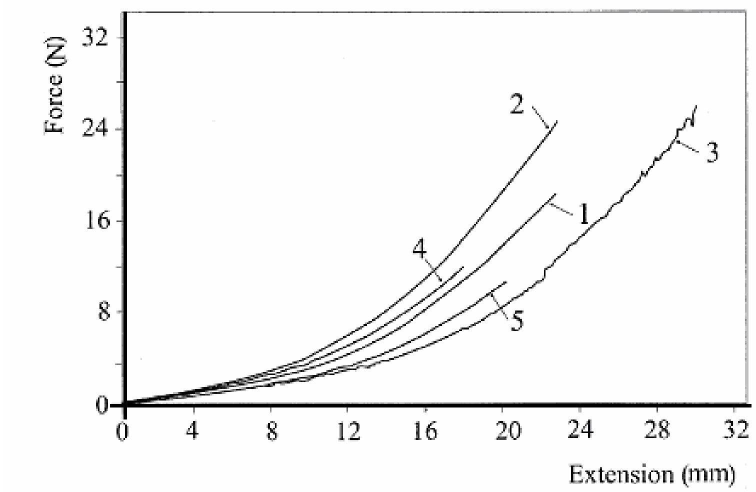

Fig. 5 − Force-extension curves of the hemp yarn 93.5 tex produced by wet spinning from bleached roving, tested in wet stage.

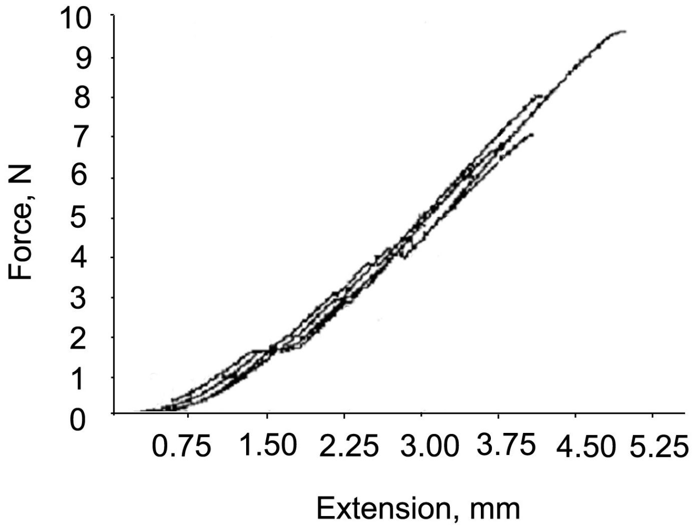

Fig. 6 − Force-extension curves of the glass yarn 66.6 tex.

The elongation of the glass yarn is 1% and for the hemp yarns is 1.5-3%. The increases of the breaking tenacity in wet stage and the reduced elongation are the advantages that the hemp yarns bring to technical articles.

4. Conclusions

Worldwide on can notice an increased tendency to use hemp fibres in the technical textiles field as a consequence to the fact that these fibres show: high resistance at tensile stress, friction, reduced breaking elongation, high spinning capacity and high length of technical fibres.

One strategic element in the textile plant domain considered by The Ministry of Agriculture and Rural Development is encouraging the association with the aim of reaching the rural development measures for new processing units.

In conclusion, the variety Cannabis Sativa L., which was cultivated in Romania, having a low content of hallucinogenic drugs (in most cases 0.2 to 0.3%) is recommended for planting. This hemp is grown and used for the production of fibres and seeds. It is characterized by plants with high waist, from 2.0 to 5.0 m, unbranched in comparison with Cannabis Indica which is short and branched.

Eligible hemp varieties are Denise, Diana, Lovrin 110, Silvana and Zenit.

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RECONSIDERAREA CÂNEPEI CA PLANTĂ INDUSTRIALĂ CULTIVATĂ ÎN ROMÂNIA

(Rezumat)

Actualmente, pe plan mondial, fibrele de cânepă sunt utilizate în multe domenii, datorită proprietăţilor deosebit de valoroase. Înainte de 1990 suprafeţele cultivate cu cânepă în România, au avut o pondere importantă în totalul culturilor de plante tehnice. În prezent, cultura cânepei, aproape nu mai există. La această situaţie a contribuit, în parte şi confuzia care se face între Cannabis Sativa şi Cannabis Indica, varietate care conţine un procent mai mare de substanţe halucinogene. Lucrarea urmăreşte evidenţierea unor proprietăţi importante ale fibrelor de cânepă. Totodată sunt precizate o serie de recomandări pentru reîntoarcerea acestei plante industriale – cânepa pentru fibre – în atenţia producătorilor şi a prelucrătorilor.

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MODELLING THE INFLUENCE OF COTTON FIBRE PROPERTIES ON YARN ELONGATION USING MONTE CARLO TECHNIQUES

BY

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Abstract. The study on cotton yarn properties has been one of the classical issues in the textiles' fundamental theories, since such research could promote the understanding of not only yarn properties but also provide valuable reference to the engineering of yarn spinning. The adaptation of the statistical approaches have necessitated many research works to discuss the effect of fibre properties, processing parameters as well as yarn structure on the strength of ring spun yarn. However the process of establishing the influence of fibre properties on yarn properties still remains a challenge due to the variability of cotton fibres. Therefore the approach of applying statistical modelling like Monte Carlo simulation in the study of yarn parameters brings a new dimension in yarn modelling. In this paper a statistical model for predicting the yarn elongation was built by using data of fibre and yarn parameters from Kenyan cotton. A Statistical model was developed using the Linear multiple regression method and then Monte Carlo simulation was performed in predicting yarn elongation. The model was used to study the influence of fibre properties on yarn elongation properties.

Key words: cotton yarn, cotton fibre, ring spinning, regression analysis, yarn elongation, Monte Carlo.

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This paper is the extended version of the paper published in abstract at the Technical Textiles – Present and Future Symposium

1. Introduction

The physical characteristics of a fibre determine its processing behavior, production efficiency and finally yarn and fabric quality. Therefore, predicting the quality characteristics of yarns, such as the tensile properties from the raw material properties, has been an essential study, since the inception of the modern textile industry. In addition to raw material, processing conditions, preparation stages, machine parameters, spinning methods also have considerable effects on the yarn properties. The application of statistical approaches in establishing a relationship between fibre and yarn quality characteristics has been the most popular method in yarn modelling. One of the most common statistical approaches is the multiple regression method. This approach has been used to investigate the interdependence of the different fibre properties and to estimate the relative contribution of each fibre property to the overall yarn properties (El Mogahzy *et al*., 1990; Ethridge *et al*., 1996; Hunter, 1988; Majumdar *et al*., 2004; Üreyen *et al*., 2006).

The tensile properties of a spun yarn have always been very important in determining the quality of the yarn, since they directly affect the winding and knitting efficiency as well as warp and weft breakages during weaving. It is, therefore, important to establish which fibre and yarn parameters influence the yarn tensile properties and derive functional relationship between them. So far, numerous mathematical and empirical models have been established for the estimation of single yarn tenacity (Hunter, 1988; Üreyen *et al*., 2006; Sasser *et al*., 1991; Subramanian *et al*., 1973; Subramanian, 2004) and count strength product (CSP) (El Mogahzy *et al*., 1990; Hunter, 1988; Hearle *et al*., 1969; Aggarwal, 1989) using fibre properties and some yarn parameters.

In this study, statistical models were developed to estimate ring yarn elongation properties by using multiple linear regressions and Monte Carlo Simulation. In addition to fibre properties, yarn linear density and yarn twist were also used as inputs.

2. Materials and Methods

In this work, a total of 54 different cotton samples were collected in fibre form from a spinning mill in Kenya. The spinning operations can affect the fibre properties in different ways, depending on the machinery line and adjustments. For the elimination of these effects, fibre properties were measured from bales delivered to the blowroom section by using the comb sorter (length, fineness, short fibre content), trash analyser (trash), micronaire tester (micronaire value) and stellometer (strength and elongation). Table 1 shows the fibre properties measured.

All samples were spun into yarns on ring spinning machine at a yarn count of Nm 14 (71.43 tex), Nm 18 (55.56 tex) and Nm 43 (23.26 tex). Each yarn count was spun at three different twist multipliers. A total of 108 spinning trials were done.

Table 1
Cotton Fibre Parameters

Variable	Units	Mean	Max	Min	Median
Fibre length (ln)	[Mm]	28.51	32.60	23.60	28.80
Short fibre content (sf)	[%]	7.37	11.75	3.50	7.25
Fibre Fineness (fi)	[dtex]	1.24	1.85	0.63	1.20
Fibre Elongation (fe)	[%]	5.35	15.00	1.20	5.50
Fibre Strength (st)	[cN/tex]	31.27	33.80	27.50	31.20
Micronaire Value (mi)	–	2.74	3.30	2.45	2.67
Trash Content (tc)	[%]	7.99	19.85	2.70	7.55

The appropriate drafting ratios were adjusted on the ring spinning machine for each sample. Other spinning conditions were kept constant. Orbit rings (42 mm diameter) and travellers (suitable weights were selected for each yarn count) were used. For each yarn sample ten cops were produced and tested. The yarn elongation was evaluated on the Universal Strength tester, twist tester (twist) and Count Tester (linear density). The measurements of the main properties are shown in Table 2.

Table 2
Cotton Yarn Parameters

Variable	Units	Mean	Max	Min	Median
Yarn Elongation (ye)	[%]	28.04	39.39	6.95	30.44
Yarn Twist (tw)	[tpi]	14.97	24.49	9.07	13.02
Yarn Linear density (tx)	[tex]	49.92	80.81	19.73	55.61

The multiple regression method has the advantage of simplicity in describing the quantitative relationship between textile material properties. Therefore this method was selected for establishing the relationships between fibre and yarn properties. At the beginning the types of relationship between selected properties (independent variables) and yarn elongation (dependent variable) were checked individually. The statistical tests indicated that there was a nearly linear relationship between fibre properties and yarn elongation. Therefore a linear multiple regression analysis method was chosen for the study in order to establish a quantitative relationship of yarn properties with respect to fibre properties, yarn count, and yarn twist. Statistical analyses were performed using the SPSS 19 and Minitab 15 programs.

Monte Carlo Simulation involved the process of using the random numbers generated from the distributions of the input variables in obtaining a probabilistic approximation to the solution of the regression model for the output variable. This process was performed using the features of ModelRisk 3 software. The Monte Carlo simulation was further used for sensitivity analysis of the model which was developed in order to evaluate the relative contribution of each predictor to the overall prediction of the yarn strength and thus the input parameters were ranked according to their coefficients in the model. The prediction of the yarn elongation was performed using a histogram plot while the sensitivity analysis was evaluated on spider and tornado plots (David, 2009).

3. Results and Discussions

The linear multiple regression model (Eq. 1) for predicting yarn elongation was developed using fibre parameters with yarn twist and yarn linear density as predictors. The model had a coefficient of regression (R) value of 0.840.

$$ye = 7.250 - 0.246tp + 0.005tx + 0.056sf + 0.001ln - 0.199fi + + 0.077fe + 0.033st + 0.172mi - 0.019tc \quad (1)$$

3.1. Prediction Yarn Elongation Model

The prediction of yarn elongation from the model (Eq. 1) was evaluated using Monte Carlo simulation and the histogram chart plotted as shown in Fig. 1. According to the histogram plot, the predicted value of the yarn strength at 80% probability is between 5.57% and 5.99%.

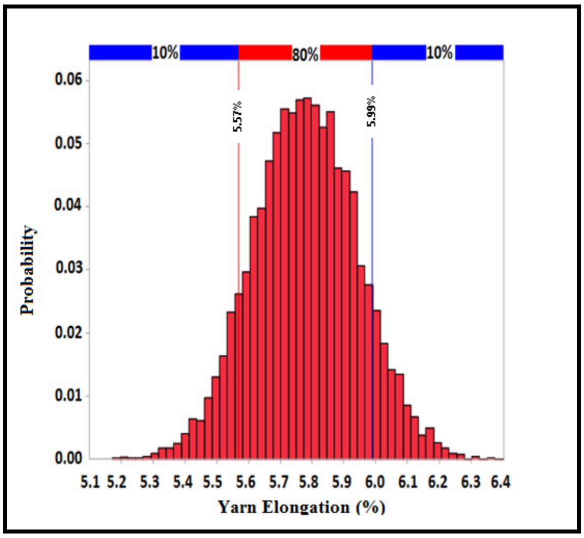

Fig. 1 − Histogram plot.

3.2. Sensitivity Analysis of Yarn Elongation Model

The spider sensitivity plot shown in Fig. 2; indicate that the yarn twist (tp) had the dominant negative influence on the yarn elongation model. Therefore an increase in yarn twist will result in a decrease in yarn elongation.

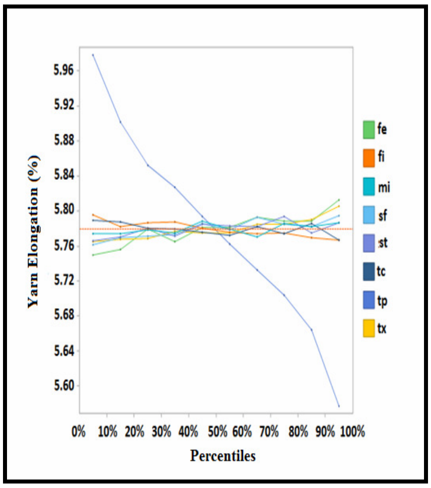

Fig. 2 − Spider Plot.

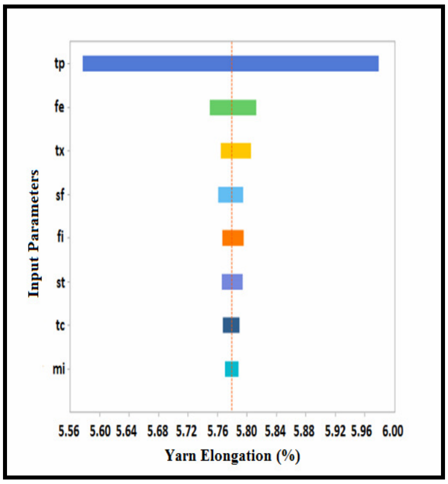

Fig. 3 − Tornado Plot.

The tornado sensitivity plot in Fig. 3, illustrates that in terms of yarn parameters yarn twist (tp) stands out as the most influential properties which affect yarn elongation in ring spun yarn. Yarn breakage in short staple ring spun yarn occurs either due to fibre slippage or fibre breakage (Lawrence, 2003). When yarn breaks due to fibre slippage, the hitherto twisted fibres have to be straightened. The straightening action has an effect of increasing the effective length of the fibre which in turn increases yarn length. This could be the reason yarn twist showed a strong impact during the prediction of yarn elongation. Apart from yarn twist, yarn linear density also showed strength influence on yarn elongation (Mwasiagi *et al*., 2007; Hunter, 1988; Üreyen *et al*., 2008). The process of stretching the fibre will be affected by other factors such as machine parameters and fibre properties. When the straightened fibre is further stretched until it breaks, the change in its length is defined as fibre elongation. The change in fibre length will cause a corresponding increase in yarn length. This could be the reason fibre elongation showed a strong impact during the prediction of yarn breaking elongation.

4. Conclusions

In this study yarn elongation were modelled by using statistical techniques. The parameters of the fibre samples were tested and recorded in terms of length, short fibre content, strength, elongation, fineness, micronaire value and trash. The yarn samples were also tested and recorded in form of elongation, twist and linear density. The fibre parameters, yarn linear density and twist were used as the inputs, while the yarn elongation was used as the outputs in the process of modelling the influence of fibre properties on yarn elongation. The statistical technique developed models that generated coefficient of regression (R) value of 0.840 for yarn elongation. The sensitivity analysis for statistical models showed that the most influential inputs on the yarn elongation model most were yarn twist, fibre elongation and linear density. The statistical modelling technique therefore was found to be significant method in yarn modelling because it was able to give the predicted value of the yarn parameters and also indicate the relative influence of the input variables on the modelled yarn parameters.

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MODELAREA INFLUENŢEI PROPRIETĂŢILOR FIBRELOR ASUPRA CARACTERISTICILOR LA ÎNTINDERE A FIRELOR FOLOSIND TEHNICI MONTE CARLO

(Rezumat)

Studiul proprietăţilor firelor din bumbac reprezintă o problemă importantă a domeniului textile, deoarece astfel de cercetări permit înţelegerea nu numai a caracteristicilor firelor, dar şi a aspectelor legate de filarea acestora. Adaptarea metodelor statistice a necesitat un volum de muncă semnificativ pentru a putea evidenţia efectul proprietăţilor fibrelor, a parametrilor tehnologici, precum al structurii firelor asupra rezistenţei firelor. Totuşi, determinarea influenţei caracteristicilor fibrelor asupra proprietăţilor firelor rămâne o problemă spinoasă, datorită neuniformităţii fibrelor de bumbac. Din acest motiv, utilizarea unor modele statistice tip simulări Monte Carlo pot deschide noi direcţii în modelarea comportării firelor. În această lucrare se prezintă un model statistic pentru predicţia comportării la întindere a firelor, folosind caracteristici ale fibelor şi firelor din bumbac kenyan. Se dezvoltă un model statistic, utilizând regresia lineară multiplă, proprietăţile la întindere a firelor fiind estimate printr-o simulare Monte Carlo. Modelele au fost folosite pentru a studia influenţa proprietăţilor fibrelor asupra alungirii firelor.

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WETTABILITY, A PROMINENT COMFORT FACTOR IN THE DEVELOPMENT OF FABRICS WITH ANTI-ALLERGIC PROPERTIES

BY

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Abstract. Transport of humidity in textile materials represents one of the most critical factors which could affect the physiological comfort. The textile materials with medical anti-allergic properties require a good sorption capacity of water and sweat from the dermis to the environment. A higher wettability of the fabrics, such as cotton, could help to manage dry or atopic skin. A wet skin could facilitate the penetration of potentially toxic substances. The higher state of humidity also favours growth and activity of microorganisms. Characterization of the wetting of textiles presents special research problems. The primary parameter reported to characterize wetting is contact angle. However, interfacial tension has also been used. The best measurement method depends on the nature of samples and the experimental constraints. In this work textile wettability has been measured using a special device called Tensiometer KRÜSS K100SF, specially designed for versatile and demanding applications in various fields like as research, development, and quality assurance. Different

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cotton textiles untreated and treated with an anti-allergic active principle were evaluated and the amount of liquid absorbed recorded during time have been reported.

Key words: wettability, comfort, medical textiles, anti-allergic active principle.

1. Introduction

Medical textiles represent a major growth area within the technical textiles industry and its range of applications continues to grow and increase in diversity with every new development. Medical textiles are used everyday in almost all healthcare activities, in the form of plasters, bandages, pressure garments, wound dressing, swabs, incontinence diapers, sanitary napkins, baby diapers, etc. (Kennedy & Mehmood, 2006). Textiles are also being used as sutures, orthopaedic implants, vascular grafts, artificial ligaments, artificial tendons, heart valves and even as artificial skins.

Recent advances in medical textiles to be used as extracorporeal devices are also significant; these include artificial kidney, artificial liver, mechanical lungs and so on. New materials are finding specialized applications like antimicrobial, hypoallergical and antifungal fibres and additives used in barrier fabrics, abdominal post-operative binders, applications in neurodermatitis treatment and various other wound management and surgical treatments. Although the type of fibre used and the fabric structure varies with the specific end use, all medical fibres must be non-toxic, non-carcinogenic, non-allergic and capable of being sterilized without suffering chemical or physical damage. In addition, for many applications absorbency is essential, favouring the use of cotton or viscose (Thamotharan, 2011).

The research proposes an approach to allergy symptoms in atopic individuals (who have an allergic predisposition) so as to prevent the disease or improve condition of the patient after an episode of allergy. This paper aims at making women's cotton pyjamas consisting of trousers and shirt for clinical trials in patients with allergic dermatitis.

Atopic dermatitis means inflammation of the skin from an unknown cause. Scientists found that genetic changes increase protease activity, causing a premature breakdown of the skin barrier. Environmental factors such as dust mites or soap also play a role in weakening the skin barrier. When the barrier is broken, contaminants can enter and cause inflammations, allergies or other diseases. A broken barrier also allows water to leave, making the skin feel dry to the touch (http://www.faqs.org/periodicals/201010/2156622381.html, 2010).

Cotton textiles, the traditional materials for various medical textiles, meet a number of requirements, such as comfort, draping, good tensile strength and steam permeability. However, they will not meet the requirements of the new standard which includes resistance to microbial penetration, resistance to liquid penetration and minimal release of particles (Abreu *et al*., 2003). In this work cotton has been loaded with propolis, which has recognized antibacterial properties (Grange & Davey, 1990; Cowan, 1999). Moreover, propolis has been reported as a local anaesthetic, thus helping to relief pain in the patient suffering from allergic dermatitis (Paintz & Metzner, 1979).

1.2. Wettability, an Important Factor for Medical Textiles

Moisture/liquid transport in textile fabrics is one of the critical factors affecting physiological comfort. Fabrics that rapidly transport moisture/liquid away from the surface of the skin make wearers feel more comfortable by keeping the skin dry. In conditions where wearers sweat a lot (*e.g.*, high level bodily activity), it is not only desirable for the fabric next to the skin to absorb liquid rapidly but also to transport it through the fabric promptly to avoid the discomfort of the fabric sticking to the skin (Liu *et al*., 2008). According to Ramesh and Koushik (2011), clothing comfort is influenced by the fluid transport properties of fabrics. The liquid wets the fibre surfaces and is transported through the spaces between the fibres. Wetting occurring through the fibre pores depends on capillary forces.

Textile materials with anti-allergic properties require a good sorption capacity of water and sweat from the dermis to the environment. A higher wettability of the fabrics, such as cotton, could help to manage dry or atopic skin (Yao *et al*., 2011). A wet skin could facilitate the penetration of potentially toxic substances. The higher degree of humidity also favours growth and activity by microorganisms (Bo, 2008). Moreover, fabric to skin friction is increased in the wet state and this may worsen the inflammation disease.

2. Materials and Method

A knitted fabric has been made of 100% cotton with an interlock structure using a yarn with count Nm 60/1. The fabric is alkaline cleaned and bleached with hydrogen peroxide; the next stage (grafting stage) is the reaction with monochlorotriazinyl-beta-cyclodextrin (MCT-β-CD) used as complexing agent to form an inclusion compound with some active principles. Three different natural active principles were used in this study. These active principles (A.P) detain upper-most anti-allergic properties. The A.P. used were an aqueous extract of *Viola tricoloris Herba*, an alcoholic solution of menthol and an alcoholic solution of propolis with therapeutic doses known by *in vivo* tests (to determine the optimal amount of product). By means of *in vivo* tests, therapeutic doses of A.P. were obtained which were applied to the textile material. For a patient of 60 kg body weight, the amount of drug applied to the textile material is obtained by multiplying the therapeutic dose by 60. Active principles have been applied on the textile surface by a spraying technique followed by heat treatment for 4 h at 50°C. We expected that these active molecules encapsulated in the cyclodextrin cavities do not evaporate and are stored over a long period of time.

We have mentioned that cotton textiles meets a lot of requirements, but also the main problems associated with 100% cotton fabrics are their high absorbency and slowness of drying which cause a sensation of wetness (Rengasamy, 2011). In our case these problems can become an advantage because small amounts of water as they are set free from the skin are essential for the release of the active principles. In practice different textile products treated with cyclodextrins which are encapsulated different flavours will start smelling only when they are near to the skin. Simultaneously organic substances from sweat are complexed (Buschmann *et al.*, 2001).

A capillary rise method was applied to evaluate the liquid sorption for fabrics untreated and treated with active principles. The quantitative wetting of fabrics was determined by surface tension and contact angle (θ). As a method for determining the mechanism of wetting it can be use contact or immersion method. Raw materials used in the textile industry have a low energy surface (except glass fibres and minerals), but also forms a capillary system whose physical properties have a decisive role in wetting process (Mitu, 2000).

Generally, textile material is a porous surface, with micro and macro capillaries containing a high amount of air which in the process of wetting is replaced with water. Surface tension between water and fabric depends on the constituent materials of the yarn, the yarn structure, on the capillary system, the swelling capacity and the presence of hydrophilic groups. Wetting ability by capillarity can be appreciated by the following ways: measure the contact angle of water droplet on the surface of the material, measure the wicking effect or absorption of liquid droplet off a glass plate (Mitu, 2000). In our research we followed a wicking capillarity method.

The paper presents only the results for untreated and fabrics treated with alcoholic solution of propolis.

Experiments were carried out on in the laboratory of high textile technology (LATT), in the premises of Città Studi (from Biella, Italy). Textile wettability has been measured using a special device called Tensiometer KRÜSS K100SF (Germany), specially designed for versatile and demanding applications in various fields like as research, development, and quality assurance. The instrument has different modules to measure: surface and interfacial tension of liquids, sorption, contact angle, the density of liquids and critical micelle concentration.

The Tensiometer is a very sophisticated hanging balance which measures the weight increase of a fabric sample, when the fabric is wetted by capillary rise. In the measurements, the hanging fabric is put in contact with the surface of a liquid without being immersed.

The fabric is considered as a bundle of capillaries. This means that for the calculation of the advancing angle, which corresponds to the contact angle between the solid and the liquid, the Washburn equation can be used:

$$\frac{l^2}{t} = \frac{\sigma_l \cdot r \cdot \cos\theta}{2\eta} \tag{1}$$

where: l is the flow front, t − the flow time, σ_l − the surface tension of liquid, r − the capillary radius, θ − the advancing angle and η − the viscosity of the liquid.

The capillary radius for the fabric must be replaced by a quantity which describes the orientation of the capillaries and the mean radius:

$$\frac{l^2}{t} = \frac{\sigma_l \cdot \left(c \cdot \overline{r}\right) \cdot \cos\theta}{2\eta} \tag{2}$$

The front flow is estimated from the increase in weight, the liquid density and the sample section. The viscosity and surface tension of the liquid are known, only two unknown remain: the required advancing angle and the material constant $c \cdot \overline{r}$. This is why a measurement with an optimally wetting liquid (*e.g*. hexane, whose virtual angle is virtually 0°), is carried out first; this gives a value for cos θ of approximately 1.

When the term: $2\eta \frac{l^2}{\sigma_i}$ is plotted against time a linear section is obtained whose slope is $c \cdot \overline{r}$, *i.e*. the required constant.

For measurements with other liquids, this constant can be inserted into the Washburn equation, so that the advancing angle θ can be determined for other liquids.

The fabrics were conditioned for 12 h in the atmospheric condition of temperature 20 ± 2ºC and relative humidity 65% ± 2% and then were cutted into strips (6 x 2.5 cm) for each direction (vertically and horizontally) and were clamped vertically with a clip like as in Fig. 1.

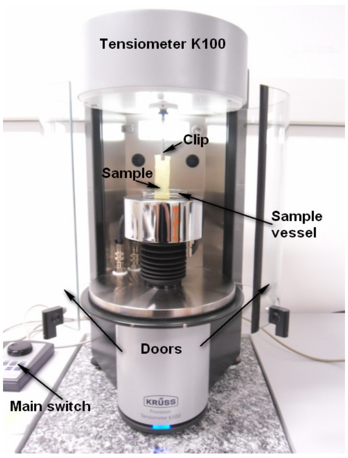


Fig. 1 − Tensiometer KRÜSS K100SF.

The knitted fabrics and testing directions of fabric were coded in following way presented in Table 1.

Table 1
The Code of Knitted Fabrics Used in Investigation

Code	Explication
A	Untreated knitted fabric, alkaline cleaned and bleached
B	Treated knitted fabric with propolis
D1	Wale direction fabric
D2	Course direction fabric

The liquid used in the capillary rise experiments was artificial sweat. It was simulated with 0.5 g L-histidine monohydrochloride H_2O (99% purity, Vickers Laboratories Limited), 5.0 g sodium chloride (99% purity, Vickers Laboratories Limited), 2.2 g sodium dihydrogen orthophosphate $2H_2O$ (99% purity, Vickers Laboratories Limited) and pH = 5.5 with hydroxide sodium 0.1 M.

During the experiments, the textile wettability was determined in the following stages:

− Surface tension determination of artificial sweat through the Wilhelmy plate method (Fig. 2);

− Fabric capillary factor (C_1 and C_2) determination using n-hexane solution (95% purity, Sigma Aldrich), two determinations for each sample;

− Sorption determination of simulated sweating solution through contact angle (θ_1, θ_2 and θ_3), three determinations for each sample.

In phase I, a metal plate was put in contact with the sweat solution surface, as shown in Fig. 2. From the measurement of the force exerted by the liquid raising on the two faces of the plate it is possible to evaluate the surface tension of the sweat solution.

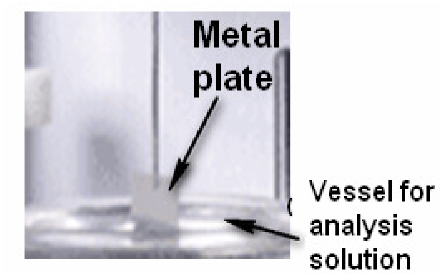


Fig. 2 − Surface tension determination by the Wilhelmy plate method.

It can be observed (in Table 2) that the sweat solution surface tension is considerably lower than that of water (73 mN/m). Since the capillary rise depends proportionally on the liquid surface tension, it is important to use a sweat solution rather than water. The surface tension values reported in the table have been obtained through the plate method before proceeding with the wicking measurements of the fabric.

Table 2
Values of Sweat Solution Surface Tension

Sample	Directions of fabrics	Surface tension [mN/m]
A	D1	38,247
A	D2	31,891
B	D1	26,135
B	D2	25,624

In phase II, a geometric factor depending on the dimension and tortuosity (sinuosity) of the capillaries of the fabric is evaluated. This factor can be determined from the Washburn equation (Metoda Washburn, 2010) by measuring the capillary rise of a liquid of very low surface tension (n-hexane) which gives a complete wettability of the fabric (contact angle $\theta = 0°$).

Finally, in phase III, the capillary rise of the sweat solution in the fabrics has been evaluated by putting the fabric sample in contact with the surface of the sweat solution and recording the weight gain with time. By knowing the geometric factor and the weight gain, the contact angle is estimated.

The data acquisition for n-hexane solution was logarithmic and for sweating solution the data acquisition was linear.

3. Results and Discussions

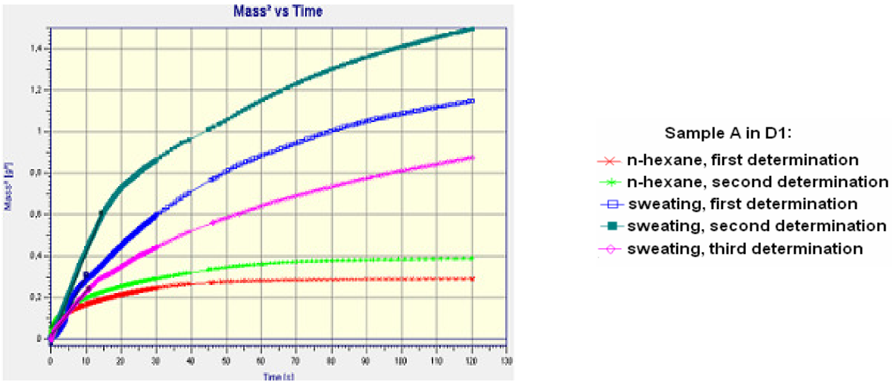
The data have been collected using the software LabDesk™. Characterization of the wetting of textiles presents special research problems. The best measurement method depends on the nature of samples and the experimental constraints. We have taken in account only the weight gain of liquid for the fabrics during the time.

In terms of hydrophilicity the fabrics can absorb water/liquid quickly, slowly or not at all. In our case, wetting capacity for untreated samples is very high, it can be observed from the height of liquid which has risen approximate 6 cm in 120 sec. The square of the amount of liquid absorbed (the mean of weight gain in g^2) *vs.* time (120 sec) for all investigated fabrics are presented in Table 3.

Table 3
The Mass of Absorbed Liquid versus Time

Sample	Directions of fabrics	Mass [g^2]	Time [sec]
A	D1	1.186	120
A	D2	0.603	120
B	D1	0.083	120
B	D2	0.084	120

The diagrams with curves for all analysed samples are illustrated in Fig. 3 and Fig. 4. The amount of liquid absorbed was recorded during time.



a

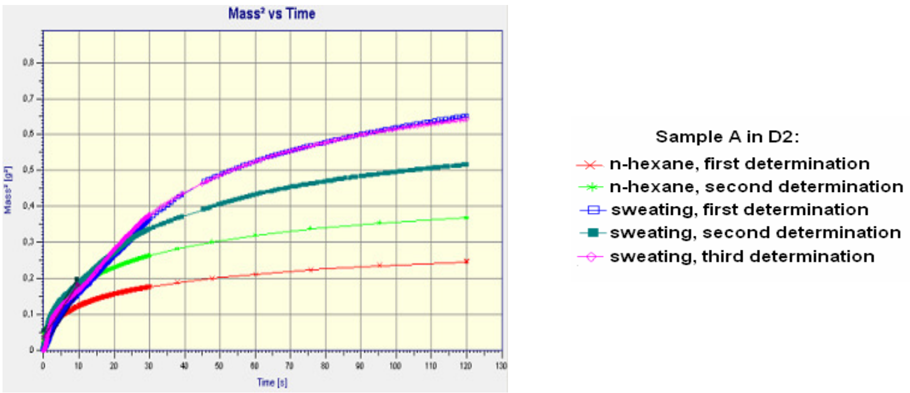


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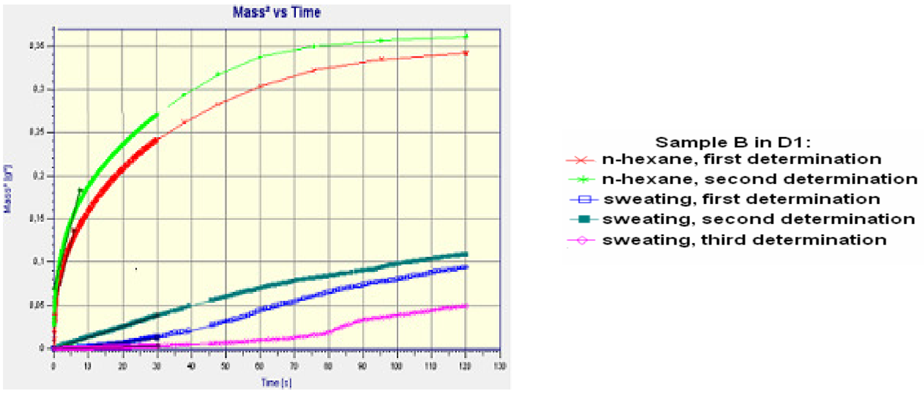
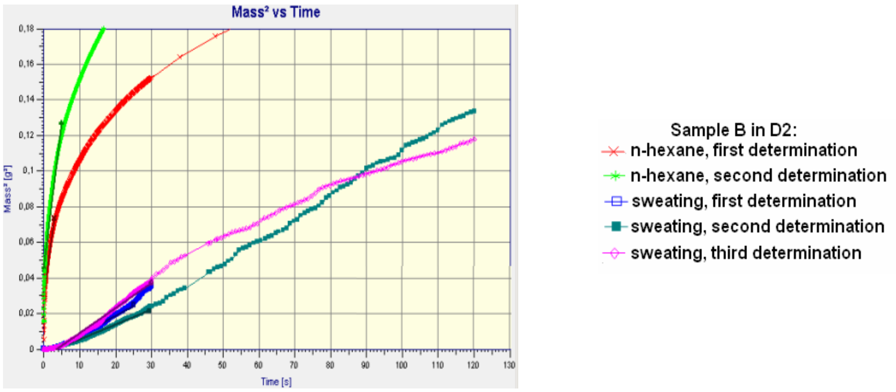
Fig. 3 – Diagram of untreated sample in both testing direction: *a* − Diagram of untreated sample in wale direction, *b* − Diagram of untreated sample in course direction.

From Fig. 3 it can be observed that the mean sorption of artificial sweat in course direction and wale direction is different for the untreated fabric: the amount of liquid absorbed for sample A in course direction was lower (0.603 g^2/120 s) in comparison with the wale direction (1.186 g^2/120 s). On the other side, no appreciable difference was observed in the two direction measurements for the treated fabric. In particular, the amount of absorbed liquid is much less for fabrics treated with the active principle than for untreated fabrics and is the same for both testing directions: 0.083 g^2 for D1 and 0.084 g^2 for D2 in 120 sec. This result may be due to propolis, which covers the surface of the fabric making it more hydrophobic.



a



b

Fig. 4 – Diagram of treated sample with propolis in both testing direction: *a* − Diagram of treated sample in wale direction, *b* − Diagram of treated sample in course direction.

However, it is noteworthy observing from Fig. 3 that measurements are not that reproducible. This may be due to a not optimal contact between the fabric and the liquid. Therefore, more effort is needed to optimize these measurement methods for fabrics, especially knitted ones, since the preparation of the sample may be difficult due to their high deformation capability.

4. Conclusions

The knitted fabric made by 100% cotton with an interlock structure presents a higher wettability when are untreated than treated with MCT-β-CD and propolis. The decrease of wettability may be due to the hydrophobic behaviour of propolis which covers the surface of the fabric. It must be noted that this method is not entirely optimized for determining the wetting capacity of the knitted materials under investigation.

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HIDROFILIA, UN FACTOR REMARCABIL DE CONFORT ÎN DEZVOLTAREA TEXTILELOR CU PROPRIETĂȚI ANTIALERGICE

(Rezumat)

Transportul umidității în materialele textile reprezintă unul dintre cei mai critici factori care pot afecta confortul fiziologic. Materialele textile medicale cu proprietăți antialergice necesită o bună capacitate de sorbție a apei şi transpirației dinspre dermă către mediul ambiant. Hidrofilia avansată a textilelor, cum ar fi bumbacul, ar putea ajuta în ameliorarea pielii uscate sau atopice. O piele umedă ar putea facilita penetrarea substanțelor potențiale toxice. De asemenea, caracterul avansat al umidității favorizează creşterea şi activitatea microorganismelor. Caracterizarea hidrofiliei textilelor prezintă probleme speciale de cercetare. Parametrul primar raportat în caracterizarea proprietății de udare este unghiul de contact. Totuşi a fost luată în calcul şi tensiunea interfacială. Cea ma bună metodă de măsurare depinde de natura probelor şi de constrângerile experimentale. În această lucrare hidrofilia a fost măsurată folosind un aparat special, numit Tensiometer KRÜSS K100SF, special conceput în diverse domenii cum ar fi cel de cercetare, dezvoltare şi asigurarea calității. S-au evaluat diferite materiale textile din bumbac netratate şi tratate cu un principiu activ antialergic şi s-au raportat cantitățile de lichid absorbit funcție de timp.

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KNITTED STRUCTURES WITH 3D SURFACE GEOMETRY FOR COSMETOTEXTILES

BY

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Abstract. The paper presents some knitted structures with 3D surface geometry that can be used as cosmetotextiles, for massaging textile support with incorporated microcapsules. The massaging aspect of the knitted fabrics will be emphasized through subjective analysis using different subjects, as well as through the analysis of psycho-sensorial properties of the fabrics – friction test and surface test (UST).

Key words: knitted structures with 3D surface geometry, massage, effect evaluation.

1. Introduction

The massaging effects on the human body are classified based on the way they are produced. The energy of different massaging manoeuvres is transformed by the different types of receptors into nervous response,

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generating a set of direct (mechanical) and indirect (reflexes) reactions.

The direct effects, produced mechanically, are based on the physical chemical properties of the skin (skin stress, elasticity, contraction and strength). Their goal is to maintain the balance between peripheral and deep circulation, helping the return flow and the lymph sorption.

The indirect effects are of reflexive nature and are sensed deep within the body or at the distance. The metamer (the influence zone of the rachidian nerve from its origin to the zone that it controls) represents the anatomical functional support. This path is made by the dermatome (the nerve portion within the dermis), miotom (the part in the muscles), angiotom (the part in the blood vessels), sclerotom (periosteum, bones), viscerotom (viscera) and neurotom. This way the influence is at circulatory system, as well as muscle and viscera levels.

The massage acts on the 3 main functions of the skin:

– *Excretion*, the elimination of metabolic products through sweat and sebaceous gland;

– *Thermal adjustment*, given by skin vessel motility;

– *Reception*, through the free nervous endings or special receptors.

The mechanical massage made by a masseur can be replaced by self-massage using a textile product with massaging effect through the friction between the fabric and the skin caused by the body movement. This process is more economical and takes more time (it takes place whenever the product is used) and is more comfortable. Microcapsules can be added to this massaging structures, incorporating essential oils used in aroma therapy or different cosmetic products (creams and oils for weight loss, anti cellulite, hydrating, etc), according to their end-use. Such textile products are also known as cosmetotextiles.

The paper intends to study some types of massaging structures made of bamboo yarns, as well as the effects these fabrics have on the human body during use.

2. Experimental Work

The experimental work included 6 types of knitted fabrics made of 100% bamboo yarns, count 34 Nm and 100% cotton yarns with the same count. The fabric variants were produced on a Stoll flat knitting machine, gauge 18E. Figs. 1 to 6 present the programming of each structure variant (Penciuc, 2008; Stoll, 2007; Comandar, 2005).

Table 1 presents the questionnaire given to 20 human subjects, regarding the massaging effect of the 6 fabric variants.

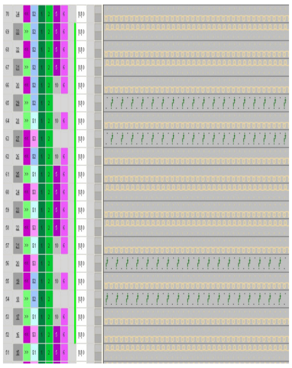

Fig. 1 − Knitted structure - variant T1.

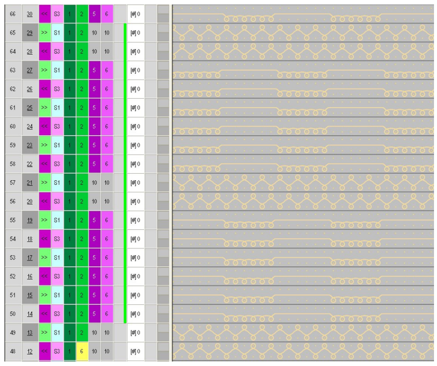

Fig. 2 − Knitted structure - variant T2.

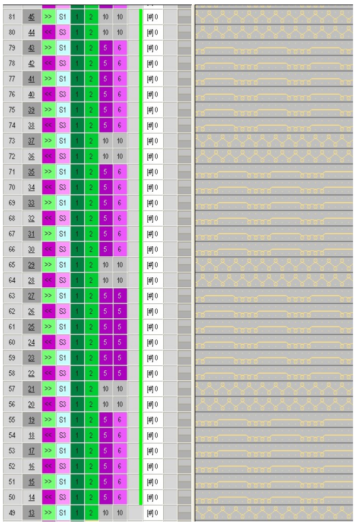

Fig. 3 − Knitted structure - variant T3.

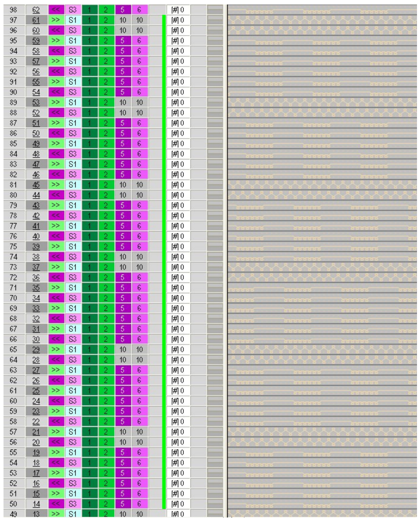

Fig. 4 − Knitted structure - variant T4.

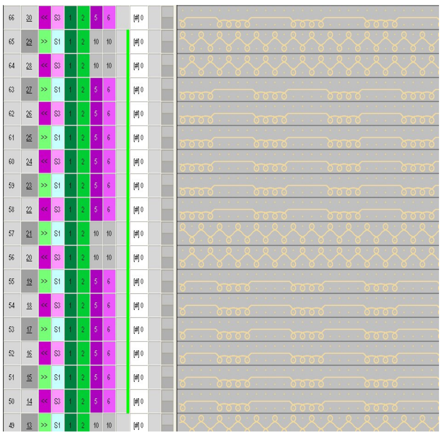

Fig. 5 − Knitted structure - variant T5.

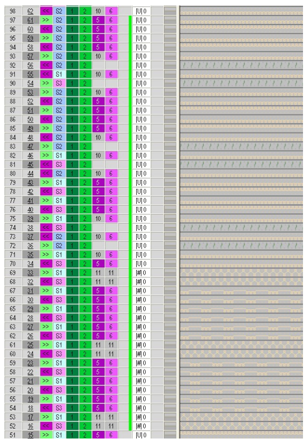

Fig. 6 − Knitted structure - variant T6.

The fabric friction was tested on the M264 Shirley Fabric Friction Tester - SDL ATLAS (Fig. 7), measuring the angle to which the fabric begins to slide on the slope covered by an abrasive fabric. The 5cmx25cm samples were cut along the wale and course direction, 5 samples each.

Fig. 7 − M264 Shirley Fabric Friction Tester.

The tactile behaviour of the fabrics was tested on a Universal Surface Tester (Fig. 8), using two types of sensors - ball 1.8 and papillary. The first smaller feeler gives the real image of the fabric surface geometry, while the second, made of silicon, generates the same effect with passing a finger over the samples.

Fig. 8 − Universal Surface Tester

3. Results and Discussions

The questionnaire asked the subjects to order the fabrics at three levels: 1 – less massaging, 2 – massaging and 3 – strong massaging effect. The results are presented in Table 1.

Table 1
Results for the Questionnaire Regarding the Massaging Effect of the Fabric Variants

	T1	T2	T3	T4	T5	T6
1	2	3	3	2	2	3
2	1	3	3	3	2	2
3	1	2	3	3	2	2
4	1	2	2	3	3	3
5	1	3	2	3	3	2
6	1	2	2	3	2	2
7	2	2	2	3	2	3
8	2	3	2	3	3	2
9	1	2	3	2	3	3
10	1	2	2	3	2	3
11	2	2	3	3	2	3
12	2	2	3	3	2	3
13	1	2	3	3	2	3
14	2	3	2	3	3	3
15	2	2	3	3	3	3
16	2	3	2	3	2	3
17	1	3	2	3	3	3
18	2	3	2	3	2	2
19	1	3	2	3	2	3
20	1	2	3	2	2	2
media	1.45	2.45	2.45	2.85	2.35	2.65

The graphic illustrated in Fig. 9 shows that the fabric variants with strongest massaging effect are fabrics T4 and T6, followed closely by variants T2 and T3, while variant T1 has the less massaging effect.

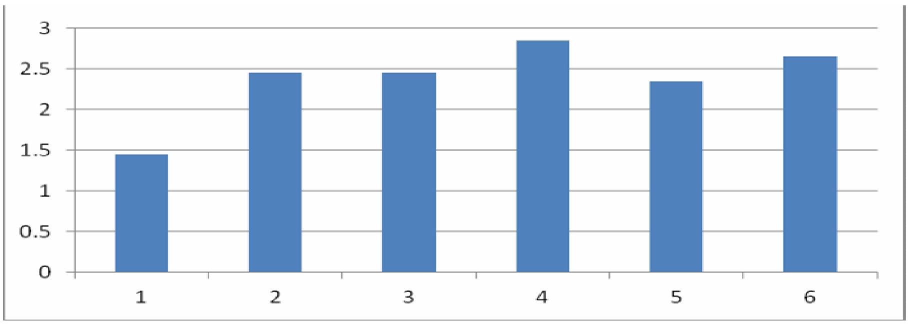

Fig. 9 – The subjective evaluation of the massaging degree for the 6 fabric variants.

Tables 2 and 3 are presenting the results for the friction testing of the fabrics on both wale and course direction.

Table 2
The Sliding Angle for Bamboo Fabrics

bamboo	Test	B1	B2	B3	B4	B5	B6	jersey
course	1	15.5	21	19	14.5	14	11.5	8.5
	2	15.5	20.5	19.5	17.5	13.5	10.5	9
	3	15	21	21.5	18.5	13	12	9.5
	4	14.5	23	22.5	20	13.5	12.5	10
	5	16.5	19	19.5	17.5	13	11	8.5
average		15.4	20.9	20.4	17.6	13.4	11.5	9.1
wale	1	12.5	16.5	12	16.5	16	12.5	8.5
	2	11.5	16.5	13	16.5	15.5	13.5	9
	3	12	17.5	9.5	17	14	13.5	8.5
	4	11.5	17.2	10.5	17.5	13.5	12.5	9.5
	5	12	17	11	18.5	14.5	13	8
average		11.9	16.9	11.2	17.2	14.7	13	8.7

Table 3
The Sliding Angle for Cotton Fabrics

cotton	Test	C1	C2	C3	C4	C5	C6	jersey
course	1	28	36.5	37.5	28.5	22.5	12.5	13
	2	25.5	37.5	38.5	27	22	15	15
	3	27.5	35	39	28	22.5	13.5	13.5
	4	25.5	36.5	35.5	25.5	22	15.5	13
	5	26.5	35	39.5	28.5	2o	14	12
average		26.6	36.1	38	27.5	21.8	14.1	13.3
wale	1	20.5	22	13.5	20.5	29	18	12
	2	17	21	13.5	25	23	18	11.5
	3	17	16	17.5	23	23	18.5	12
	4	15	23.5	20	24	26	22	13
	5	14.5	20	14.5	24.5	23	21	14
average		16.8	20.5	15.8	23.4	24.8	19.5	12.5

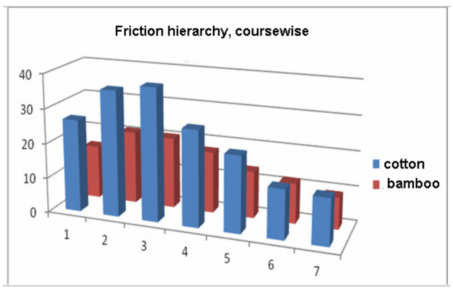

Fig. 10 − Variation of the sliding angle for cotton and bamboo fabrics, course direction.

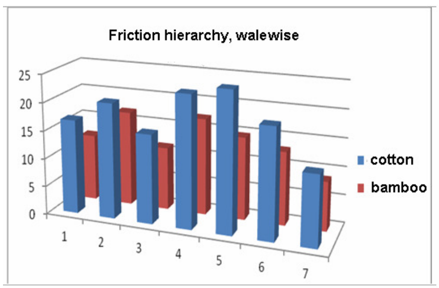

Fig. 11 − Variation of the sliding angle for cotton and bamboo fabrics, wale direction.

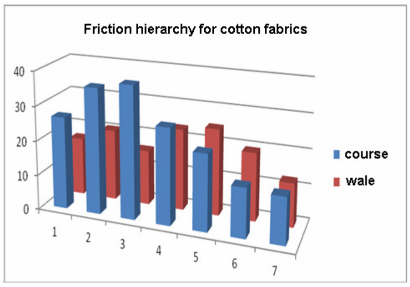

Fig. 12 − Variation of the sliding angle for cotton.

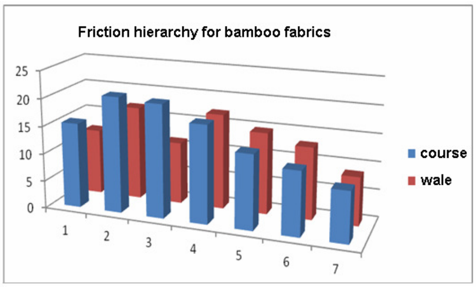

Fig. 13 − Variation of the sliding angle for bamboo fabrics.

Figs. 10 and 11 show the raw material influence the friction behaviour of the samples, the bamboo fabrics having a lower friction coefficient than the cotton variants on both testing directions. The graphics from Figs. 12 and 13 are showing that the sliding angle is higher on the course testing direction with the exception of variant T6. This suggests that the fabric direction within the product is important and that the massaging effect is stronger when the fabrics are used course wise.

The surfaces of the fabric variants were analysed with Universal Surface Tester using two types of sensors – ball 1.8 and papillary, conform ISO 4288 (1996). Figs. 14 to 16 exemplify the 3D and 2D results. This analysis also gives information regarding the permanent, elastic and total deformation, caused by the passing of the two sensors over the fabric surface. The first passing is without a pretension of the feeler, followed by a 70 mN pretension passing (equivalent to pressure the fabric may cause at skin level during use) and another passing without pretension. The results illustrate the elastic, permanent and total deformation.

The 2D diagrams (*egg*. Fig. 16) are used to calculate the degree of roughness for the 6 fabric variants presented in Tables 4 and 5.

Table 4

Permanent, Elastic and Total Deformations and Degree of Roughness, Determined Using Ball 1.8 as Feeler

Fabric variant	Feeler - ball 1.8						Degree of roughness Rz [μm]
	Permanent deformation [μm]		Elastic deformation [μm]		Total deformation [μm]		
	mean	st. dev.	Mean	st. dev.	Mean	st. dev.	
1	77.03	56.077	814.28	176.907	891.31	175.997	748
2	221.48	111.99	1202.65	384.096	1424.12	381.286	760
3	133.13	50.691	682.04	270.764	815.17	282.909	702
4	143.17	79.816	779.59	191.899	922.77	232.233	719
5	163.3	66.645	839.46	197.064	1002.76	221.204	795
6	72.05	298.87	867.5	437.677	939.55	295.005	801.8

Table 5

Permanent, Elastic and Total Deformations and Degree of Roughness, Determined Using Papillary Feeler

Fabric variant	Feeler - papillary						Degree of roughness Rz [µm]
	Permanent deformation [µm]		Elastic deformation [µm]		Total deformation [µm]		
	mean	st. dev.	Mean	st. dev.	Mean	st. dev.	
1	67.03	50.949	408.4	36.892	475.43	64.028	110
2	56.76	48.865	625.16	82.092	681.92	82.346	260.5
3	86	80.894	559.72	113.272	665.71	170.366	263.1
4	45.81	22.009	478.06	120.46	523.87	118.979	728
5	70.11	57.267	584.14	69.657	654.25	89.466	216.7
6	47.11	25.173	526.01	72.477	573.12	79.545	295.3

where: R_z – maximum height of the profile = average o f all heights calculated for each length, as shown in Fig. 14.

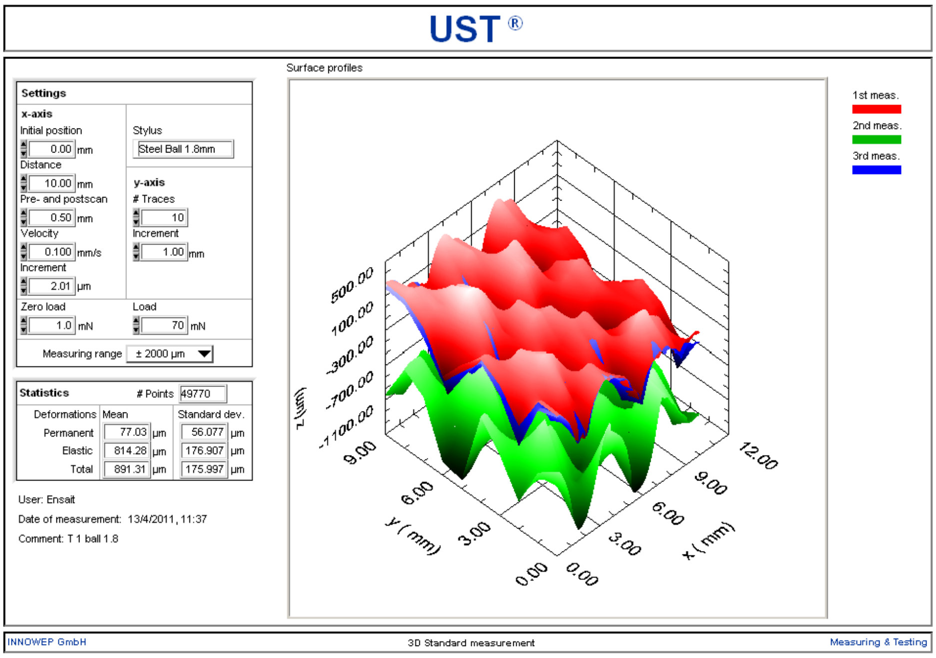

Fig. 14 − 3D image on UST, with ball 1.8 feeler, for fabric variant T1.

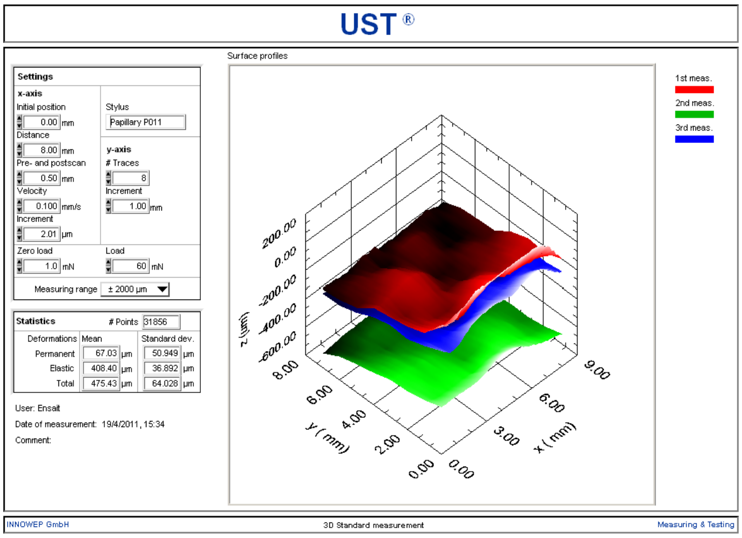

Fig. 15 – 3D image on UST, with papillary feeler, for fabric variant T1.

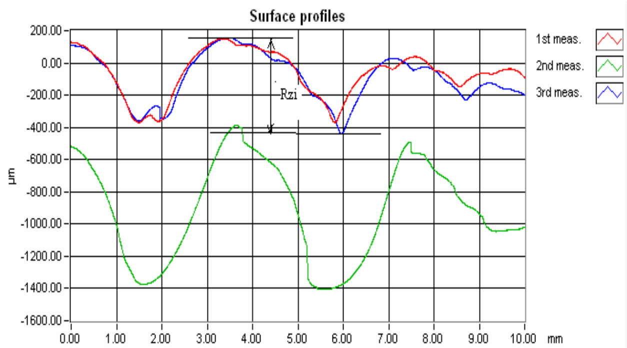

Fig. 16 – Calculus of the degree of roughness for a testing length.

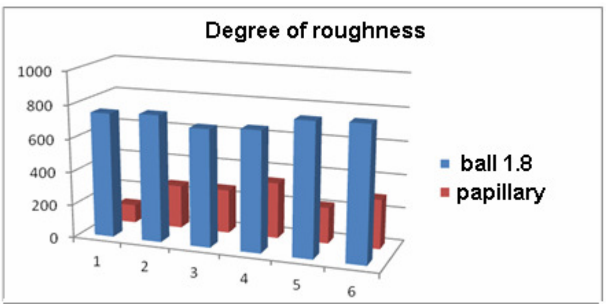

Fig. 17 – Degree of roughness for the fabric variants, determined with ball 1.8 and papillary feeler.

The graphic presented in Fig. 17 shows that the degree of roughness obtained using ball 1.8 feeler is higher than the one determined with the papillary feeler. The difference is normal, due to the fact that the lower dimensions of the ball offer more accurate information regarding the surface geometry of the fabrics. The type feeler also influences the roughness hierarchy of the fabric variants. For example, following the surface analysis with ball 1.8 feeler, variant T6 has the highest degree of roughness, while after the analysis made with the papillary feeler, the highest value belongs to fabric variant T4.

Comparing the graphics presented in Figs. 9 and 17, there a certain similarity in the hierarchy of the knitted fabrics given by subjective, respectively objective evaluation.

4. Conclusions

The long massages in specialised rooms can be replaced by the use of cosmetotextiles made of massaging knitted fabrics, that are less expensive, take more time (as long as the product is worn) and are effective.

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TRICOTURI CU SUPRAFEȚE CU GEOMETRIE 3 D PENTRU COSMETOTEXTILE

(Rezumat)

Lucrarea prezintă tricoturi din bătătură cu o suprafață cu geometrie 3D, care pot fi folosite pentru cosmetotextile, ca suport pentru depunerea de microcapsule. Se studiază efectul de masaj al tricoturilor utilizând analiza prin metode subiective, pe un grup de subiecți, dar şi obiective, prin definirea caracteristicilor psiho-senzoriale – determinarea comportării la frecare şi analiza de suprafață (UST).

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MODERN TECHNIQUES FOR CUSTOMIZED FOOTWEAR DESIGN

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Abstract. This article proposes a complete package of applications for customized footwear design using Delcam-Crispin software. Revolutionary CAD/CAM systems are the next generation of design solutions and engineering computer-aided design for the shoe industry. Designed exclusively for use with the latest operating systems and environments, they provide the full range of utilities in dedicated packages that are among the most intuitive and easy to use, compared to the currently products on the market. 3D computer aided design techniques (3D CAD) enables direct modelling of footwear on the last, so even before the product is made it can be analysed in terms of aesthetic, functional, technological and economic criteria. Starting with a last, it can rapidly be designed a complete footwear model, in any colour or texture combination. The result is a 3D realistic view of the product, ideal for presenting it to the customers, buyers or producers. Whether it uses 3D or 2D CAD systems, the user must take into account the criteria arising from the functions that footwear must meet.

Key words: footwear, last, design, modelling, customer.

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1. Introduction

Customers are looking for two basic characteristics when they purchase a footwear product: footwear appearance and its dimensional comfort. The footwear that does not correspond to the foot shape and dimensions and also it does not take over/absorb the foot modifications that appear while walking is the main cause for prevalence and evolution of structural and functional foot anomalies. Thus, the health of the entire body is affected, too (Mihai *et al*., 2009; Mihai & Curteza, 2005).

For a long period of time manual methods were used to retrieve the foot and respectively the shoe parameters, but the development of 3D scanning devices and the possibility of 3D visualization and modelling, the automatically analysis, the finding and interpreting patterns made it possible to develop different models of footwear depending on the foot conformation (Savadkoohi & De Amicis, 2009).

The last is the basic instrument for the footwear constructive design and for footwear manufacturing process. The lasting process is done with the uppers on the last, so, the shape and dimensions of the last will determine the shape and dimensions of the footwear. Dimensional comfort when wearing a footwear product is determined by this correspondence between the foot and the interior space of the shoe (Farrell & Simpson, 2003; Fujita & Yoshida, 2004).

2. Method

The last is the basic instrument for the footwear constructive design and for footwear manufacturing process. The lasting process is done with the uppers on the last, so, the shape and dimensions of the last will determine the shape and dimensions of the footwear. Dimensional comfort when wearing a footwear product is determined by this correspondence between the foot and the interior space of the shoe. Designing a new last is firstly based on foot anthropometrics and biomechanics. There are restrictive factors affecting the last's shape and its dimensions: acceptable limits of foot tightening by the footwear, modification of foot dimensions while walking, footwear constructive type, materials characteristics, and footwear manufacturing technology. Also, one has to take care of the general design requirements of the footwear. The footwear is considered as being comfortable when, throughout its shape and inner dimensions, it helps the foot to achieve its functions.

On these lines, by using the Compare module of *LastMaker-Delcam Crispin* software, a last can be transformed in different areas, toes (Fig. 1), heel, and length, width, in order to meet consumer needs and to be in line with the latest trends. This template can be done in a 2D design software starting form a picture of the last at a scale 1:1. When is imported in *LastMaker*, the user has to choose the 3 points that defines the template and when is accepted, the toe cap will be changed so that will fit the foot (Fig. 2).

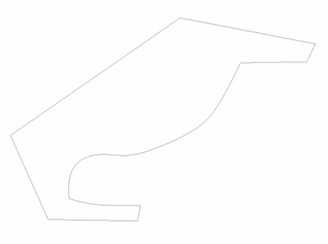


Fig. 1 − Template for the toe cap.

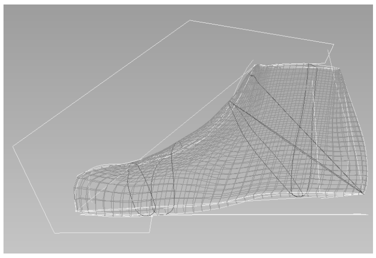


Fig. 2 − Modifying the last using a template.

When the final shape of the last is obtained, the data are exported and transmitted to a CNC machine or to a rapid prototyping equipment and a real last could be manufactured and, on the virtual last, the 3D design will be made.

3. Results and Discussions

3.1. 3D Modelling and 2D Design

3D ShoeDesign module of Crispin-Delcam integrated design system it is a software solution designed for virtual models, starting from a 3D predefined shape, respectively the last shape from the data base. The shoe model proposed can be obtained in a great variety of colour combination, textures, and panels. This software work facilities (sessions) are made of useful instruments available to the designer which has the possibility of manipulating any model in the virtual space, like, for example: establishing the positioning lines and control points, flattening the tree-dimensional surface of the model to obtain the patterns, visualize the model by rotating it with various angles, the simultaneous view of two-dimensional design (patterns) and the 3D model, the simultaneous opening of windows-work sessions. By using another module form Crispin-Delcam software, the lasts are imported, basic and model lines are created and panels with specific thickness, materials and textures are defined. These 3D lines and panels are exported and modified in a 2D application, namely pattern engineering; thus the entire set of cutting patterns will be resulting at the end.

Crispin Delcam CAD/CAM integrated system has some software modules that allow the following operations:

− last preliminary process, establishing position line;

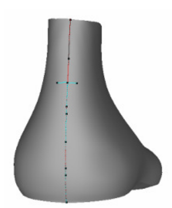


Fig. 3 − Posterior symmetry line.

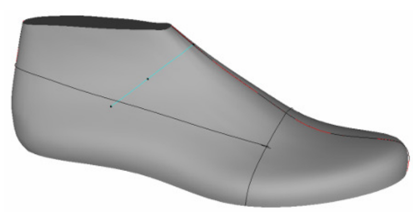


Fig. 4 − Quarter and vamp’s lines.

– 3D model drawing, directly on the last, visualize the model form different angles;

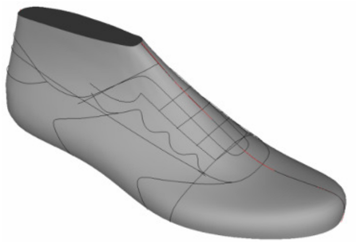


Fig. 5 – Exterior design lines.

Fig. 6 – Interior design lines.

– development of the model according with reality (colour, textures, seams, logos, accessories, ornaments);

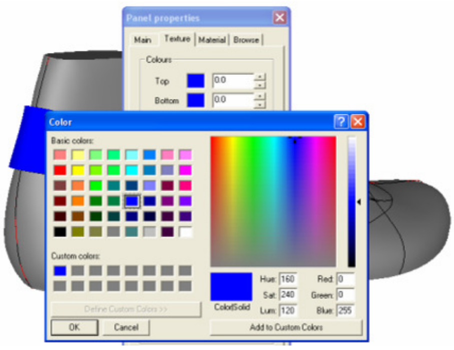


Fig. 7 – Patterns colour.

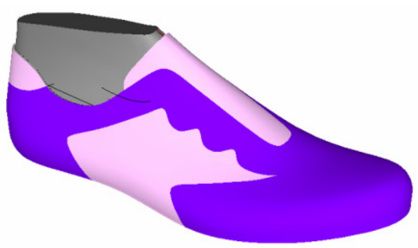
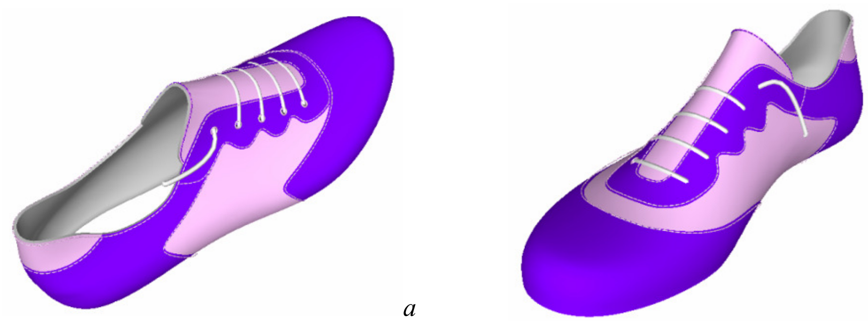


Fig. 8 – Footwear model’s patterns.

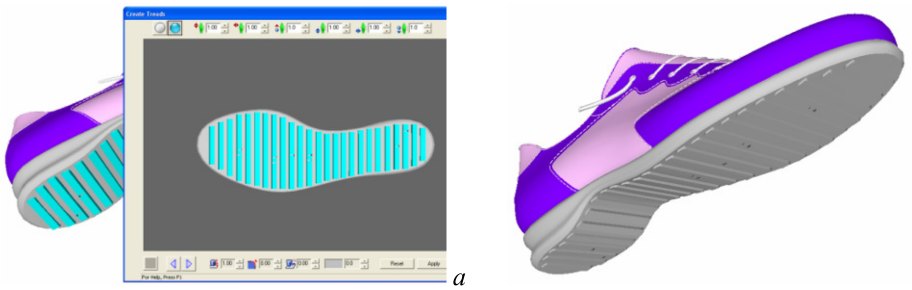
– development of the basic model in order to obtain a collection of models which will be presented to the beneficiary before being physically made;



a *b*

Fig. 9 – Footwear’s feature (laces, seams).

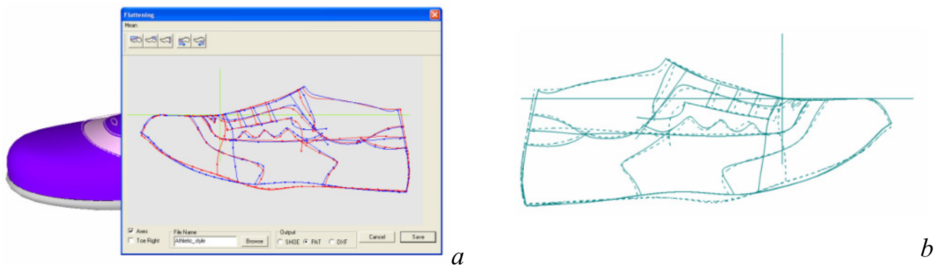
− sole and heel designed directly on the last, in harmony with the model designed;



a *b*

Fig. 10 − Footwear’s sole and sole skid model.

− flatten the 3D model;



a *b*

Fig. 11 − Flattening the model.

3.2. 2D Design – Engineer

Engineer software allows producing 2D footwear patterns in the fastest possible time, allowing one to get sample and graded patterns to manufacturing and tooling faster. Fast, accurate grading saves the cost of outside grading by a third party, whilst helping to control the costs involved with tooling.

With Delcam CRISPIN Engineer one can:

− flattened forms and style lines are used in order to create a model;

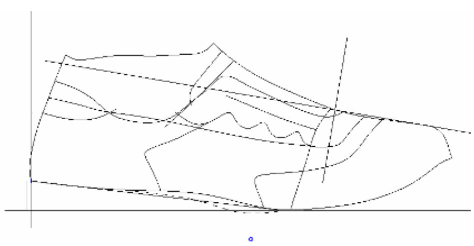


Fig. 12 − Basic lines design.

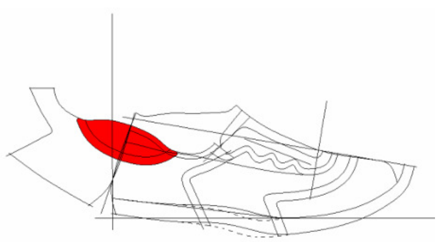


Fig. 13 − Back tab facing pattern.

– apply offsets for skiving and folding the margins, markers for alignment and stabs for eyelets and perforations;

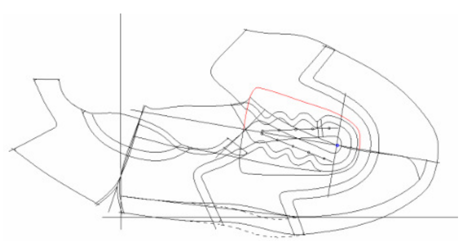


Fig. 14 – Mirror tongue lines.

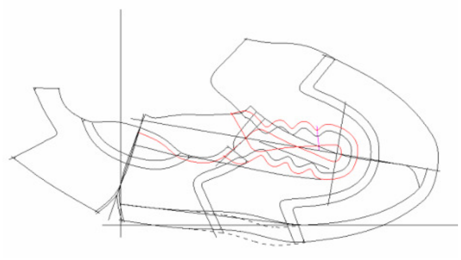


Fig. 15 – Line offsets.

– test each part for pattern efficiency;

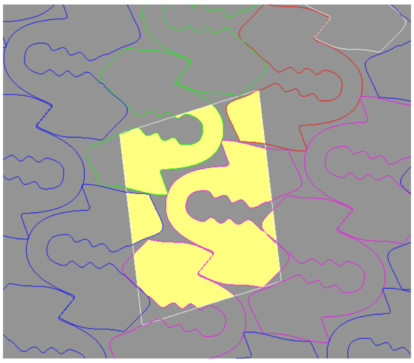


Fig. 16 – Vamp pattern assessment.

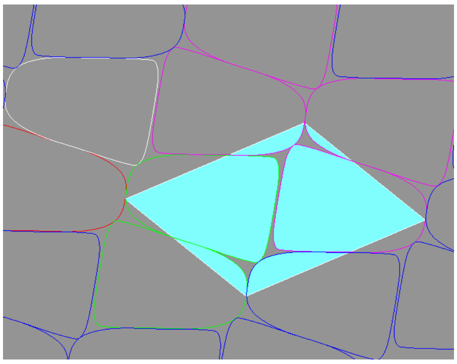


Fig. 17 – Tongue pattern assessment.

– grade to any size range and fitting, including group grading;

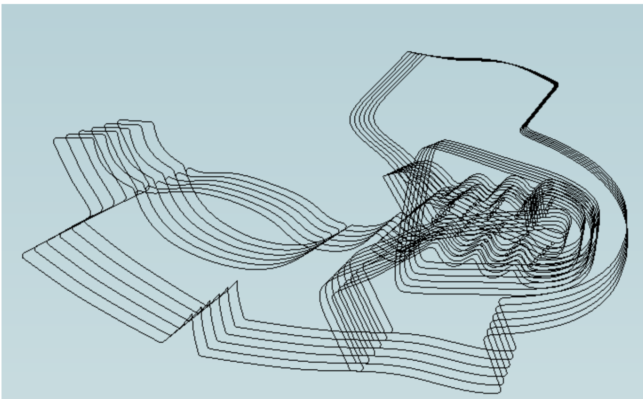


Fig. 18 – Pattern grading.

4. Conclusions

Starting with a last, it can be rapidly designed a complete footwear model, in any colour or texture combination. The result is a 3D realistic view of the product, ideal for presenting it to the customers, buyers or producers.

The advantage consists in:

− a rapidly way to design a model;
− reduces the product time to market;
− modifying operation directly on the model;
− applying or eliminating new components;
− model visualization from different angles by interactively rotating the last;
− line visualization on the last and also on the mean forme in the same time;
− reduces the number of physical samples;
− importing the flattening model in 2D module and pattern making;
− accurate flattening and fit and proper shape for pattern development;
− fully 2D grading in any type of measuring system (French, English, Metric);
− efficiency of pattern assessments.

Crispin Software provides the important tools in order to produce high quality footwear and offers a comprehensive, powerful, easy-to-use footwear design and manufacturing solution that requires minimal training. It's providing programs for the full lifecycle of a complete footwear product.

Acknowledgements. This paper was realised with the support of EURODOC "Doctoral Scholarships for research performance at European level" project, financed by the European Social Found and Romanian Government.

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TEHNICI MODERNE DE PROIECTARE A ÎNCĂLŢĂMINTEI PERSONALIZATE

(Rezumat)

În cadrul acestui articol se propune un pachet complet de aplicații pentru proiectarea încălțămintei personalizate, utilizând sistemul Delcam-Crispin. Sistemele revoluționare CAD/CAM reprezintă noua generație de soluții de proiectare şi inginerie asistată de calculator în industria încălțămintei. Realizate în mod exclusiv pentru a fi folosite în cele mai noi sisteme şi medii de operare, acestea furnizează întreaga gamă de utilități în pachete dedicate, care sunt printre cele mai intuitive şi uşor de folosit în comparație cu produsele existente în acest moment pe piața de profil. Tehnicile 3D de proiectare asistată de calculator (CAD 3D) oferă posibilitatea modelării încălțămintei direct pe calapod, astfel încât, chiar înainte de a fi confecționat, produsul poate fi analizat din prisma criteriilor estetice, funcționale, tehnologice şi economice. Pornind de la un calapod, se poate realiza rapid modelul unui produs de încălțăminte complet, în orice combinație de culori sau texturi. Rezultatul este o vizualizare 3D realistică a produsului, ideală pentru prezentări adresate cumpărătorilor şi producătorilor. Indiferent că sunt sisteme CAD 3D sau 2D, utilizatorul acestora trebuie să țină cont şi de criteriile ce decurg din funcțiile pe care trebuie să le îndeplinească încălțămintea.

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STATE OF THE ART AND NEW PERSPECTIVE ON BALISTIC VEST DESIGN

BY

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Abstract. A ballistic vest, bullet-proof vest or bullet-resistant vest is an item of personal armour that helps absorb the impact from firearm-fired projectiles and shrapnel from explosions, and is worn on the torso. Soft vests are made from many layers of woven or laminated fibres and can be capable of protecting the wearer from small-calibre handgun and shotgun projectiles, and small fragments from explosives such as hand grenades. The paper presents the state of the art in the field and some promising perspectives on ballistic vest design.

Key words: personal protection; ballistic vest; women body armour.

1. Introduction

A ballistic vest, bulletproof vest or bullet-resistant vest is an item of personal armour that helps absorb the impact from firearm-fired projectiles and shrapnel from explosions, and is worn on the torso. Soft vests are made from many layers of woven or laminated fibres and can be capable of protecting the wearer from small-calibre handgun and shotgun projectiles, and small fragments from explosives such as hand grenades.

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Metal or ceramic plates can be used with a soft vest, providing additional protection from rifle rounds, and metallic components or tightly woven fibre layers can give soft armour resistance to stab and slash attacks from knives and similar close-quarter weapons (New World Encyclopedia contributors, 2011). Soft vests are commonly worn by police forces, citizens, security guards, and bodyguards, whereas hard-plate reinforced vests are mainly worn by combat soldiers, police tactical units, and hostage rescue teams.

Modern body armour may combine a ballistic vest with other items of protective clothing, such as a combat helmet. Vests intended for police and military use may also include ballistic shoulder and side protection armour components, and bomb disposal officers wear heavy armour and helmets with face visors and spine protection.

The basic principle of a ballistic vest is the following: Ballistic vests use layers of very strong fibre to “catch” and deform a bullet, mushrooming it into a dish shape, and spreading its force over a larger portion of the vest fibre. The vest absorbs the energy from the deforming bullet, bringing it to a stop before it can completely penetrate the textile matrix (New World Encyclopedia contributors, 2011). Some layers may be penetrated but as the bullet deforms, the energy is absorbed by a larger and larger fibre area.

While a vest can prevent bullet penetration, the vest and wearer still absorbs the bullet's energy. Even without penetration, modern pistol bullets contain enough energy to cause blunt force trauma under the impact point. Vest specifications will typically include both penetration resistance requirements and limits on the amount of impact energy that is delivered to the body.

Vests designed for bullets offer little protection against blows from sharp implements, such as knives, arrows or ice picks, or from bullets manufactured of non-deformable materials, *i.e.*, those containing a steel core instead of lead. This is because the impact force of these objects stays concentrated in a relatively small area, allowing them to puncture the fibre layers of most bullet-resistant fabrics.

Textile vests may be augmented with metal (steel or titanium), ceramic or polyethylene plates that provide extra protection to vital areas. These hard armour plates have proven effective against all handgun bullets and a range of rifles. These upgraded ballistic vests have become standard in military use, as soft body armour vests are ineffective against military rifle rounds. Corrections officers and other law enforcement officers often wear vests which are designed specifically against bladed weapons and sharp objects. These vests may incorporate coated and laminated para-aramid textiles or metallic components.

2. Ballistic Vest Design

The design of the ballistic vest depends of many parameters:

Sizing - The standard sizes are XS to XXL. In order to give a range of sizes, some manufacturers have a size chart, where all the sizes correspondences appear. A new program called Pro-Fit is purposed only for Sigma Six and Delta Five vests. The aim is to have a better knowledge of the customer's sizes and needs in order to improve their vests (Global Armour, Body Armour Overview, 2011).

Body measurements - The ballistic vests are no longer unisex. Many companies offer female vests because women are often more fine (Global Armour, Measurements Guide, 2011):

− Chest circumference and torso length determine the size of the vest, sometimes also the waist.

− For men and for women, the asked measurements are not exactly the same. More details are required to make a female vest, mainly due to the chest complex shape. For women, the needs is : height, weight, cup size, bust, under bust, waist size, chest breadth, front length standing, front length sitting, back length standing, bust height standing, bust height sitting, bust width, bust coverage, side height standing. The needs for men are reduced to: height, weight, chest circumference, waist circumference, chest breadth, front length standing, front length sitting, back length standing.

Influence of parameters - Before designing personal protection it is important to consider 4 main factors: the price, the performance, the weight and the comfort.

− The Price depends on: the used materials (fabrics, UD, yarns), the protected areas, the numbers of layers, the position of panels during the cutting (to minimize the waste), the transformation, assembling and production costs.

− The Performance depends on: the material link and type (panels, stitching), the number of layers, the protected areas, the layers pilling up, the type, position and alternation of darts, the bonding of layers (stitching, quilting, sticking).

− The Weight depends on: the materials (density, weight), the number of layers, the protected areas, the bonding of layers (stitching, quilting, sticking).

− The Comfort: the total weight of the vest, the shape of the vest, the type and the position of darts, the protected areas, the number of layers.

3. Advance Mass Customization of Ballistic Wear

These days, we need mass customization of ballistic wear in order to meet the demand and specific requirements. As the term describes, mass customization means bulk production in an economic way. Advance mass customization of ballistic wear deals with specific software and techniques that make it more economic and feasible.

The entire process' flowchart is given in Fig. 1 (Cichocka *et al*., 2009):

The process of making ballistic protection vests for the women starts by the digitalization of the human body. This 3D measurement method shows various defects coming from the hidden regions or the measurement noise.

The 3D output variables of the scanner must necessarily be corrected and filtered to obtain a body without defect. Morphology can then be extracted to create a model of human body usable by CAD software (Boussu *et al*., 2008; Maillet *et al*., 2009).

After that, the pattern for the body is being created. In this case the 2D basic pattern for the women's vest associated with measurement has been taken into account to create the morphological customized model. On the 3D model is hung an ease model obtained from the difference between scanned nude and clothed body of the customer. The curves network of this ease model is the support to create the 3D surface of the basic model of the 3D waistcoat. Now we can design directly on this surface the lines defining the model of the front and back lady's vest. The vest patterns are defined by the surface 3D defined by these characteristic lines. These lines are also used for positioning and defining the lines defining the surface of the first protective layer. Pattern of this one is compared with 2D pattern layer 1 for ballistic soft panel. This test can adjust the position of these lines according to the protective region. Dark method is used to control the protection in this zone layer by layer.

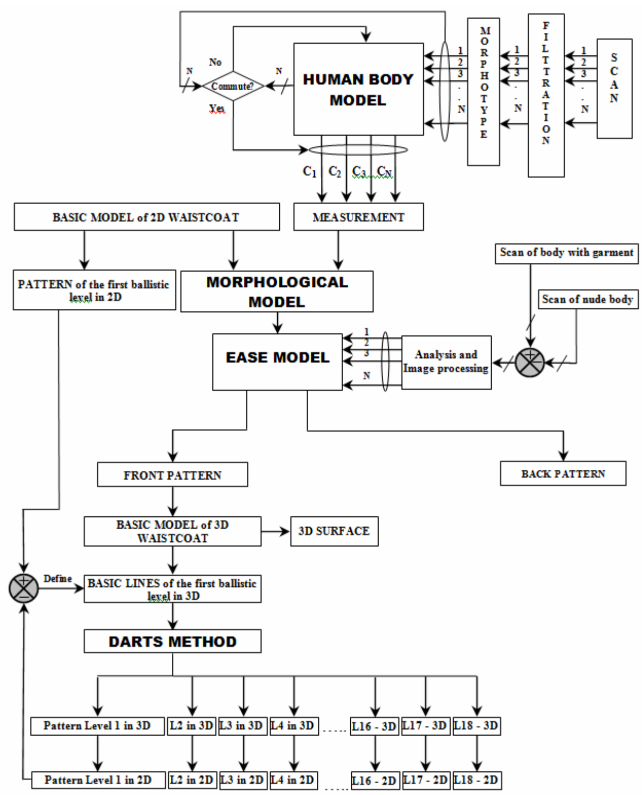

Fig. 1 − Process to design ballistic vest.

3.1. Body Measurement – Morphological Model

In order to create the pattern of the basic vest that will define the support of the future ballistic panel and ballistic vest, we need the correct measurements of the human body. It's possible to take the measures using 2 methods: Numeric method – body scan, Traditional method – tape of measurement.

If we compare these two different methods we can easily find that the body scan represents a better solution because this method eliminates human mistakes. We use the scanner tool to ensure to exact measurement of human body.

We need the following measurements to design the lady's vest with the 2D method (Cichocka *et al*., 2007a):

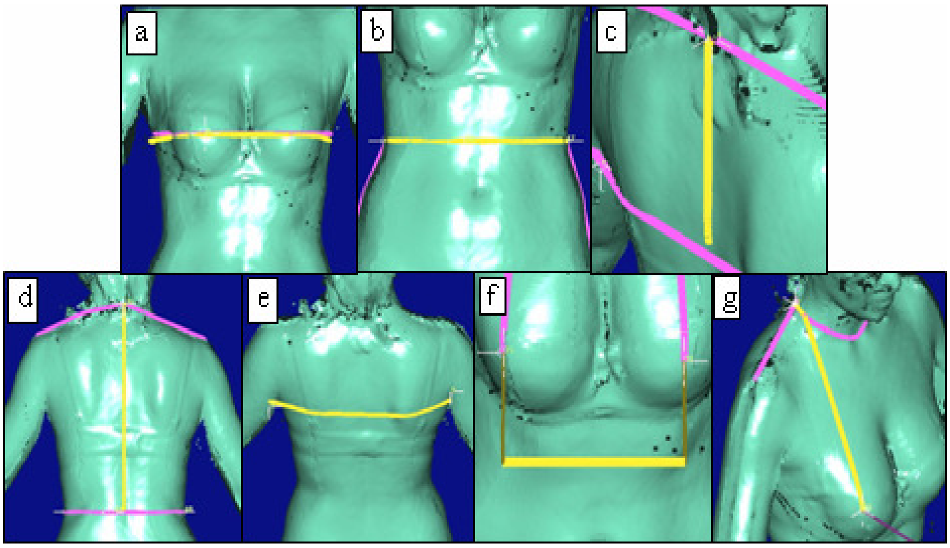

Fig. 2 − Main measurements to define the lady's vest: *a* − ½ bust/chest girth, *b* − ½ waist girth, *c* − neck to across back width, *d* − neck to waist centre back, *e* − ½ across back width, *f* − ½ bust point's width, *g* − bust point to neck.

3.2. Ease Model

The 3D design of the Basic pattern directly uses the scanned 3D model (Maillet *et al*., 2009). On this one, we place and create a morphological model by the extraction of various and strategic morphological contours Fig. 3 *a*).

A 3D ease model is created by choosing the check-points of ease on the contours of morphological model (Fig. 3 *b*). These points are located symmetrically compared to the front and back medium lines, and then shifted in the plan of contours of an ease value fitting to the person.

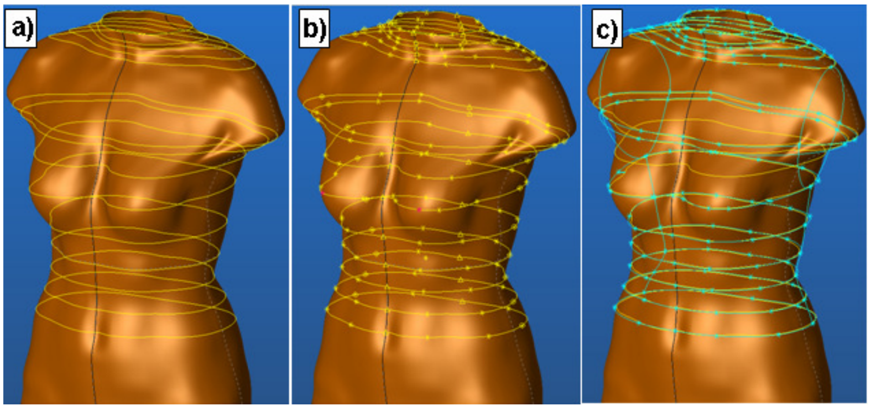

Fig. 3 – Design the morphological model and customisation.

From these space points, we create new contours on which are added the complementary construction lines defined on 2D pattern (Fig. 3 *c*). A surface model is based on this curves network to define the remains including the morphological model 4 *a*). The surface of the ease model can be created and used for define the protective limit. The surface of the ease model can be created and used for define the protective limit (Fig. 4 *b*).

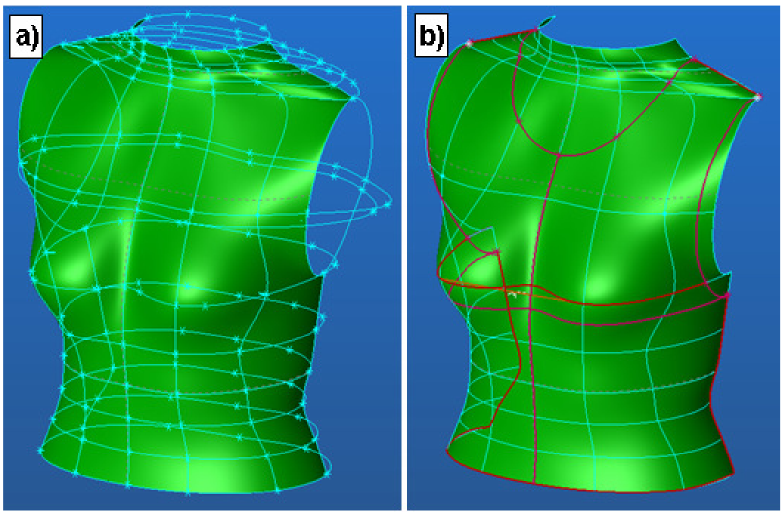

Fig. 4 – Ease model surface and protective limit.

3.3. 3D/2D Pattern Design

In the 3D concept, we follow the same method as 2D pattern making. The difference compared to the 2D method is that the starting points of the various lines limiting pattern are fixed on the lines of the basic pattern for a lady's vest to preserve an associative and parametric link with this one (Figs. 5 *a* and *b*). This link will be used later on to have an adaptive model of the lightweight ballistic vest. Basically the dart method is used to create patterns in this process (Figs. 5 *c* and *d*). Fig. 6 shows the gap on the layer 1 between the traditional method (symmetrical method) and the mass customization method (Cichocka *et al*., 2007b).

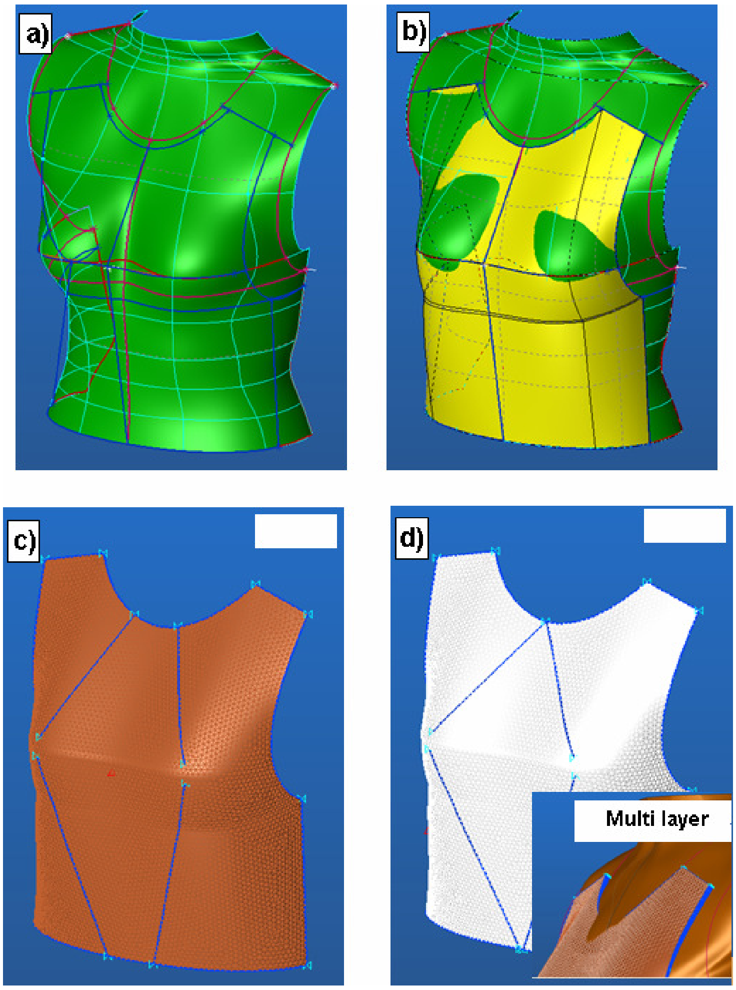

Fig. 5 – 3D design ballistic soft panel - Control the dart positions.

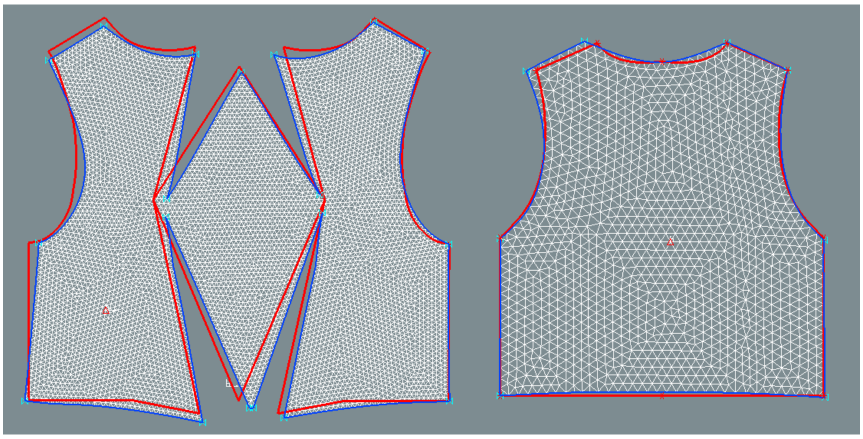

Fig. 6 − Difference between standard method and mass customization method (layer 1).

4. Conclusions

These days advanced mass customization of ballistic vest is important to meet the demands and specific requirements of different body sizes and shapes so that the body can be protected. Various software programs that help in scanning the body and making a garment of the perfect size for the customer thereby giving maximum protection have been developed.

Fashionization of ballistic wear is another important step in this field. Designer clothes are sewn with secret bullet proof lining inside of them. These clothes are perfect for casual wear and offer full protection against all rounds up.

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REALIZĂRI ACTUALE ŞI NOI PERSPECTIVE ÎN DOMENIUL PROIECTĂRII VESTELOR ANTIGLONȚ

(Rezumat)

Vesta antiglonț reprezintă una din piesele echipamentului de protecție personală care se poartă pe tors şi contribuie la absorbția impactului generat de proiectile sau schije. Vestele uşoare de protecție antiglonț sunt realizate din mai multe straturi de țesătură sau materiale laminate care conțin fibre şi pot proteja purtătorul la impactul cu gloanțe de calibru mic sau schije rezultate din explozia grenadelor de mână. Lucrarea prezintă realizări actuale şi perspective în domeniul proiectării acestui tip de veste antiglonț.

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FERRIMAGNETIC SUBSTANCES USED FOR PRODUCING MAGNETISABLE TEXTILE PRODUCTS (COMPOSITE YARNS)

BY

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Abstract. In this article are presented possibilities of producing of composite magnetisable textile products from textile yarns, ferromagnetic powders and binding materials. There are presented aspects regarding the characteristics of ferromagnetic substances used in mixture with binding materials, for coating textile products (composite yarns) and the textile products used (matrix carrier yarns). The ferromagnetic products are tested using a Vibrating Sample Magnetometer, and the textile products used as support materials are analyzed from the physical, mechanical and structural point of view.

Key words: composite magnetisable yarns, barium ferrite, magnetisable mixture, VSM.

1. Introduction

The connection between science and technology leads to obtaining and diversify of materials with specific properties. Applications of the magnetism in textile technology area contribute to the development of the specific products with special technical applications as textronics (electronics textiles informatics technologies), biomedical, industrial, aero-spatial, chemical, electronic,

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electromagnetic shielding fields (Rubacha & Zieba, 2006). In the terms of actual industrial development was established a request for obtaining of the magnetic textile elements as fibres/filaments, knit or woven fabrics.

The magnetic textile fibres/filaments are obtained by various physical and mechanical processes. Magnetic cellulose fibres can be prepared either by 'lumen loading' or by in situ synthesis of ferrites. By using the lumen-loading technology, commercially available magnetic pigments can be introduced into the lumens of softwood fibres. Lumen-loaded fibres act as magnetic dipoles allowing manipulation of fibre orientation. In situ synthesis of iron oxide particles is performed through oxidation of ferrous hydroxide precipitated with caustic from the ferrous ion-exchanged form of sodium carboxy-methyl cellulose fibres. The fibres are characterized by conductometric titration to determine the number of functional groups available for the in situ chemistry. Superparamagnetically responsive fibres have smaller and less coloured pigments which are only magnetic in the presence of a field (Rubacha & Zieba, 2007; Marchessault & Rioux, 1992).

Another technology for obtaining of the magnetic filaments includes theirs implementation by an aqueous spinning solution. The researches accomplished by Zięba and others, have led to obtaining of magnetic cellulosic filaments of N-oxide-N-methylmorpholine (NMMO) concentrated solutions. Using Lyocell process (Green & Ormeaux, 1985) in the spinning solution was introduced a mixture of soft magnetic powders (Fe-MB Nano crystalline alloys (M = Nb, Cu, Hf, Zr, Si) or carbonyl iron powder (CIP)) and hard magnetic powders are barium ferrite ($BaFe_{12}O_{19}$). Thus the composite is obtained by cellulose filaments that represent the matrix and the charging degree provided by magnetic powder used. The mass percentage of magnetic powder introduced in the spinning solution does not exceed 50%. Above this limit tensile strength and filament tenacity obtained decreases a lot. Magnetic filaments obtained are ordered in the form of bunch with a linear arrangement, respecting the law of magnetic induction (Mefford & Brown, 2010).

A special case of magnetic fibres, the $Ba_xSr_{1-x}Fe_{12}O_{19}$ (x = 0–1) ferrite hollow fibre made of the gel-precursor transformation process were developed by Materials used for preparation are $Ba(NO_3)_2$, $Sr(NO_3)_2$, $Fe(NO_3)_3$•$9H_2O$ and citric acid (Song *et al.*, 2010).

The Textiles magnetic yarns represent another special case of textile composite presented by Brauer *et al.*, which are composed of yarns (the base) and amorphous yarns (Brauer, 2000). The detectable magnetic markers that was obtained possess the purpose of supervision and antitheft. Rodgers and others in the patent have advanced the idea of using yarn coated with magnetic material to produce fabric for therapeutic purposes (Rodgers, 2003). The obtaining of the magnetisable composite yarns involves using of some coating recipes of the yarns viewed as carrier matrix. In this article are presented possibilities of producing of composite magnetisable textile products from textile yarns, ferromagnetic powders and binding materials. There are presented aspects regarding the characteristics of ferromagnetic substances used in mixture with binding materials, for coating textile products and the textile products used.

2. Experimental Part

In this part are presented the characteristics of the main elements that go into the composition of the magnetic composite yarns. These are matrix carrier yarns and magnetisable composite mixtures consisting of barium ferrite, polyvinyl acetate and glycerine in different percentage ratio by mass/volume.

To obtain magnetisable composite yarn they were chosen two yarns, considered as diamagnetic matrix carrier, with different finesses and structures, to highlight the differences that occur during and after the deposit magnetisable mixtures:

− Yarn (A) Nm 85/3, 100% cotton, made by worsted technology, for use as sewing thread.

− Yarn (B) Nm 25/3 cotton outer layer (30%) and PES core (70%) used as sewing thread for leather and substitutes (Avram *et al*., 2002)

These yarns have into own structure three simple yarns because it offers a section closer to circle and ensure more uniform distribution of magnetisable mixture around the yarn.

The ferromagnetic powders presented in the paper are: *isotropic barium ferrite* (FB 1). Hexagonal Barium ferrite was purchased from "ROFEP" Urziceni, România having the chemical formula ($BaFe_{12}O_{19}$) is a hard magnetic material from ferromagnetic class materials made by iron oxide (Fe_2O_3) and barium carbonate ($BaCO_3$). The barium ferrite powder having a magnetoplumbite structure is known as ceramic permanent magnets. They are widely used in magnetic recording and microwave devices owing to their high maximum saturation magnetization, appropriate Curie temperature, high theoretical maximum coercively, high magneto-crystalline anisotropy and excellent chemical stability. However, these hexagonal ferrites shown in Fig. 2 are quite heavy as into the bulk ceramic materials that limit their applications in many high-tech fields (Dinzard & Sabara, 2011).

Ferrite magnets are still widely used although they have less magnetic strength than rare earth magnets (SmCo, NdFeB). Comparing ferrite magnets and rare earth magnets could be concluded by determining the ratio of residual (Br), which is about 1:3, the ratio of coercive force (Hc), which is also 1:3 and the ratio of the maximum energy product (BHmax), which is about 1:10. Many methods of synthesis have been developed to obtain a low production cost of powder particles of barium ferrite. The rare earth magnets are used where weight and size are very important from the cost and performance point of view (Nowosielski *et al*., 2007). The main characteristics of Isotropic barium ferrite FB 1 are presented in the Table 1 (Catalog B.L.C.)

Table 1
The Characteristics of Barium Ferrite 1

Characteristics	Measure unit	Isotope barium ferrite (FB 1)
Remanent induction (B_r)	Tesla	0.2
	Gauss	>2000
Coercitive field (H_c)	10^4Amper/meter	131369.4
	Oersted	>1650
Maximum magnetic field (H max)	10^4Amper/meter	80
Curie temperature (Tc)	ºC	450

The binder used, in mix with FB1 ferromagnetic powder, is *polyvinyl acetate*. This is vinyl resin and is one of the clear, water-white, thermoplastic synthetic resins produced from its monomer by emulsion polymerization. Polyvinyl acetate, abbreviated as PVA has the advantage over the other resinous adhesives in that it is available in the form of an emulsion that is readily diluted with water, is easily applied, and is safe to use because it contains no flammable solvents (Carmines & Gaworski, 2005).

Glycerine was used as plasticizer for obtaining mix recipes. Glycerine is a trihydric alcohol. There are four recognized names for glycerine, the other three being glycerol, 1, 2, 3 - propanetriol and trihydroxypropane. The two most common names are glycerol and glycerine. Glycerine is widely used by the food, cosmetic and pharmaceutical industries, because it can serve many functions such as a humectant (moisture absorbing), plasticizer (softening), bodying agent, flavouring, denaturant, emollient (smoothing), antimicrobial, thickener and solvent (Toxqui *et al*., 2006).

The magnetisable composite yarns are made by coating the carrier matrix yarns with magnetisable mixtures. The coating is considered as a thin layer with properties different from those of the inclusion and the matrix (Grosu *et al*., 2011a). By aggregation of the three components, magnetisable mixture obtained is regarded as a composite in terms of physical characteristics of different components.

Coating yarns is made using a special installation created for this purpose. The installation, according to Fig. 1, allows depositing of the thin layer of magnetisable mixture previous realized on the matrix surface (the yarn) in a magnetic field of orientation of the components magnetic particles. Coating is realized into a special depositing room of the magnetisable mixture, then the composite yarn leaves the depositing room through a calibration system of a layer thickness deposited and comes to be dry and set, optimal for winding (Grosu *et al*., 2011b). In Fig. 4 are presented SEM images of composite yarns A1 and B1. These images were accomplished with a VEGA II-LHM device with BRUKER detector at Faculty of Materials Science and Engineering from Iaşi.

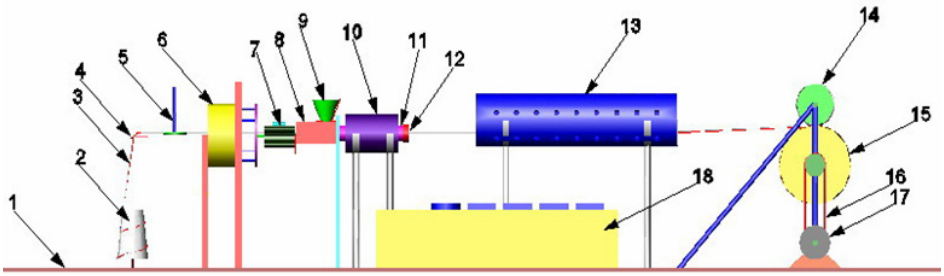

Fig. 1 − Experimental laboratory installation for coating of textile yarns with magnetic composite: *1* − framework support, *2* − bobbin at feed, *3* − yarn, *4* − leader yarn, *5* − adjustable tensioning device yarn, *6* − electrical engine, *7* − gear train, *8* − magnetisable mixture feed room, *9* − magnetisable mixture feed hopper, *10* − electromagnet, *11* − magnetisable mixture room deposit on the yarn, *12* − spinneret, *13* − drying and fixation room of the composite yarn, *14* − winding reel of the composite yarn, *15* − grooving drum, *16* − transmission belt, *17* − electric drive grooving drum, *18* − electrical control block.

3. Results and Discussions

The structure of magnetisable mixture with components of barium ferrite with particle size around 1-2 µm as shown in Fig. 2, polyvinyl acetate and glycerine forming a thin film of about 3-4 µm with glossy surface on these carrier matrix surface A and B. Magnetisable mixture obtained from the three components is heterogeneous and can show areas with clusters of ferromagnetic particles in binder (Fig. 3).

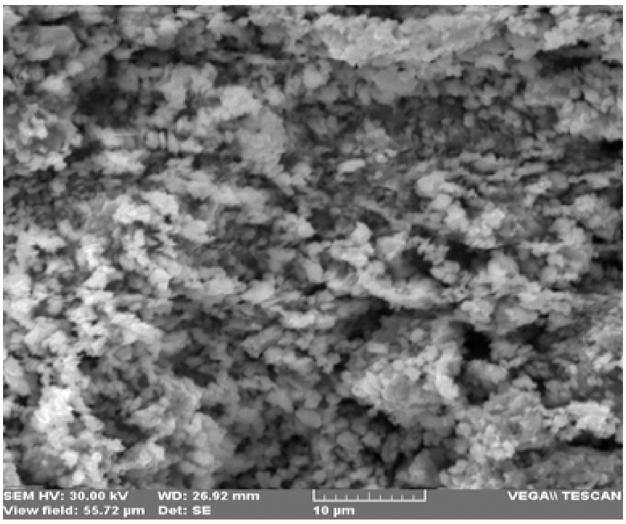

Fig. 2 − SEM image of hexagonal Isotropic barium ferrite (FB 1).

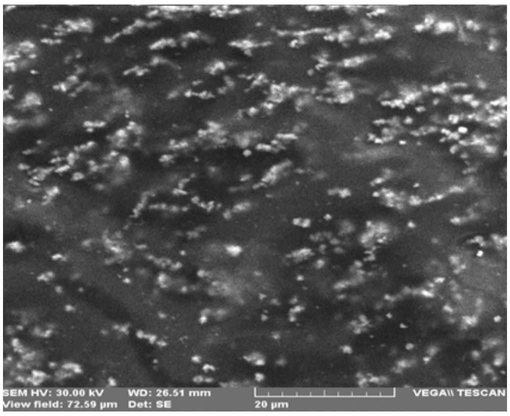

Fig. 3 − SEM image of magnetisable composite mixture with 45% wt of FB 1.

In Fig. 4 is seen that composite yarns A1 and B1 also presents a high degree of unevenness. This is due to both differences of the diameter of each carrier matrix structure the content of free fibres of yarns structure, also the magnetisable mixture in homogeneity.

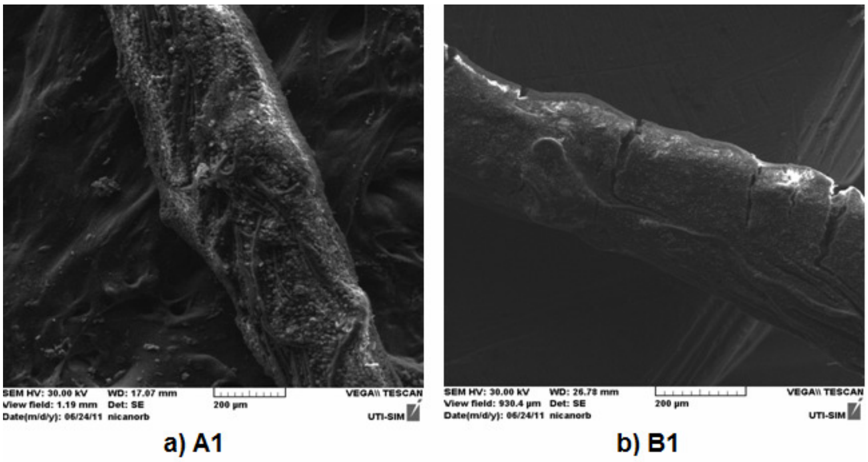

Fig. 4 − SEM images of magnetisable composite yarns.

The main properties of the deposited magnetisable mixture layer are besides providing of the magnetic characteristics of the composite yarns ensuring the mechanical protection of it. The magnetisable composite yarn obtained using the installation shown in Fig. 1 presents magnetic properties in the presence or not of an external magnetic field. They are marked using a VSM installation, the result being the curves of magnetic hysteresis Figs. 5,…,7. So were analyzed *versus* the value of saturation magnetization (M_s), residual magnetization (M_r) and coercive field (H_c), respectively applied maximum field

(H) plotting hysteresis curves for isotropic barium ferrite (FB 1), for obtained magnetisable mixture and for realized magnetisable composite yarns.

It is observed from the analysis results in according to the Table 2 that the M_s and M_r sizes has decreasing values in the case of magnetisable mixture R, respective of composite yarns A1 and B1 due to the higher volume of diamagnetic inclusions (binder, plasticizer, fibres).

Table 2
The Magnetic Parameters

Materials	Magnetic parameters	
	M_s [emu/g]	M_r [emu/g]
FB1	49.59	31
R	40.61	23.82
A1	25.7	14.82
B1	10.85	6.18

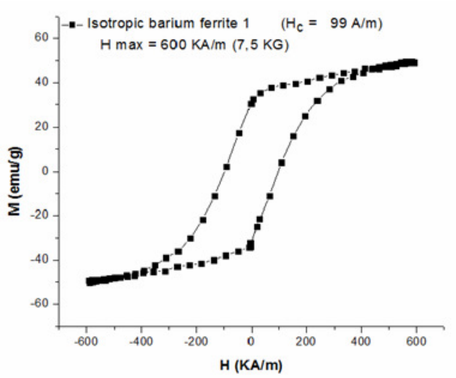

Fig. 5 − Hysteresis curve of Isotropic barium ferrite.

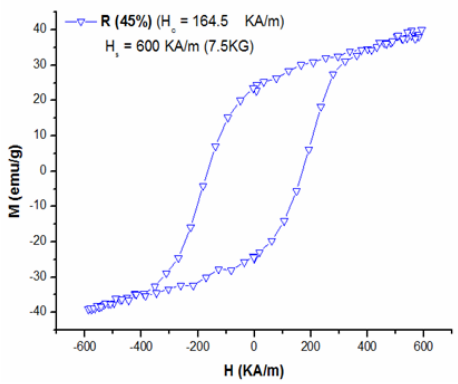

Fig. 6 − Hysteresis curve of magnetisable mixture recipe.

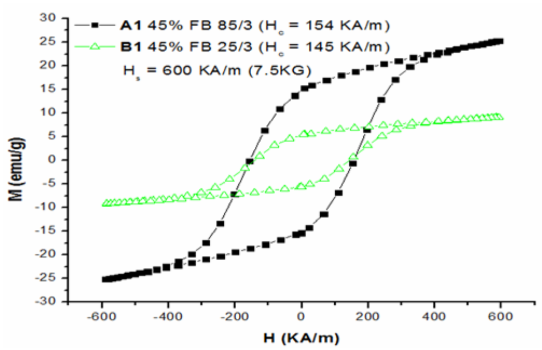

Fig. 7 − Hysteresis curve of composite yarns A1 and B1.

The measurements were realized at The National Institute of Research and Development for Technical Physics in Iaşi. The composite yarns A1 and B1 present different values of charging degrees with magnetisable mixture, due of the different fineness's of carrier yarns. Thus the composite yarn A1 presents a charging degree of 75% by his own mass and the yarn B1 has charging degree just around 34%. It was mentioned that both composite yarns were made in the same conditions keeping constant the calibration system during their coating. The physical and mechanical properties of the composite yarns were presented comparing with those of support yarns matrix. Thus in the Table 3 is presented breaking strength, elongation, with average values, standard variation and coefficients of variation, tenacity, slope and mechanical.

In the Table 3 is observed that between the initial yarn A and the composite yarn A1 differences from statistically point of view are insignificant because the value $t_{cal} < t_{tab}$ for a certain statistically of 90% and the number of values is 5, where t is the Student test. The difference is significant between the initial yarn B and the composite yarn B1 because the t value is 2.13 high than 2.015 reference value. Regarding the elongation at break, differences between the initial yarn and the composite yarns are insignificant from statistically point of view. The difference between the yarn A and the yarn A1 is much higher than for the initial yarn B from B1 from the tenacity point of view. It can be appreciated regarding the B1 composite yarn charging degree with magnetisable mixture being smaller has a reduced influence to tenacity from the initial yarn B.

Depending on the slope is noticed an increase of it in instance of the composite yarn A1 the yarn A because of a charging degree higher of the A1 yarn that makes the rigidity to sag of the yarn to be higher.

Table 3

The Mechanical Characteristics of Yarns A, A1, B, B1

Physical and mechanical characteristics		Yarn type			
		A	A1	B	B1
Breaking strength	Average, [N]	9.34	8.64	47.4	49.11
	Standard deviation	0.46	0.96	1.80	0.07
	Coefficient of variation	4.96	11.12	3.81	0.15
Elongation at break	Average, [%]	4.16	3.68	11.29	11.43
	Standard deviation	0.21	0.44	0.90	0.88
	Coefficient of variation	5.28	12.12	8.04	7.75
Specific resistance (Tenacity), [N/tex]		0.26	0.06	0.38	0.26
Elongation at break, [mm]		10.41	9.19	28.29	28.74
Slope, [N/mm]		0.89	0.93	1.67	1.71
Mechanic Work, [J]		0.048	0.041	0.676	0.753

Thus the magnetically mixture from the composite yarn structure does not influence significantly but it is identified an increase of rigidity to sag. Also by increasing the content of ferrite into the coating influence the yarn rigidity by slope stiffness wire strain effort. On the B1 yarn is noticed an increase of slope towards the initial yarn B, which highlights the increase of rigidity to sag. Regarding the mechanic (L) are not presented influences too higher to the deposition layers on the A1 and B1composite yarns where differences exceed to 10%.

4. Conclusions

Obtaining of the magnetisable composite yarns involves using of some coating compositions of the yarns viewed as carrier matrix. In this article are presented possibilities of producing of composite magnetisable textile products from textile yarns, ferromagnetic powder (isotropic barium ferrite 1) binding material (polyvinyl acetate) and plasticizer (glycerine). The yarns used as carrier matrix are: yarn (A) Nm 85/3,100% cotton, made by worsted technology, for use as sewing thread and yarn (B) Nm 25/3 cotton external layer (30%) and PES core (70%) used as sewing thread for leather and substitutes.

The coating process is achieved using a special installation that allows depositing a thin layer on the matrix surface through the calibration elements.

Both composite yarns are obtained together with the magnetic powder used and magnetisable mixture was tested using a VSM device being plotting magnetic hysteresis curves for each one.

The values of magnetic parameters decrease begin from the magnetic powder, continuing with magnetisable mixture and it is observed that B1 values are less then A1 values. That is due to charging degree of the yarn that is less than charging degree of the A1 composite yarn that is around 50%.

The future directions of the research aim to obtain the composite woven fabric by coating with magnetic layer and producing of the woven fabric from composite yarns and test own behaviours.

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SUBSTANȚE FERIMAGNETICE UTILIZATE PENTRU REALIZAREA PRODUSELOR TEXTILE MAGNETIZABILE (FIRE TEXTILE COMPOZITE)

(Rezumat)

În acest articol sunt prezentate posibilităţile de realizare ale produselor textile magnetizabile din fire textile, pulberi ferimagnetice, materiale de liere şi plastifianţi. Sunt prezentate aspecte privind caracteristicile substanţelor ferimagnetice utilizate în amestec cu materialele de liere pentru acoperirea elementelor textile şi ale elementelor textile utilizate. Produsele ferimagnetice sunt testate utilizând un dispozitiv numit magnetometru cu proba vibrantă (VSM), iar produsele textile utilizate ca matrice suport sunt analizate din punctul de vedere al caracteristicilor fizice, mecanice şi structurale.

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STUDY CONCERNING THE SYNERGETIC EFFECT OF SYNTHESIS AGENTS ONTO THE SURFACE CRYSTALLINITY OF SOME FIBROUS SUBSTRATES COATED WITH NANOOXIDES, WITH DIRECT IMPLICATION ONTO THERMAL PROTECTION

BY

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Abstract. The research is first aiming at a novel preparing technique in order to obtain nanoscale ZnO coated/covered fibrous composites, by using the cumulative/synergic effect of MMT (montomorilonite) and MCT-β-CD (monochlorotriazinyl–β-cyclodextrin) that interfered in the preparation technique. Secondly, the study was focused towards the imparting of thermal protection to the fibrous nanocomposites.

To characterize the probes composition, shape, size and crystallinity, investigations technique, such as: Fourier transformed infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and X-ray powder diffractometry (XRD) were used.

Key words: crystallinity, nano-oxides, thermal protection, coating, fibrous substrates, hydrothermal synthesis.

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1. Introduction

Exposure to various factors such as heat, UV light, irradiation ozone, mechanical stress and microbes generates the degradation of polymeric materials. This process is promoted by oxygen, humidity and strain, and results in such flaws as brittleness, cracking, and fading (Corrales *et al*., 2002; Verdu *et al*., 2003;, Allen *et al*., 1998; Perera, 2003).

ZnO particles have many applications, such as: varistors and other functional devices, reinforcement phase, wear resistant phase and anti-sliding phase in composites in consequence of their high elastic modulus and strength. Otherwise, ZnO particles exist in anti-electrostatic or conductive phase due to their current characteristics (Singhal *et al.*, 1997; Hingorani *et al*., 1993). Few studies have been concerned with the application of ZnO nanoparticles in coatings system with multi-properties. The nanocomposite system coatings can be obtained by the traditional coatings technology, *i.e.*, by filling with nanometre-scale materials.

In terms of their structure and functional properties, coatings can be modified by filling with nano-materials. Super-hardness, wear resistant, heat resistance, corrosion resistance, and about function, anti-electrostatic, antibacterial, anti-UV and infrared radiation all or several of them can be realized (Wu *et al*., 2000; Chen *et al*., 2003; Xu & Xie, 2003; Patscheider *et al*., 2001).

The objectives of this work were to investigate the synergetic effect of both functionalization agent - MCT- β -CD (MONOCHLOROTRIAZINYL– β - CYCLODEXTRIN) and MMT (montmorillonite) onto the thermal stability of ZnO nanocoated linen fibrous samples. In order to improve surface thermal barrier, MMT emulsion was applied onto the supports, prior to the ZnO coating.

Montmorillonite (MMT) is a type of inorganic natural clay which has silicate (SiO_4) tetrahedral sheets arranged into a two-dimensional network structure (Fan *et al*., 2003). With its ordered structure (nano-scale pattern) montmorillonite (MMT) can provide thermal resistance, wrinkle resistance and antibacterial properties on textiles (Song *et al*., 2003; Mani *et al*., 2003; White & Bertoniere, 2002; Stretz *et al*., 2001; Ogata *et al*., 1995; Jiang *et al*., 2007).

The instrumental methods were conducted to measure the particle sizes of the reduced zinc oxide particles in order to characterize the surface morphology and chemical composition of the treated supports.

2. Materials and Methods

2.1. Materials

100% linen supports, each of size 3 cm × 3 cm, previously desized, scoured and bleached have been used for this study. One of the supports has been functionalized with a certain concentration of MCT-β-CD (monochlorotriazinyl– β -cyclodextrin).

2.1.1. *Determination of the Concentration of Dispersing Agent for Preparing the MMT Emulsion*

Bentonite montmorillonitic-clay (MMT), was provided by firma Riedel-de Haen Chemicals Company. Given the compositional complexity of clay materials, we considered useful as a first step to perform an ion exchange process, for their cleansing, in order to reach the transition cations sodium form. The clay exchange in Na+ form was conducted using a 1M NaCl solution with a ratio a solid/liquid 1:10.

2.1.2. *Preparation of Composites*

To make composite samples, the particle sizes of the MMT were reduced by an ultrasonic crashing machine. In order to prepare a good MMT emulsion such that the tiny particles of MMT should be well dispersed and will not be aggregated again to form large molecules, a non-ionic dispersing agent, SETAVIN from CLARIANT, were used for dispersing the MMT clay to form an emulsion. The emulsion was prepared by adding 1.0 g of MMT into 42 mL of dispersing agent. The emulsion was magnetically stirred for 2 h. Then the emulsion was crashed by an ultrasonic crashing machine for 30 min, to reduce the particle sizes.

In addition, a binder resin (from ARALDITE Company) was also prepared. The thus obtained resin solution was carefully poured drop wise. The mixture was then slowly stirred until homogeneous. Subsequently, ZnO nanoparticles emulsion with concentration of 0.5% was added. The resulting composite was allowed to stand overnight to remove air bubbles, before use. The synthesis of this oxide by hydrothermal method has been described elsewhere.

After preparing the MMT-ZnO emulsion, it was padded onto the four different linen fibrous supports:

1 – Reference linen support (without MCT-β-CD (MONOCHLOROTRIAZINYL-β-CYCLODEXTRIN)) and ZnO;

2 – Linen support with MCT-β-CD (MONOCHLOROTRIAZINYL-β-CYCLODEXTRIN) and without ZnO;

3 – Non-functionalized linen support without MCT-β-CD (MONOCHLOROTRIAZINYL-β-CYCLODEXTRIN) and with ZnO;

4 – Functionalized linen support with MCT-β-CD (MONOCHLOROTRIAZINYL-β-CYCLODEXTRIN) and ZnO, using a padding machine.

Due to the poor dispersing properties of the MMT particles, this padding process should be carried out as soon as possible, *i.e.* not longer than 30 min after finishing the crashing process, in order to prevent the reduced particles from aggregating again to form larger molecules. The emulsion was padded onto the linen supports by the padder with the pressure of 3 kg/cm^2 and the speed of 5 rpm. Two padding times were investigated including 3 padding times and 6 padding times, and subsequently the wet pick-up was 84.21% and

88% respectively. Finally, the treated fabrics were dried at 140°C, for 3 min.

Washing was carried out to remove the by-products. The treated supports were immersed for 5 min in 0.2 g/L sodium lauryl sulphate dodecyl, to remove the unbound nanoparticles. Then the fibrous supports were rinsed at least 10 times to completely take out all the soap solution. The samples thus washed were air-dried. Simultaneously, a reference emulsion made by MMT without ZnO was applied for comparison.

Thermal Treatment relied into two main stages, into the calcinations oven. Firstly, the samples were subjected to an increasing of temperature up to 150°C; secondly the probes were heated up to 350°C, 450°C, respectively.

2.2. Instrumental Methods

The instrumental methods were used to measure the size of ZnO particles to quantify the surface morphology as well as to follow the chemical modification of the treated supports.

X-ray diffraction (XRD)

Diffractograms were recorded using a PW1710 diffractometer using Cu-*K*α radiation (k = 1.54 Å) source (applied voltage 40 kV, current 40 mA). Scattered radiation was detected in the 2θ = 10–80° range at a speed of 1.5° min^{-1}.

Evaluation of crystallinity

The extent of crystallinity (I_C) was estimated by means of Eq. (1), where I_{020} is the intensity of the 020 diffraction peak at 2θ angle close to 22.6°, representing the crystalline region of the material, and I_{am} is the the minimum between 200 and 110 peaks at 2θ angle close to 18º, representing the amorphous region of the material in cellulose fibres (Mwalkambo & Ansell, 2002; Ye & Farriol, 2005; Scherrer, 1939). I_{020} represents both crystalline and amorphous materials while I_{am} represents the amorphous material.

$$I_C = \frac{I_{020} - I_{am}}{I_{020}} \text{x}100, \ [\%] \tag{1}$$

A *shape factor* is used in x-ray diffraction to correlate the size of sub-micrometer particles, or crystallites, in a solid to the broadening of a peak in a diffraction pattern. In the Scherrer Eq. (2), τ is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the grain size. The dimensionless shape factor has a typical value of about 0.9, but varies with the actual shape of the crystallite. The Scherrer equation is limited to nano-scale particles:

$$\tau = \frac{K \cdot \lambda}{\beta \cos\theta} \tag{2}$$

where: K is the shape factor, λ − the x-ray wavelength, β − the line broadening at half the maximum intensity (FWHM) in radians, θ − the Bragg angle (Tserki *et al.*, 2005).

3. Results and Discussions

Due to unknown causes, the dispersion of the MMT particles was not made in a proper manner onto all four samples we proposed for study; consequently we were forced to consider for investigation only the last two samples:

• 3 – Non-functionalized linen support without MCT-β-CD (MONOCHLOROTRIAZINYL- β-CYCLODEXTRIN) and with ZnO;

• 4 – Functionalized linen support with MCT-β-CD (MONOCHLOROTRIAZINYL-β-CYCLODEXTRIN) and ZnO

3.1. EDX Investigation

The results provided by SEM investigation have been reported elsewhere. Concluding, the difference between the morphology of the two studied samples is the following. The increasing of the temperature induced the increasing of the zinc oxide nanoparticles sizes on the functionalized linen sample. On the contrary, the non-functionalized sample, the dimensions of the nanoparticles decreased. *EDX - elemental analysis* is shown in Figs. 1,…,6 and Tables 1,…,6, indicating that ZnO nanocomposites contain different percentage content of zinc oxide.

For sample 3:

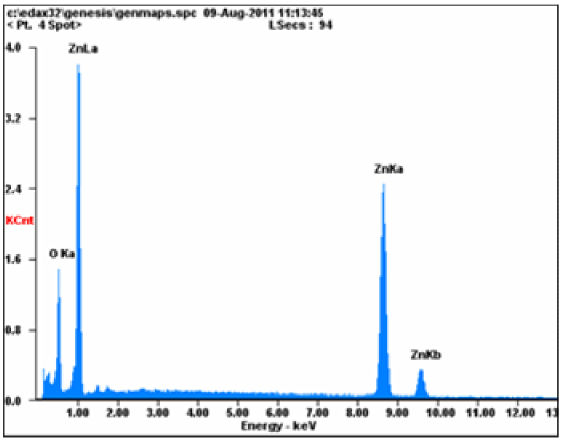


Fig. 1 − EDX analysis for 3–Non-functionalized linen support (without MCT-β-CD (MONOCHLOROTRIAZINYL-β-CYCLODEXTRIN)) with ZnO thermally non-treated.

Table 1
Surface Composition from EDX Measurements (*Wt: Weight Percent, At: Atomic Percent*)

Element	Wt [%]	At [%]
OK	28.91	62.43
ZnK	71.09	37.57

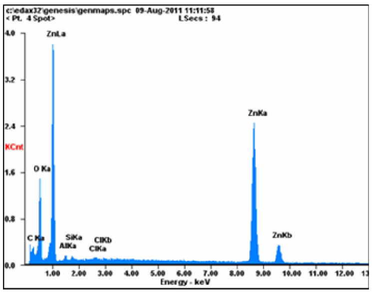


Fig. 2 – EDX analysis for 3-Non-functionalized linen support (without MCT-β -CD (MONOCHLOROTRIAZINYL-β-CYCLODEXTRIN)) with ZnO treated at 150ºC.

Table 2

Surface Composition from EDX Measurements (Wt: Weight Percent, At: Atomic Percent)

Element	Wt [%]	At [%]
CK	17.14	35.61
OK	27.40	42.74
SiK	00.77	00.68
ClK	00.26	00.18
ZnK	54.43	20.78

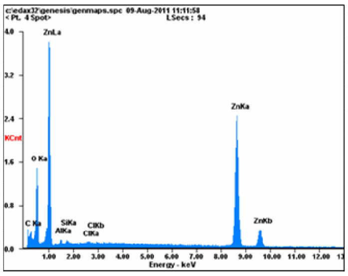


Fig. 3 – EDX analysis for 3-Non-functionalized linen support (without MCT-β-CD (MONOCHLOROTRIAZINYL-β-CYCLODEXTRIN)) with ZnO treated at 350ºC.

Table 3

Surface Composition from EDX Measurements (Wt: Weight Percent, At: Atomic Percent)

Element	Wt [%]	At [%]
CK	17.00	35.40
OK	27.08	42.18
AlK	01.20	01.11
SiK	00.76	00.67
ClK	00.26	00.18
ZnK	53.64	20.45

For sample 4:

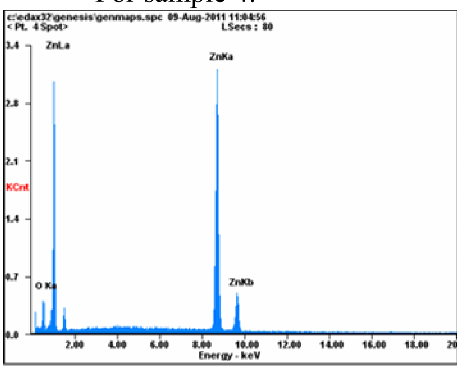


Fig. 4 – EDX analysis for 4-Functionalized linen support (without MCT-β-CD (MONOCHLOROTRIAZINYL-β-CYCLODEXTRIN)) with ZnO thermally non-treated.

Table 4

Surface Composition from EDX Measurements (Wt: Weight Percent, At: Atomic Percent)

Element	Wt [%]	At [%]
OK	08.94	28.63
ZnK	91.06	71.37

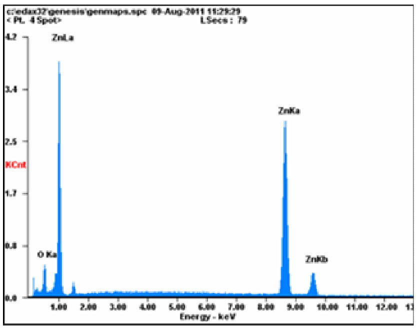


Fig. 5 − EDX analysis for 4-Functionalized linen support(without MCT- β -CD (MONOCHLOROTRIAZINYL– β - CYCLODEXTRIN)) with ZnO treated at 150ºC.

Table 5
Surface Composition from EDX Measurements (*Wt: Weight Percent, At: Atomic Percent*)

Element	Wt [%]	At [%]
OK	12.05	35.89
ZnK	87.95	64.11

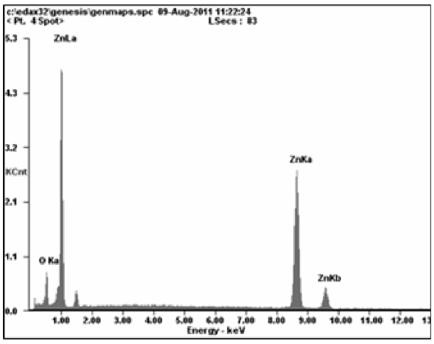


Fig. 6 − EDX analysis for 4-Functionalized linen support(without MCT- β -CD (MONOCHLOROTRIAZINYL– β - CYCLODEXTRIN)) with ZnO treated at 450ºC.

Table 6
Surface Composition from EDX Measurements (*Wt: Weight Percent, At: Atomic Percent*)

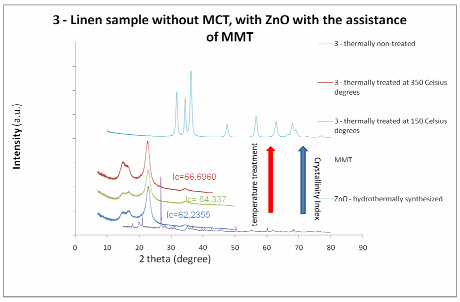
Element	Wt %	At %
OK	15.35	42.56
ZnK	84.65	57.44

From the EDX investigation a decreasing of zinc oxide content with the increasing of the treatment temperature is remarkable, for both studied samples.

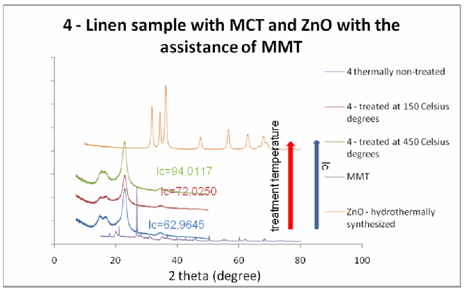
Concluding, EDX analysis revealed a proportionally decreasing of ZnO percentage content with the increasing of thermal treatment.

XRD patterns interpretation

The XRD patterns in Fig. 7 exhibit for 3 indexed sample specific peaks for flax fibres, meaning: four well defined peaks at 15.1°, 16.8°, 22.0° and 34.4°, the values of 15.1° and 16.8° for the 2 θ reflection, corresponding to the 110 crystallographic planes, respectively (Fig. 7 *a*). The other two peaks at 22.0 and 34.4 correspond to the 002 and 004 planes, which is in accordance with the literature (Revola *et al*., 1987).



a



b

Fig. 7 − XRD pattern of linen samples: *a* − without MCT-B-CD (MONOCHLOROTRIAZINYL–B-CYCLODEXTRIN), with ZnO, with the assistance of MMT, *b* − with MCT-B-CD (MONOCHLOROTRIAZINYL–B-CYCLODEXTRIN), with ZnO, with the assistance of MMT.

In the case of sample 4, three peaks appeared around 15.5°, 22.0° and 34.4°, corresponding to the 110, 002 and 004 planes, respectively (Fig. 7 *b*), which is due to the large amount of amorphous regions present in cellulose, and also to the presence of amorphous lignin and hemicelluloses, which agrees with the results of Tserki *et al*. (2005).

The peak characteristic to flax fibers (Pos. [°2Th.] ranging between 30-40 degrees) seemed to overlap to those specific to both ZnO and MMT. Although more attenuated or less visible, due to the small concentration of the ZnO solution, in our opinion, the peaks could be assigned to ZnO, as well.

In the 3 indexed treated at 150ºC sample, it is noticeable that the apex of the peak specific for MMT –is also found in the sample treated at 350ºC, meaning that MMT was not thermally degraded. Moreover, the crystallinity index increased, from a thermal stage to other. Generally, through this calcination process, it can be stated that the CI is maintained approximately constant, meaning that the assistance of MMT prior to the application by padding of zinc oxide, stabilizes in a certain extent the fibrous support.

For 4 indexed probe, a more enhanced CI has been obtained, probably due the existence of MCT-β-CD(MONOCHLOROTRIAZINYL–β-CYCLODEXTRIN) on the fibrous surface support, playing the role of encaving/entrapping/entrapment of the ZnO nanoparticles, as it can be found in a previous study. However, the XRD analysis revealed the fact that the entrapment was not good-enough. This can be explained also by the fact that the repeated cycles of washing and rinsing conducted to remove the byproducts, contributed also to the washing away of the ZnO unbound particles.

According to (Revola *et al*., 1987), a correlation between crystallite size and CI should be done, in order to generate a clear overview regarding the crystallinity of the studied supports and their novel nanoscale pattern surface achieved by the last being responsible of the thermal protection/barrier.

Table 7
Correlation Between Crystallite Size and CI

Sample	Crystallite size perpendicular to the (020) planes, by XRD measurement, [nm]	d spacing of the (020) planes distance, [Å]	C.I. (crystallinity index)
3–non-functionalized linen support with ZnO thermally non-treated	10.08	0.3889	0.622
3–non-functionalized linen support with ZnO treated at 1500C	10.32	0.8786	0.643
3–non-functionalized linen support with ZnO treated at 3500C	10.62	0.3928	0.669
4–functionalized linen support with ZnO thermally non-treated	26.74	0.9060	0.6296
4–functionalized linen support with ZnO treated at 1500C	11.92	0.387	0.7202
4–functionalized linen support with ZnO treated at 4500C	10.96	0.3929	0.9401

From Table 7 above the followings can be observed:

− at 4 indexed sample, CI increases with the decreasing of the crystallites nanoparticles;

− in case of the non-functionalized linen fibrous support, there is a proportionally increasing of CI with the augmentation of nanoparticles size.

Consequently, comparing the two probes, the 4 indexed sample has CI higher, meaning an improved crystallinity, however the 3 sample revealed also an augmentation of the crystallinity, but a reduced one. The perspective research will be oriented to more improved way/technique of fixing the ZnO nanoparticles in order to achieve/add multi-functionality of the studied fibrous nanocomposites.

4. Conclusions

The study emphasized the ideas of:

− a novel preparing technique in order to obtain nanoscale ZnO coated/covered fibrous composites, by using the cumulative/synergic effect of MMT and MCT-β-CD (MONOCHLOROTRIAZINYL–β-CYCLODEXTRIN)-β cyclodextrin;

− the thermal stability and degradation mechanism of ZnO nanocoated linen fibrous samples;

− Synergetic barrier attributes conferred by the the two new components that interfered in the preparation technique: MMT and MCT-β-CD (MONOCHLOROTRIAZINYL– β -CYCLODEXTRIN).

From the SEM photos and comparing the morholplogy of 3 and 4 indexed probes before and after thermal treatment, some randomly distributed conglomerations are noticeable, in case of *3 - non-funtionalized sample*, while on the functionalized linen sample, the particles have bigger dimensions and uniformly distributed.

EDX investigation highlighted a decreasing of zinc oxide content with the increasing of the treatment temperature is remarkable, for both studied samples.

The XRD analysis revealed the fact that the entrapment was not good-enough:

− at 4 indexed sample, CI increases with the decreasing of the crystallites nanoparticles;

− in case of the non-functionalized linen fibrous support, there is a proportionally increasing of CI with the augmentation of nanoparticles size.

The perspective research will be oriented to more improved way/technique of fixing the ZnO nanoparticles in order to achieve/add multi-functionality of the studied fibrous nanocomposites.

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STUDIU PRIVIND EFECTUL SINERGETIC AL AGENȚILOR DE SINTEZĂ ASUPRA CRISTALINITĂȚII DE SUPRAFAȚĂ A UNOR SUBSTRATURI FIBROASE PELICULIZATE CU NANOOXIZI, AVÂND IMPLICAȚII DIRECTE ASUPRA PROTECȚIEI TERMICE

(Rezumat)

Cercetarea vizează, mai întâi, evidențierea unei noi metodologii de preparare în vederea obținerii compozitelor fibroase acoperite cu nano-oxizi de zinc, prin folosirea efectului sinergic/cumulative a montmorilonitului (MMT) şi a monoclor-triazinil-beta-ciclodextrinei (MCT-β-CD), care interferă în tehnica de preparare. În al doilea rând, studiul este orientat spre protecția termică a nano-compozitelor fibroase obținute.

În vederea caracterizării compoziției, morfologiei şi cristalinității probelor obținute, s-au folosit diferite tehnici de investigare, precum: difracția cu raze X, spectroscopia FT-IR, precum şi analiza elementală EDX.