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## COMPARISON OF BP-BASED AND BP-HYBRID ANN MODELS TO PREDICT YARN TENSILE PROPERTIES

BY

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**Abstract.** Although gradient based Back propagation (BP) training algorithms have been widely used in Artificial Neural Networks (ANN) models for the prediction of ring spun yarn properties, they still suffer from serious drawbacks which include lower learning speed and tendency to converge to local minimums.

A study of the performance of gradient based Levenberg-Marquardt Back propagation algorithm (LMBP), Differential Evolution algorithm (DE) and a hybrid LMBP-DE algorithm during the prediction of yarn strength was undertaken.

From performance point of view the hybrid LMBP-DE algorithm gave the best performance during the prediction of yarn tensile properties (elongation and strength) followed closely by the LMBP algorithm. The performance of the DE was poor when compared to LMBP-DE and LMBP algorithms.

**Key words:** artificial neural network, differential evolution, cotton fibre, ring spinning, yarn, strength, elongation.

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## 1. Introduction

### 1.1. Design of Artificial Neural Network Models

Artificial Neural Network (ANN) can be trained to perform a particular function by adjusting the values of the connections (weights) between the elements of the ANN (Demuth *et al.*, 2005; Ham & Kostanic, 2003; Hagan *et al.*, 2002). The most commonly used ANN architecture is the multi-layer feed forward perceptron (MLP), in which all the information flows forward. Theoretically all the parameters of an MLP need to be carefully designed to obtain an optimum performance of the network. Several researchers (Holena, 2008; Scarselli & Tsoi, 1998; Cybenko, 1989) have demonstrated that under general regularity conditions, a sufficiently complex single hidden layer feed-forward network can approximate any member of a class of functions to any desired degree of accuracy, where the complexity of the single hidden layer feed forward network can be measured by the number of hidden units in the hidden layer. Thus a single hidden layer feed-forward network can be regarded as having universal approximation properties. Based on the aforementioned discussions an ANN model can be designed having MLP architecture and one hidden layer. Sigmoidal and Linear functions can be used in the hidden and output layers respectively (Demuth *et al.*, 2005).

### 1.2. Training MLP

#### 1.2.1. Backpropagation Algorithm

Once the ANN model has been designed it has to be trained. For the training algorithms, which use the gradient of the performance function to determine how to adjust the weights, the gradient can be determined by using a technique called Backpropagation (BP). The technique involves performing computations backwards through the network (Ham & Kostanic, 2003). Standard BP algorithms uses gradient descend or gradient descent with momentum for training. The above mentioned methods are however too slow for practical problems. Faster training algorithms have been designed and they fall into two classes, namely the heuristic techniques and the numerical optimization techniques (Demuth *et al.*, 2005). The Numerical optimization techniques are further subdivided into three categories; conjugate gradient, quasi-Newton and Levenberg-Marquardt Backpropagation algorithms (LMBP). LMBP has been reported to be more efficient when compared to other BP algorithms (Hagan *et al.*, 2002). LMBP like all other BP training algorithms however suffers from some major drawbacks which include lower speeds due to iterative nature of the algorithms and lower performance due to the presence of local minima, which leads to inconsistency and unpredictability in the performance of the ANN.

### 1.2.2. *Differential Evolution Algorithm*

Apart from BP there are other types of training algorithms, which include the differential evolution (DE) algorithm. DE is an evolutionary algorithm, which is used to optimize the minima of functions (Price & Storn, 2006; Madavan, 2002; Storn & Price, 1997). The DE algorithm is a population based direct search method, which operates in four steps; namely Initialization, Mutation, Recombination, and Selection. In comparison with other evolutionary algorithms, DE algorithm has many advantages, such as faster convergence speed, stronger stability and easy to design (Karaboga & Okde, 2004; Vesterstrom & Thomsen, 2004). The performance of the DE algorithm when used as a training algorithm in feed forward networks has however been reported to be inferior when compared to LMBP (Ilonen *et al.*, 2003).

### 1.2.3. *Hybrid Algorithms*

In view of the problems encountered when using the LMBP and the DE algorithms, hybrid algorithms for training ANN have been designed to make use of advantages found in different training algorithms (Shapiro, 2002; Yao, 1999). Global search algorithms have been reported to be useful in the search for a solution for the local minima problem experienced during the training of ANN (Floreano *et al.*, 2008; Cho & Chow, 1999; Montana & Davis, 1989). DE which functions as a global (in parameter space) optimization method and LMBP as a local (in parameter space) optimization method can be combined so as to compliment each other. Since DE starts by searching for a solution in a global manner, (*i.e.* in the entire parameter space) this may lead to a large number of possible solutions and hence the need to run the algorithm for many iterations, in order to arrive at the best performance value. The disadvantages of the LMBP algorithms of being unstable and getting stuck in local minima can be minimized by the introduction of an algorithm, which can assist in the initial weights and biases search (Jenkins, 2006). This idea has led to the design of a hybrid optimization algorithm, christened as LMBP-DE algorithm, which uses DE for the initial selection of weights and thereafter trains the algorithm using the LMBP algorithm. The designed LMBP-DE was used to predict the tensile properties of yarn and its speed, stability and performance compared to that of LMBP and DE algorithms.

## **2. Experimental Work**

### **2.1. Materials**

Cotton lint and carded ring spun yarn samples were collected from a textile firm in Kenya. The age and technology of the machinery technology were also considered and yarn samples were collected from lots deemed to have been manufactured under similar conditions. For every yarn sample collected, a

sample of the corresponding cotton lint mixture used to spin the yarn was also collected. Table 1 gives the details of the cotton samples collected. The quality characteristics of the cotton lint and yarn samples were measured under standard testing conditions.

**Table 1**  
*Characteristics of Cotton Lint*

Variable	Mean	Max	Min
Fibre length, [mm]	28.51	32.6	23.6
Short fibre content, [%]	7.37	11.75	3.5
Fibre Fineness, [dtex]	1.24	1.85	0.63
Fibre Elongation, [%]	5.35	15	1.2
Fibre Strength, [cN/Tex]	31.27	33.8	27.5
Micronaire Value	2.74	3.3	2.45
Trash Content, [%]	7.99	19.85	2.7

In this research work, a total of 54 different cotton lint samples were collected from a spinning mill in Kenya. The yarns were spun using 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. Two sets of yarn samples were collected from each cotton type. Therefore a total of 108 yarn samples were collected. For each yarn sample collected ten cops were tested. The tensile properties, twist and count were evaluated using Universal Strength tester (strength and elongation), twist tester (twist) and Count Tester (linear density) respectively. The measurements of the main properties are shown in Table 2.

**Table 2**  
*Cotton Yarn Parameters*

Variable	Mean	Max	Min
Yarn Linear density, [Tex]	49.92	80.81	19.73
Yarn Twist, [tpi]	14.97	24.49	9.07
Yarn Elongation, [%]	28.04	39.39	6.95
Yarn Strength, [cN/Tex]	819.31	1401.7	187.6

## 2.2. Methods

ANN algorithms for the prediction of yarn strength and elongation were designed based on the MLP architecture discussed in the 1.1. The inputs of the algorithms were seven fibre characteristics as given in Table 1. Yarn count and twist were also included as inputs. Two sets of models were designed; one was used to predict yarn elongation and the other yarn strength. The algorithms were trained using LMBP, DE and a hybrid LMBP-DE algorithm. The performance of the algorithms, which was measured using mean squared error (mse) was

first monitored as the number of neurons in the hidden layer was varied from 2 to 20 in steps of twos and an optimum algorithm identified. At every level of neuron, for example 2 neurons in the hidden layer, the algorithms were run 20 times and the average mse value and training time recorded. The Coefficient of Variation (CV) was also calculated.

### 3. Results and Discussions

#### 3.1. Elongation Prediction Algorithms

The performance of the elongation prediction algorithms trained using LMBP, DE and LMBP-DE is given in Table 3, which indicated that the performance of LMBP and LMBP-DE improved as the number of neurons in the hidden layer was increased.

**Table 3**  
*Performance of Elongation Algorithms*

No. of Neurons	mse values		
	LMBP	DE	LMBP-DE
2	0.1503	0.4933	0.1264
4	0.0439	0.4583	0.0256
6	0.0138	0.5281	0.0105
8	0.0040	0.5644	0.0018
10	0.0039	0.6291	0.0016
12	0.0030	0.6197	0.0013
14	0.0024	0.7081	0.0014
16	0.0017	0.7252	0.0013
18	0.0018	0.7723	0.0014
20	0.0017	0.7648	0.0014

The performance of DE on the other hand deteriorated as the number of neurons in the hidden layer was increased. The performance of DE is also poorer when compared to the performance of LMBP and LMBP-DE. LMBP stabilized when the number of neurons in the hidden layer reached 16 at an mse value of 0.0017. The performance of LMBP-DE stabilized much earlier at an mse value of 0.0013 when the number of neurons in the hidden layer was 12.

The maximum and minimum mse values for the elongation prediction algorithms are given in Tables 4 and 5 respectively. For maximum mse values DE algorithm recorded a higher mse value. This is an indication of poor performance. Considering the maximum mse value for the three algorithms it can be concluded that LMBP-DE showed a better performance followed by LMBP and lastly DE.

A similar result was observed when considering minimum mse values for the three algorithms with LMBP-DE showing the best performance followed

by LMBP and lastly DE.

**Table 4**  
*Maximum Values for Elongation Model*

No. of Neurons	maximum mse values		
	LMBP	DE	LMBP-DE
2	0.3025	0.801	0.1779
4	0.167	0.639	0.0469
6	0.04516	0.655	0.02419
8	0.01434	0.679	0.0045
10	0.0137	0.728	0.0031
12	0.00469	0.733	0.00177
14	0.0054	0.798	0.0024
16	0.0034	0.87	0.00204
18	0.00349	0.882	0.00211
20	0.00321	0.89	0.00207

**Table 5**  
*Minimum Values for Elongation Model*

No. of Neurons	minimum mse values		
	LMBP	DE	LMBP-DE
2	0.0825	0.295	0.0806
4	0.0111	0.292	0.0103
6	0.00242	0.259	0.00135
8	0.001214	0.295	0.00099
10	0.001339	0.516	0.0008
12	0.00111	0.481	0.00102
14	0.00113	0.579	0.001
16	0.00106	0.533	0.000761
18	0.00112	0.584	0.00096
20	0.00109	0.615	0.0009

The time taken by the three training algorithms is given in Table 6. DE takes a longer time to train as compared to the other two algorithms. At every level the LMBP took the shortest training time. Considering both the performance and training time LMBP-DE gave an optimum performance of 0.0013 taking 4.40 sec with 12 neurons in the hidden layer as compared to LMBP, which gave its optimum performance of 0.0017 taking 6.87 sec with 16 neurons in the hidden layer.

**Table 6**  
*Training Time for Elongation Algorithms*

No. of Neurons	Time, [sec]		
	LMBP	DE	LMBP-DE
2	1.09	6.78	1.11
4	1.30	12.17	1.44
6	1.72	17.79	2.01
8	2.27	24.93	2.78
10	2.99	32.57	3.59
12	3.70	40.83	4.40
14	5.17	53.89	6.24
16	6.87	64.97	8.03
18	9.00	81.80	10.45
20	11.80	107.30	13.44

The study of the CV of the three elongation prediction algorithms (Fig. 1) indicated that DE always gave a lower CV, followed by LMBP-DE. This implies that from the stability point of view, the best algorithm is DE, followed by LMBP-DE and lastly LMBP.

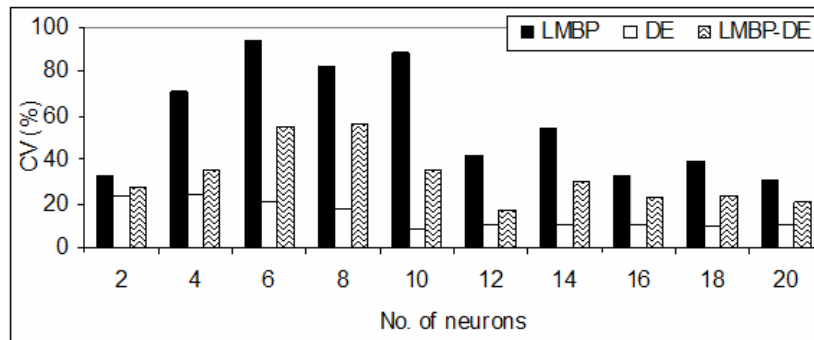


Fig. 1 – CV for elongation algorithms mse.

From mse point of view the performance of the DE algorithms was poorer when compared to the other two algorithms. Therefore a study of the difference of the means for the LMBP and LMBP-DE was undertaken and the analysis of variance (ANOVA) results are given in Table 6. The performance of the LMBP and LMBP-DE strength algorithms showed a significant difference at all the levels of neurons except at 2 and 6 neurons in the hidden layers. Considering the above mentioned result together with the results of the performance given in Tables 3,...,6 it is evident that the LMBP-DE algorithm showed a better performance when compared to LMBP algorithm during the prediction of yarn elongation.

**Table 7**  
*The ANOVA Results for LMBP and LMBP-DE Elongation Algorithms*

No. of Neurons	F value	P value	F critical
2	3.2176	0.0808	4.0982
4	6.2654	0.0167	4.0982
6	1.1749	0.2852	4.0982
8	6.8868	0.0124	4.0982
10	7.3045	0.0107	4.0982
12	23.919	$1.87 \times 10^{-5}$	4.0982
14	10.825	0.0022	4.0982
16	8.2540	0.0066	4.0982
18	6.1425	0.0178	4.0982
20	4.78	0.035	4.0982

### 3.2. Strength Prediction Algorithms

Table 8 gives the performance of the LMBP, DE and LMBP-DE algorithms used to predict yarn strength. As is the case for the elongation prediction, the strength prediction algorithms increased with the increase in the number of neurons in the hidden layer for the LMBP and LMBP-DE algorithm while DE recorded a converse behaviour. The optimum algorithm for LMBP occurred at an mse value of 0.0008 when the number of neurons in the outer layer was 14. The optimum value for the LMBP-DE also occurred when the number of neurons in the hidden layer was 14 at an mse value of 0.0003. For the DE algorithm the optimum strength prediction algorithm occurred at an mse value of 0.3419, when the number of neurons in the hidden layer was 4.

**Table 8**  
*Performance of Strength Algorithm*

No. of Neurons	mse values		
	LMBP	DE	LMBP-DE
2	0.0201	0.4505	0.0168
4	0.0098	0.3419	0.0057
6	0.0031	0.4250	0.0014
8	0.0022	0.5224	0.0011
10	0.0017	0.5509	0.0012
12	0.0012	0.6283	0.0006
14	0.0008	0.6221	0.0003
16	0.0007	0.6473	0.0003
18	0.0006	0.6932	0.0003
20	0.0006	0.7058	0.0003

The maximum and minimum mse values for the three algorithms during the prediction of yarn strength are given in Tables 9 and 10 respectively. DE consistently



showed a poorer performance when compared to the other two algorithms.

**Table 9**  
*Maximum Values for Strength Model*

No. of Neurons	maximum mse values		
	LMBP	DE	LMBP-DE
2	0.0276	0.695	0.025
4	0.035	0.649	0.022
6	0.0067	0.59	0.0029
8	0.011	0.642	0.0015
10	0.0054	0.69	0.0016
12	0.0046	0.772	0.0011
14	0.0016	0.743	0.0005
16	0.0014	0.763	0.0007
18	0.0015	0.825	0.0007
20	0.0015	0.829	0.0007

LMBP-DE on the other hand showed a better performance in all occasion except when the number of neurons in the hidden layer was 8 for the maximum mse values and when the number of neurons in the hidden layer was 6 and 18 for the minimum mse values. Consequently LMBP-DE can be adjudged to have shown a better performance followed by LMBP and lastly DE.

**Table 10**  
*Minimum Values for Strength Model*

No. of Neurons	minimum mse values		
	LMBP	DE	LMBP-DE
2	0.01301	0.178	0.0099
4	0.0024	0.163	0.0018
6	0.00138	0.162	0.00014
8	0.00132	0.157	0.0004
10	0.00112	0.301	0.00025
12	0.00024	0.527	0.00004
14	0.00007	0.437	0.000007
16	0.00007	0.498	0.000001
18	0.00003	0.55	0.00006
20	0.00006	0.564	0.00001

As indicated in Table 11, DE strength prediction algorithm recorded the slowest speed when compared to the other two algorithms, while LMBP recorded the fastest training speed, followed closely by LMBP-DE. Considering both time and performance the optimum algorithm for the LMBP occurred when the number of neurons in the hidden layer was 18 taking 8.83 sec, while that for LMBP-DE occurred when the number of neurons in the hidden layer was 14 taking 6.59 sec for training.

**Table 11**  
*Training Time for Strength Algorithm*

No. of Neurons	Time, [sec]		
	LMBP	DE	LMBP-DE
2	1.05	6.77	1.10
4	1.26	11.85	1.47
6	1.69	17.81	2.00
8	2.26	24.78	2.80
10	2.85	32.27	3.52
12	3.78	41.21	4.63
14	5.30	52.10	6.59
16	7.05	64.10	8.20
18	8.83	78.20	10.55
20	11.47	104.80	13.56

The optimum algorithm for the DE showed an mse value of 0.3419, taking 11.85 sec for training when the number of neurons in the hidden layer was 4. Fig. 2 gives the CV for the three strength prediction algorithms. Just like in the case of elongation prediction algorithm, LMBP again showed the worst CV as compared to the other two strength prediction algorithms. This could be an indication of the DE strength prediction algorithm being more stable followed by LMBP-DE and lastly LMBP.

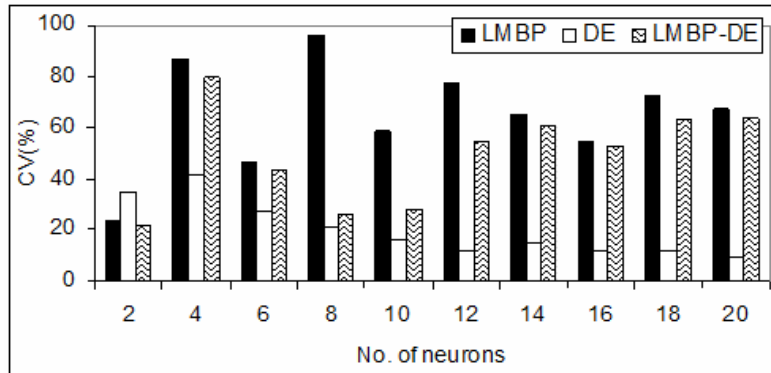


Fig. 2 – CV for strength algorithms mse.

The ANOVA results for the LMBP and LMBP-DE strength prediction algorithms given in Table 12 indicate that the algorithms showed significant difference in their performance for all levels of neurons, except when the number of neurons in the hidden layer was 4. When the results in Table 12 are considered together with the results in Tables 8,...,11, it is evident that LMBP-DE showed a better performance when compared to the LMBP.

**Table 12**  
*ANOVA Results for LMBP and LMBP-DE Strength Algorithms*

No. of Neurons	F value	P value	F critical
2	5.62130	0.0229	4.0982
4	3.34190	0.0758	4.0982
6	23.9484	$1.85 \times 10^{-5}$	4.0982
8	5.88180	0.0202	4.0982
10	4.98700	0.0315	4.0982
12	6.58210	0.0144	4.0982
14	18.8918	$9.97 \times 10^{-5}$	4.0982
16	15.9957	0.0003	4.0982
18	10.5952	0.0024	4.0982
20	8.49040	0.0061	4.0982

#### 4. Conclusions

A study of the performance of LMBP, DE and a hybrid LMBP-DE algorithms during the prediction of yarn tensile strength properties (strength and elongation) was undertaken as the number of neurons were varied from 2 to 20 in steps of two. In terms of speed (training time), LMBP was better than LMBP-DE and DE was last. From performance (mse) point of view however LMBP-DE was better than LMBP and the DE algorithm remained last again. Considering the CV of the mse value the DE algorithm gave the best (least CV) results followed by LMBP-DE and LMBP was third. The optimum elongation prediction algorithm for the LMBP-DE took 4.4 sec for training, showed a performance of 0.0013 with 12 neurons in the hidden layer, while that of LMBP took 6.87 sec for training with an mse value of 0.0017 with 16 neurons in the hidden layer. DE elongation prediction algorithm took 12.17 sec for training with an mse value of 0.453 and 4 neurons in the hidden layer. For the strength prediction the optimum algorithm for LMBP-DE was recorded at an mse value of 0.0003 taking 6.59 sec for training when the number of neurons in the hidden layer was 14, while LMBP showed an mse value of 0.0006 taking 8.83 sec for training at 18 neurons in the hidden layer. DE strength prediction algorithm took 11.85 sec for training and showed an mse value of 0.3419 when the number of neurons in the hidden layer was 4. It can therefore be concluded that LMBP-DE algorithm gave the best optimum algorithm for the prediction of yarn elongation and strength followed by LMBP algorithm and lastly DE algorithm.

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STUDIU COMPARATIV AL MODELELOR ANN DE TIP BP  
ȘI BP-HIBRID UTILIZATE PENTRU ESTIMAREA COMPORTĂRII LA  
ÎNTINDERE A FIRELOR

(Rezumat)

Deși algoritmi Backpropagation (BP) bazați pe gradient au fost des folosiți în rețelele artificiale neurale (ANN) ca modele pentru estimarea proprietăților firelor filate, există încă multe probleme serioase, inclusiv viteză mai scăzută de învățare și tendința de a converge către minimurile locale. Lucrarea prezintă un studiu privind nivelul de performanță a algoritmilor Levenberg-Marquardt Backpropagation (LMBP), de evoluție diferențială (DE) și algoritmului hibrid LMBP-DE în cazul predicției rezistenței firelor. Din punct de vedere al performanței, algoritmul hibrid a avut cele mai bune rezultate privind predicția comportării la tracțiune (alungirea și rezistență), urmat de algoritmul LMBP. În comparație cu cele două algoritmi, rezultatele obținute cu algoritmul DE au fost mai slabe.



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## INVESTIGATION ABOUT THE AIR PERMEABILITY OF WEFT KNITTED STRUCTURES

BY

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**Abstract.** In recent years, the importance of the thermo-physiological (thermal) comfort has rapidly increased with the development of textile technology. Nowadays it is one of the most important characteristic especially for technical uses such as medical textiles, protective clothes and sportswear. Thermal comfort mainly depends on heat, moisture and air transfer properties. In this study, the air permeability parameter was investigated in detail and a simple equation is derived for weft knitted structures based on two parameters: *thickness* and *porosity*.

**Key words:** thermal comfort, air permeability, thickness, porosity, knitted structures.

### 1. Introduction

Thermal comfort can be defined as a pleasant state of psychological and physical harmony between a human being and environment (Önder & Sarier, 2004). Factors affecting thermal comfort are numerous, including heat exchange

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within clothing, air permeability, the transfer and evaporation of moisture, among others. Clothing must assist the thermal control function of the body under changing physical loads in such a way that the thermal and moisture management is balanced and that a microclimate is created next to the skin. This physiological effect is extremely important, especially in the case of clothing for medical, protective and sports. Items of clothing with poor thermo-physiological wear characteristics not only detract from the well-being of the human but also impair his/her physical performance and may even act as a health hazard (Umbach, 1993; Bivainytė & Mikučionienė, 2011).

Air permeability (being a biophysical feature of textiles) determines the ability of air to flow through the fabric (Bivainytė & Mikučionienė, 2011). It is an important factor in the comfort of a fabric as it plays a role in transporting moisture vapour from the skin to the outside atmosphere. The assumption is that vapour travels mainly through fabric spaces by diffusion in air from one side of the fabric to the other (Mavruz & Ogulata, 2009; Karaguzel, 2004).

In the previous researches it is found that air permeability is significantly affected by fiber, yarn and fabric characteristics and also finishing treatments; whereas the fabric structure (such as weight, thickness, tightness and porosity) is accepted as the most important factor affecting air permeability, among these characteristics. It is logical to expect that structural characteristics have an impact on air permeability; this is dependent on porosity. However there is limited experimental evidence in the literature that correlate these properties (Bivainytė & Mikučionienė, 2011; Mavruz & Ogulata, 2009; Çay *et al.*, 2007; Ogulata & Mavruz 2010; Militký *et al.*, 2004).

In this study, theoretical values for air permeability are determined by multi regression analysis based on thickness and porosity values. This new prediction equation can be defined air permeability property for knitted structures regardless of material type and structure. The values and the trends in the change of the air permeability will be used in the next step of this investigation in order to determine its influence over the different heat transfer mechanisms - conductivity, convection and radiation, responsible for the heat transfer.

## 2. Materials and Methods

Analysis was carried out on different weft knitted fabrics produced on Stoll CMS 302-TC electronic flat knitting machine (E8) with three different tightness factors in plain, 1x1 rib and interlock structures using 33Tex\*4 wool yarn (Table 1).

The air permeability values were measured using TexttestFX3300 device according to EN ISO 9237 ( $\Delta p = 100$  Pa,  $S_w = 200$  cm<sup>2</sup>). Also the physical fabric properties like weight, thickness and porosity properties were defined under standard atmospheric conditions according to the related standards.



In order to determine the effects of fabric properties on air permeability; a multi regression analysis was performed using SPSS Software. At the end of the study, the predicted and measured air permeability values of various knitted fabrics produced on the same machine with different materials (42Tex\*3 Co and 20Tex\*7 PES yarns, Table2) were also compared.

### 3. Results and Discussions

The results of all measurements are given in Tables 1 and 2.

**Table 1**  
*Characteristics of the Wool Knitted Fabrics*

SAMPLE	Interlock - Wool			1x1 Rib - Wool		
	Tight -1-	Medium -2-	Loose -3-	Tight -4-	Medium -5-	Loose -6-
Weight, [g/m <sup>2</sup> ]	739.00	704.00	611.00	582.00	533.00	446.00
Thickness, [mm]	2.95	3.07	3.28	2.48	2.61	2.76
Porosity, [%]	0.81	0.83	0.86	0.82	0.85	0.88
Air Permeability, [L/m <sup>2</sup> /s]	697.33	863.33	1113.33	809.33	1016.67	1330.00

**Table 1**  
*Continuation*

SAMPLE	Plain - Wool		
	Tight -7-	Medium -8-	Loose -9-
Weight, [g/m <sup>2</sup> ]	334.00	294.00	240.00
Thickness, [mm]	1.46	1.47	1.48
Porosity, [%]	0.83	0.85	0.88
Air Permeability, [L/m <sup>2</sup> /s]	1143.33	1493.33	2250.00

**Table 2**  
*Characteristics of the Cotton and Polyester Knitted Fabrics*

SAMPLE	Plain - Cotton			Plain - PES		
	Tight -10-	Medium -11-	Loose -12-	Tight -13-	Medium -14-	Loose -15-
Weight, [g/m <sup>2</sup> ]	383.00	324.00	270.00	388.00	349.00	280.00
Thickness, [mm]	1.53	1.56	1.63	1.45	1.53	1.66
Porosity, [%]	0.83	0.86	0.89	0.81	0.83	0.88
Air Permeability, [L/m <sup>2</sup> /s]	1330.00	1723.33	2353.33	950.67	1396.67	2083.33

In previous researches it was noted that airflow through textiles is mainly affected by the pore characteristics of fabrics. However in our study, the

statistical analysis indicates a low correlation between porosity and air permeability (Fig. 1,  $R^2 = 0.70$ ).

That's why another parameter is also needed to determine air permeability and the thickness values are integrated to the equation as an independent variable. The result of regression analysis indicates that the air permeability characteristic can be defined with porosity and thickness values (Table 3).

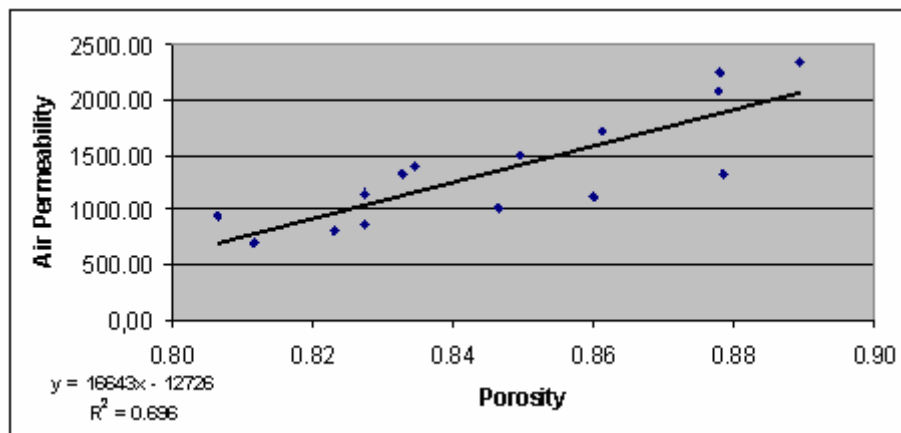


Fig. 1 – Relationship between porosity and air permeability values.

**Table 3**

*Regression Analysis for Air Permeability Based on Thickness and Porosity*

Coefficients <sup>a</sup>					
Model		Unstandardized Coefficients		Standardized Coefficients	
		B	Std. Error	Beta	
1	(Constant)	-8915.190	2367.137		-3.766 0.009
	Porosity	12898.186	2737.341	0.688	4.712 0.003
	Thickness	-334.044	93.543	-0.521	-3.571 0.012

a. Dependent Variable: Air permeability

A new simple equation is derived from this statistical analysis as following Eq. (1). This equation has high coefficient of correlation (Table 4,  $R^2 = 0.88$ ).

$$\text{Air permeability} = -8915.190 + 12898.186 P - 334.044 h \quad (1)$$

where: P = Porosity, [%]; h = Thickness, [mm].

**Table 4**  
*Coefficient of Correlation for New Model*

Model Summary				
Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	0.936 <sup>a</sup>	0.877	0.836	190.64303

Predictors: (Constant), Thickness, Porosity

Fig. 2 shows the predicted and measured air permeability values for analyzed knitted samples.

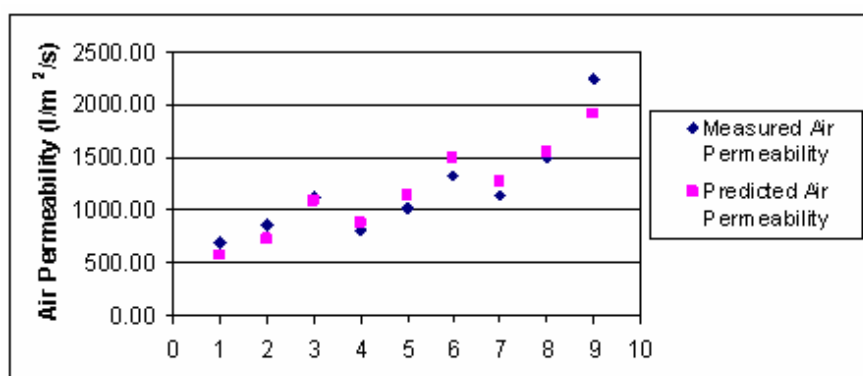


Fig. 2 – Predicted and measured air permeability for analyzed structures.  
The horizontal axis presents the number of the sample according to Table 1.

Table 5 and Fig. 3 show the predicted and measured air permeability values for some other knitted structures.

**Table 5**  
*Measured and Predicted Air Permeability Values for Different Knitted Structures*

Sample			Thickness [mm]	Porosity [%]	Measured Air Permeability [L/m²/s]	Predicted Air Permeability [L/m²/s]	Real Error (%)
Plain Cotton	Tight	-10-	1.53	0.83	1330.00	1316.89	0.99
	Medium	-11-	1.56	0.86	1723.33	1672.73	2.94
	Loose	-12-	1.63	0.89	2353.33	2011.63	14.52
Plain PES	Tight	-13-	1.45	0.81	950.67	1002.26	-5.43
	Medium	-14-	1.53	0.83	1396.67	1338.51	4.16
	Loose	-15-	1.66	0.88	2083.33	1852.99	11.06

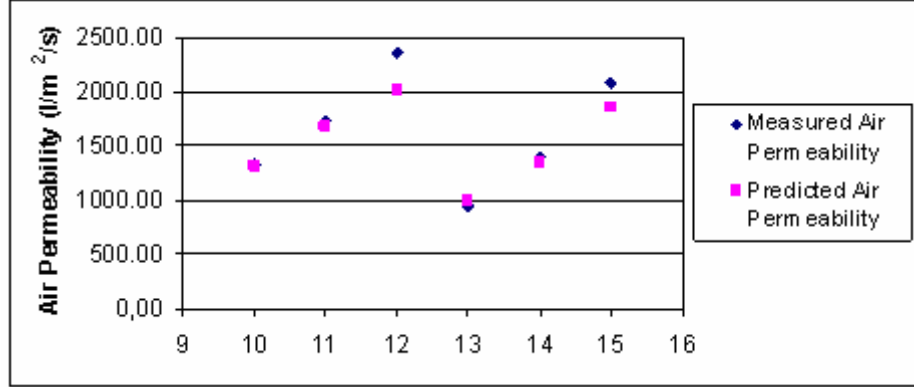


Fig. 3 – Measured and predicted air permeability values for different knitted structures.

These results indicate that the formulated equation is effective in prediction of air permeability for also different structures with the following limits:

$$\text{Min 75\% porosity – Min 1.32 mm thickness} \quad (2)$$

However it is known that the porosity and thickness properties of weft knitted structures are also within these limits.

By analysing the equation, it can be seen that for the similar structures the porosity has about 40 times higher influence on the the air permeability than the thickness (Fig. 4).

For the investigation of the thermal comfort, the heat transfer in the body-clothing-environment system is classified into various mechanisms such as heat conduction, convection and thermal radiation transfer. The air permeability and especially the porosity determine which of these mechanisms will be more effective and thus will influence the thermal properties significantly.

Additionally, if we have the geometry of the knitted structure from the simulation programs, where it is possible to calculate the porosity and thickness numerically (Kyosev & Renkens, 2004), then using this equation it is possible to calculate the air permeability of these structures based on the simulation (Fig. 5).

If the XY-projection is used, the porosity can be calculated by the following equation based on this unit cell.

$$\text{Porosity} = \frac{\text{Surface of the unit cell} - \text{Surface of the yarns}}{\text{Surface of the unit cell}}, [\%] \quad (3)$$

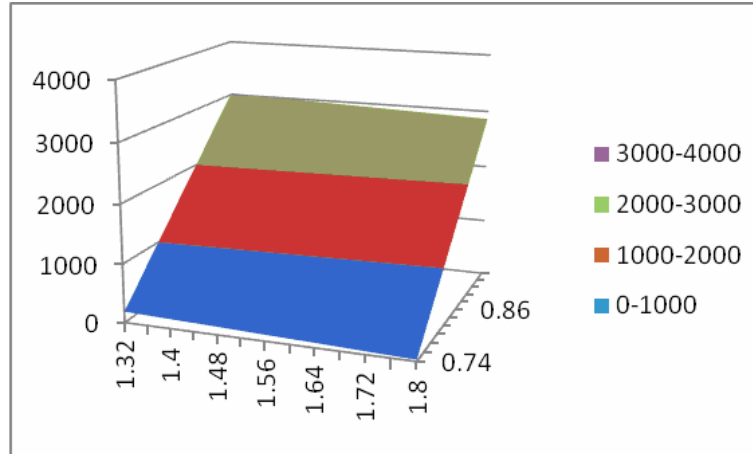


Fig. 4 – Predicted air permeability values for knitted structures based on different thickness and porosity values.

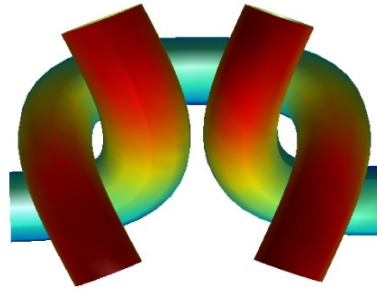


Fig. 5 – Simulated a weft knitted unit cell.

Since the data about the yarn axis is available as coordinates, as well fibre volume fraction can be calculated on the basis of the volume of the yarns in the unit cell:

$$\text{Fiber\_Volume\_Fraction} = \frac{\text{Yarn\_volume}}{\text{Unit\_cell\_volume}} = \frac{\sum_{\text{all\_yarns}} L_i \cdot S_i}{w \cdot b \cdot t}, [\%] \quad (4)$$

where:  $L_i$  is the length of each yarn piece in the unit cell;  $S_i$  – the cross section area of this yarn piece and  $w$ ,  $b$  and  $t$  – the width, height and the thickness of the unit cell. Using the fiber volume fraction the porosity of the structure in the 3D meaning can be calculated. This porosity is an approximation of the real porosity, because the cross section of the yarns consists as well the air inside the staple yarns,

but anyway the calculated value can be used as approximation of the porosity so, that the properties of the fabrics can be calculated prior the real production.

#### 4. Conclusions

In this study, a new equation was derived based on thickness and porosity parameters with a high correlation coefficient. This high value indicates that air permeability can be mainly determined by fabric structural characteristics, regardless of material type. By this relation, the characteristics of fabrics can be engineered according to the end-use requirements before production. So this prediction will have an important role especially for the researches of new fabric structures.

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## STUDII PRIVIND PERMEABILITATEA LA AER A TRICOTURILOR DIN BĂTĂTURĂ

(Rezumat)

În ultimii ani, importanța confortului termo-fiziologic (termic) a crescut semnificativ odată cu dezvoltarea tehnologiilor textile. În prezent, confortul reprezintă una din cele mai importante caracteristici, în special în cazul unor aplicații tehnice, cum ar fi textilele medicale, îmbrăcămintea de protecție și cea sportivă. Confortul termic depinde în principal de caldura, umiditate și proprietățile de transfer ale aerului. În prezentul studiu este investigată permeabilitatea la aer în cazul tricotelor din bătaură, obținându-se o ecuație simplă care se bazează pe doi parametri: grosime și porozitate.





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## THE INFLUENCE OF ZINC OXIDE TREATMENT ONTO COMFORT PARAMETERS OF TEXTILE MATERIALS

BY

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**Abstract.** The paper shows the results of experimental researches aiming at the comfort features of textile materials antimicrobial treated with zinc oxide and designed to the Board Police uniforms clothing. The most efficient, the optimal way for the antimicrobial treatment was established, based on the values obtained for parameters like: air permeability, vapours permeability and roughness, well correlated with the requirements imposed by the destination.

**Key words:** antimicrobial agent, comfort characteristics, uniform, material textile material, zinc oxide.

### 1. Introduction

The textile materials designed for garment manufacturing for the State Police represents a good environment for bacteria and fungi development. Subsequently, the occurrence of unpleasant odours, discomfort, health issues, staining or degradation, lack of performance, etc. might be possible. For that

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reason, the thermo-physiological comfort, UV protection and antibacterial properties are very important (Zampetakis & Katsaros *et al.*, 2006).

Researchers in the domain are focused on increasing clothing life time, by using some innovative protection techniques, aimed at finding some optimal solutions for increasing the functional character of clothing. For this reason, some special methods for applying active agents onto textiles surfaces are used, so that the garments have increased life time and are multifunctional.

Fabrics with improved quality indices can be obtained by using textiles with special characteristics in terms of both processing and physical-mechanical and chemical characteristics (Avram & Mustață, 1999).

Antimicrobial protection is very important for garments used by active people, such as Police troopers. This type of protection is obtained using special treatments. Some specific agents met in the antimicrobial applications are presented in Fig. 1.

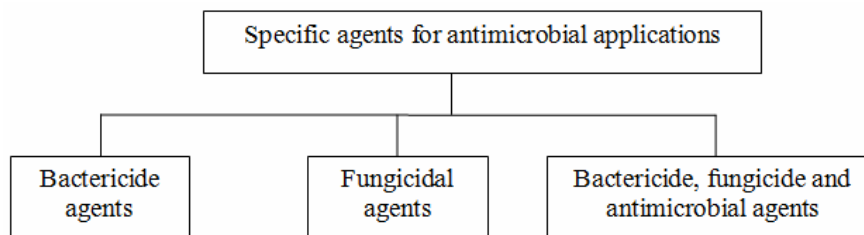


Fig. 1 – Specific agents for antimicrobial applications.

These agents are:

- *Bactericide agent* - used to inhibit bacteria development. The textiles impregnated with such a substance will eliminate the presence germs;
- *Fungicide agent* - used to inhibit fungi augmentation;
- *Bactericide, fungicide and antimicrobial agents*: are substances killing all three types of microorganisms.

## 2. Materials and Methods

In case of State Police uniforms, antimicrobial finishing insures the enhancement of comfort parameters.

The paper presents an experimental study concerning the antimicrobial finishing of a textile woven fabric made of 100% PES yarns, with twill structure, using zinc oxide based active agent that is applied by padding method. The antimicrobial agent is padded as an emulsion and then squeezed.

The comfort characteristics determined in order to define the comfort state of the treated fabric are: air and vapour permeability  $P_a$  and respectively  $P_v$ , resistance to vapour transfer  $R_v$  and roughness  $R_q$  have been determined for:

- Untreated PES fabric (witness sample);
- Fabric treated with Apretan solution;
- Fabric treated with 1% solutions of Apretan and zinc oxide (2g ZnO + 16 g binder + 200 mL water);
- Fabric treated with 1% solutions of Apretan and zinc oxide (2g ZnO + 16 g binder + 20 mL methanol + 200 mL water);
- Fabric treated with solution of Apretan, 1% zinc oxide and methanol after a 48 h treatment with 2g ZnO + 16 g binder + 20 mL methanol + 200 mL water.

The finishing treatment with zinc oxide included the following stages:

- padding – has been performed on a Werner Mathis AG padder, at 2 bars pressure;
- drying – on a Vetter beck, for 3 min at 110°C;
- cross linking – on a Vetter beck, for 3 min, at a temperature of 150°C.

Air permeability (Pa) was determined according to ASTM D737 (2008), vapour permeability (Pv) according to STAS 9005-79, resistance to vapour transfer (Rv) according to ASTM D1518 Guarded hot plate method (2008). The fabric roughness (Rq) was measured using DIGITAL INSTRUMENTS NANOSCOPE, based on surface analysis methods.

Vapour permeability and resistance to vapour transfer were calculated using:

$$P_v = \frac{24M}{At} \quad (1)$$

$$R_v = d / P_v, [\text{mm. h. m}^2/\text{g}] \quad (2)$$

where:  $P_v$  – vapour permeability,  $[\text{g}/\text{m}^2\text{h}]$ ;  $M$  – mass loss;  $A$  – internal area of the dish,  $[\text{m}^2]$ ;  $t$  – time between weighings = 72 h;  $d$  – fabric thickness,  $[\text{mm}]$ ;  $R_v$  – resistance to vapour transfer,  $[\text{mm. h. m}^2/\text{g}]$ .

### 3. Results and Discussions

The comfort characteristics are presented in Table 1 and plotted in the graphics from Figs. 2, 3 and 4.

The data presented in Table 1 show that the antimicrobial treatments increase the fabric thickness with almost 2%.

Fig. 2 presents the variation of air permeability. It shows that variant 5 (PES woven fabric treated with a solution with Apretan + 1% zinc oxide + methanol, after 48 h in emulsion) has the highest value, with approximately 18% more than the witness sample.

**Table 1**  
*Comfort Characteristics*

	Variant	Pa [L/m <sup>2</sup> /s]	d [mm]	Pv [g/m <sup>2</sup> /day]	μ [g/m <sup>2</sup> h]	Rv [mm*m <sup>2</sup> h/g]	Rq [nm]
1	Woven fabric PES	519.443	0.23	1.15	12.2340	0.0188	21.032
2	PES + Apretan	559.039	0.234	1.13	12.1243	0.0193	39.057
3	PES + Apretan + + 1% ZnO	558.835	0.234	1.098	11.7	0.02	32.638
4	PES + Apretan + + 1% ZnO + + methanol	594.148	0.234	1.16	12.413	0.01885	39.335
5	PES + Apretan + + 1% ZnO + + methanol for 48 h	601.158	0.234	1.27	13.526	0.0173	23.309

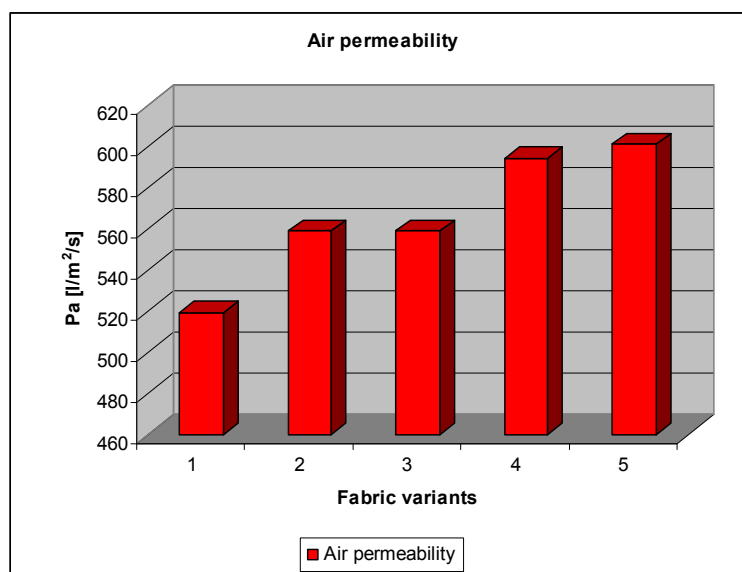


Fig. 2 – Air permeability for the tested samples.

The garment destination requires high values for vapour permeability, corresponding to low resistance to vapour transfer. The data plotted in Fig. 3 show that the maximum resistance to vapour transfer corresponds to variant 3, while the minimum value to variant 5.

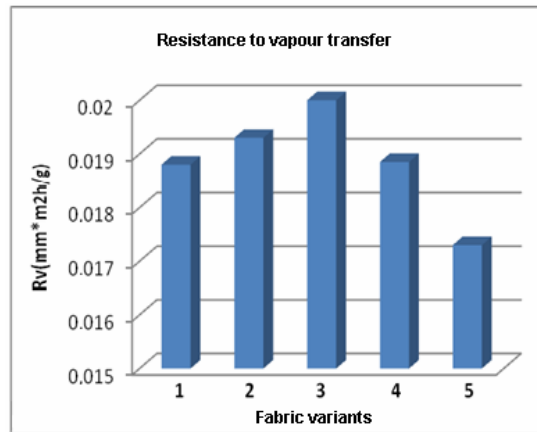


Fig. 3 – Resistance to vapour transfer for the tested samples.

Correlated to the data regarding air permeability, it can be estimated that variant 5 (fabric treated with solution of Apretan, 1% zinc oxide and methanol after a 48 h treatment with 2g ZnO + 16 g binder + 20 mL methanol + 200 mL water) is the best option when considering the comfort characteristics.

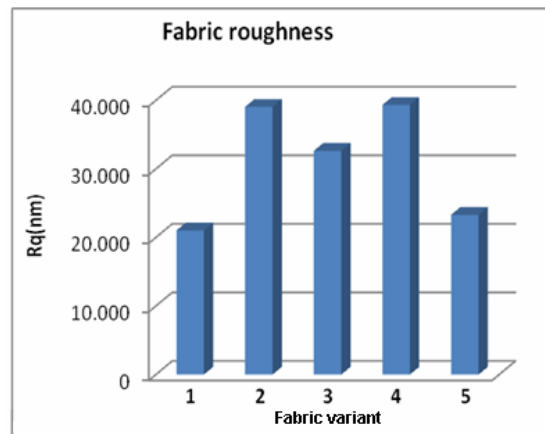


Fig. 4 – Fabric roughness.

The graphic in Fig. 4 shows that variant 5 presents the closest value to the witness (non-treated fabric), indicating that this treatment leaves the smoothest surface.

Fig. 5 illustrates the data obtained from surface analysis when determining fabric roughness. The highlighted area represents the zone in which the roughness has been determined for variant 5.

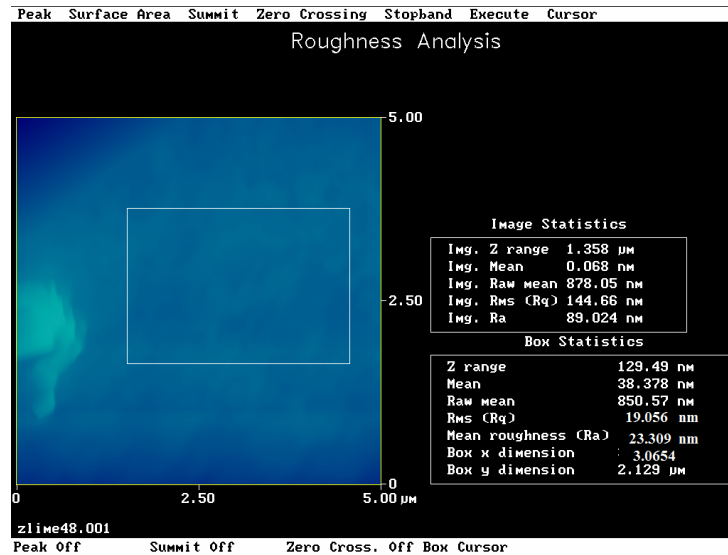


Fig. 5 – Microscopic image of roughness for the sample treated with PES + Apretan + 1% ZnO + methanol for 48 h.

#### 4. Conclusions

The materials treated with zinc oxide and analysed in the present study, are designed for police clothing/uniform. Subsequently, the requirements regarding the comfort parameters are as follows: high air permeability, low vapour resistance, low roughness. From the analysis of the values displayed in Table 1 and plotted in Figs. 2,...,4, it can be concluded that the woven fabric from PES yarns treated with APRETAN solution, 1% solution zinc oxide and methanol, after a 48 h treatment in acrylic emulsion responds best to the comfort requirements imposed by destination.

**Acknowledgments.** Authors are grateful to the financial support provided by the European Funds and Romanian Government, EURODOC “Doctoral Scholarships for Performance at European Level Research”, 88/1.5./S/59410 and POSDRU No. /89/1.5/S/49944.

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INFLUENȚA TRATAMENTULUI CU OXID DE ZINC  
ASUPRA PARAMETRILOR DE CONFORT AI  
MATERIALELOR TEXTILE

(Rezumat)

Articolul prezintă cercetări experimentale asupra caracteristicilor de confort ale materialelor textile tratate antimicrobial cu oxid de zinc și care sunt proiectate pentru uniforme de Poliție de frontieră. A fost stabilit modul optim al tratamentului antimicrobial bazat pe valorile obținute ale parametrilor ca: permeabilitate la aer, permeabilitate la vapori și rugozitate, corelate cu cerințele impuse de destinație.





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TEXTILE. PIELĂRIE

## THE ROLE OF PELTIER CELLS IN THE CONTROL OF THE MICROCLIMAT IN CLOTHING

BY

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**Abstract.** Currently, the problems facing the textile industry regard the stiff competition and one of the solutions to meet this competition is to find the niches allowing for development opportunities. Many companies specializing in the production of materials and textiles have been, for a long time, improving and perfecting products and processes, which today is not enough. In this context, after the development of technical and functional textiles, the latest developments in textiles concern the emergence of the smart textiles, which is a group of products with many innovative applications. Recent research in this area concerns the attachment of electronic systems to clothing, in order to protect the wearer from extreme factors such as too low or too high temperatures. This paper takes into consideration the current concerns manifested in Europe with an original character and aims to a scientific approach to the role of control and heat exchanging using Peltier cells included in clothing. Starting from the presentation of Peltier cells, from experiments made, the paper shows the level of energy contribution such items bring in clothing ensembles that are exposed to high or low temperatures. The energy transfer is expressed as comfort parameter, namely thermal resistance.

**Key words:** Peltier cells, thermal comfort, thermal resistance.

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## 1. Introduction

Normal body temperature is achieved through heat processes – thermolitic process for heat production and thermogenesis processes for heat loss. The two types of processes are in a constant dynamic equilibrium provided by the intervention of biological factors regulating the production and also the loss of heat. The quantity of heat produced in the body in a certain period is equal to the quantity of heat lost through the various ways in the same time. Much of this quantity of heat is lost through clothing. Therefore, its features should be chosen so as to ensure the balance of heat exchange, thus avoiding the difference between thermogenesis and thermolysis. The heat source in this case is the body and clothing represents thermal protection, facilitating the heat transfer to the environment.

Often it is necessary for the body to be helped with an additional external energy when heating and cooling are needed, so that the energy balance is ensured. This is the reason why the research presented in this paper considered the use of Peltier cells. The overall amount of heat in the body is assessed based on the different forms of metabolism: basal, energy, total. Basal metabolism is the quantity of heat lost per unit area in unit time, expressed in kcal/m<sup>2</sup> h. It is different according to sex and age.

In order to achieve an improved thermal transfer between the wearer and the environment, thus creating clothing with new functions, the concept of clothing involves, in addition to a classical approach, the use of high performance materials, advanced technologies, the so called “high-tech” technologies and integrated micro-systems.

Current research in smart materials is intended to adapt structures that can have the ability to change characteristics depending on environmental exposure strain. Intelligent textile materials meet these requirements; they have a dynamic behaviour with the ability to react and change certain characteristics (shape, colour, size, etc.) and can be produced by introducing substances with special properties (polymer, ceramic substances, metal alloys, paraffins and salts) in textile materials of different nature. Textiles with phase change materials (PCMs) have the ability to store body heat and release it when needed. The substances used for heat storage and re-usage are inorganic salts and mixtures of salts and paraffins. Paraffin is encapsulated and integrated into the fabric, the fibre mass or is “suspended” in foams of various types. Although products made of PCM materials present extensive use, they still have some disadvantages:

- the balance between the heat generated by the body during strenuous physical exercise and the heat released in the environment is not always respected;
- intensity and duration of effect of thermal insulation depends on thermal storage capacity of PCM microcapsules, the quantity of the material and fabric structure, and therefore need a suitable dosage depending on the type of work performed for each product.

These disadvantages can be compensated by products with incorporated electronic devices, which are an ideal interface between the human body and environment. In order to ensure comfort at both high and low temperatures, Peltier cells can be introduced between certain layers of clothing. The use of Peltier cells has the role of microclimate temperature regulation, according to environment thermal conditions, thus ensuring wearer comfort. While Peltier effect is known for a long time, being used successfully in other technical fields and even in medicine, it is still in the research phase in the textile industry. Peltier effect was discovered by Jean-Charles Peltier, a French physicist, in 1834. The opposite phenomenon is called the Seebeck effect. As both phenomena are reversible, some researchers call them Seebeck-Peltier effect. Fig. 1 presents the structure of a Peltier cell.

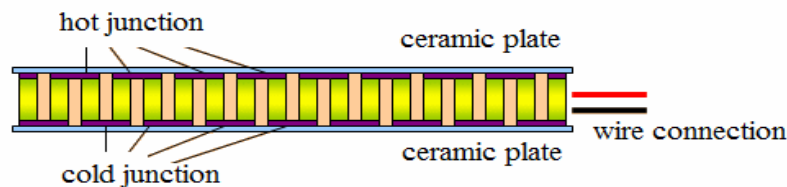


Fig.1 – Peltier cell structure.

When a Peltier cell works, a quantity of heat absorbed by the cold side ( $P_C$ ) and another amount of heat ( $P_H$ ) is released from the hot side. Since the cell absorbs the energy that is converted to heat,  $P_H$  is higher than  $P_C$  according to Fig. 2.

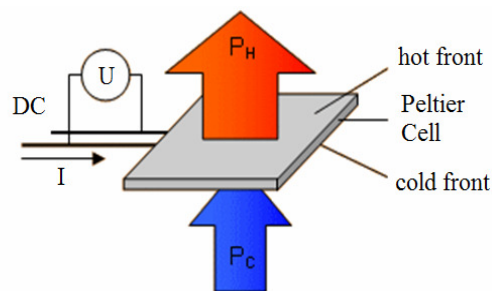


Fig. 2 – Heat transfer from the Peltier cells.

Energy balance is:

$$P_H = P_C + U I \quad (1)$$

where:  $P_C$  – power absorbed in cooling, [W];  $P_H$  – hot ceded power, [W];  $U$  – voltage, [V];  $I$  – intensity of electric current, [A].

Peltier effect consists in transferring the heat from a cold to a warmer zone, using electricity. When inserting a voltage source of intensity  $I$  in the loop consisting of two wires from different metals, one junction cools, while the other heats. An Eq. (2), similar to Eq. (1) can be applied so the cooling capacity ( $Q$ ) is proportional to the difference in Peltier coefficients of metals  $\Pi_A$ ,  $\Pi_B$  and current intensity  $I$ , as illustrated in Fig. 3.

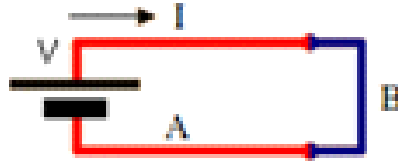


Fig. 3 – Electric scheme Peltier cell.

$$Q = AB \cdot I = (\Pi_B - \Pi_A) \cdot I, \text{ [J]} \quad (2)$$

where:  $\Pi_{AB}$  – Peltier coefficient for the entire thermocouple, [V·h];  $\Pi_A$ ,  $\Pi_B$  – Peltier coefficients for each material, [V·h];  $I$  – intensity of electric current, [A].

Peltier coefficient is the amount of heat absorbed by the lower junction per unit time (Wikipedia, 2011). Heat transfer is controlled by the current flow direction (polarity). Heat is thermal energy contained in matter. Temperature measures the tendency of a body to accept or release heat. Heat will always transfer from places with high temperature to places with low temperature. The heat contained in a body depends on its temperature, mass and specific heat ( $C_s$ ). Specific heat is a constant for each substance. The specific heat of water is 4186 J/(kg·K). The energy needed to heat the body from temperature  $T_1$  to  $T_2$  is:

$$Q = m \cdot C_s \cdot (T_1 - T_2), \text{ [J]} \quad (3)$$

where:  $Q$  – quantity of heat to warm the body from  $T_1$  to  $T_2$ ;  $T_1$  – initial temperature, [K];  $T_2$  – final temperature, [K];  $m$  – body mass, [kg];  $C_s$  – specific heat, [J/kg·K].

Heat transfer between two solid bodies depends on the temperature difference and the contact area. If one body is a fluid (liquid or gas), it also depends on the speed of the liquid surface contact. Heat transfer is physically and mathematically similar to the flow of electricity. Temperature difference is similar to voltage and heat flux is similar to electricity. Fig. 4 illustrates similarity between the transfer of heat and the flow of electricity.

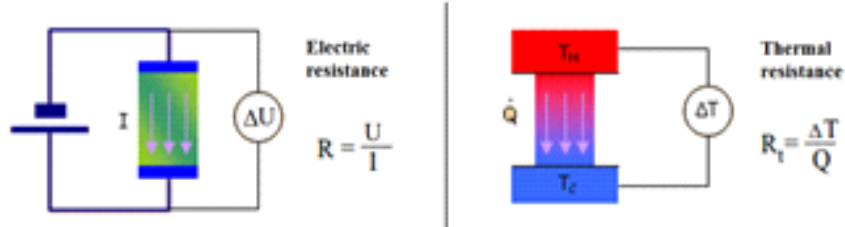


Fig. 4 – Equivalence between heat transfer and flow of electric current.

where:  $R$  – electric resistance,  $[\Omega]$ ;  $U$  – voltage,  $[V]$ ;  $I$  – current intensity,  $[A]$ ;  $R_t$  – thermal resistance,  $[K/W \cdot h]$ ;  $\Delta T$  – temperature difference,  $[K]$ ;  $Q$  – heat flux,  $[W \cdot h]$ .

## 2. Materials and Methods

A single Peltier cell was inserted between clothing layers in order to optimize heat transfer. The position of the Peltier cell is presented in Fig. 5.

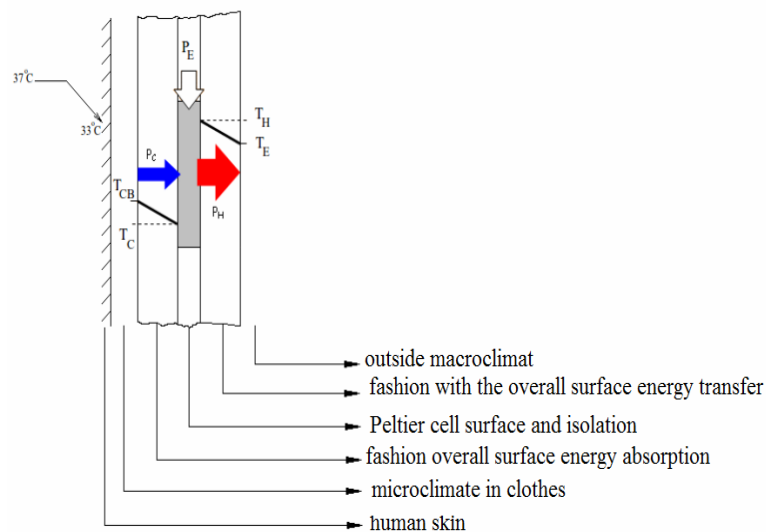


Fig. 5 – Positioning of Peltier cell between the clothing layers.

The ensemble of Peltier cell and clothing layers was subjected to different temperature differences ( $\Delta T$ ) and the transferred heat was determined between the layers. The heat was measured so that the experimental values could indicate how to minimise this heat exchange with the environment. The experiments were made in laboratory, varying conditions for temperature, humidity and air velocity.

Testing equipment is made from a thermally insulated enclosure that can be cooled to temperatures of  $-50^{\circ}\text{C}$  and also heated to temperatures above  $50^{\circ}\text{C}$ . The testing equipment is presented in Fig. 6.

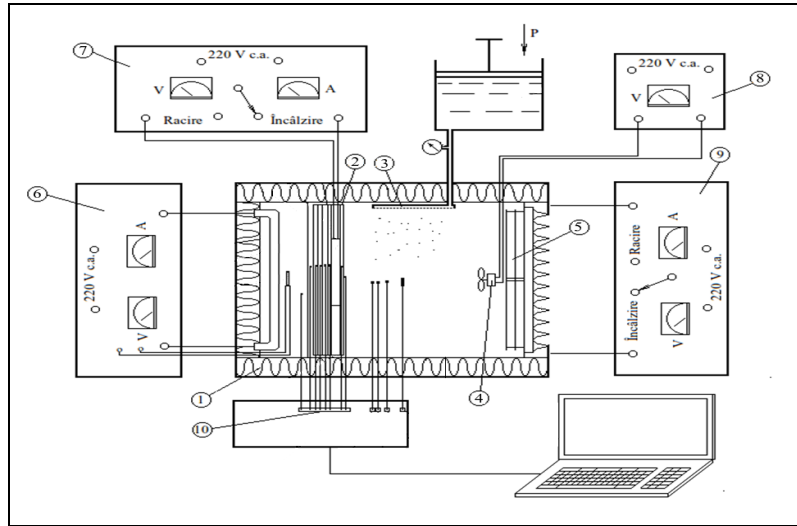


Fig. 6 – Block diagram of testing equipment:

- 1 – thermally insulated enclosure, 2 – layers of fabrics, 3 – installation of water spray, 4 – fan, 5 – Peltier cells cascaded, 6 – variable power source body simulation, 7 – variable power source, Peltier cell incorporated in the sample, 8 – power source variable fan, 9 – power source of microclimate, 10 – transducers and data acquisition interface.

The temperature difference between the inner and outer layer containing the Peltier cell was varied with  $10^{\circ}\text{C}$  increment  $-\Delta T = 10^{\circ}\text{C}$ ,  $\Delta T = 20^{\circ}\text{C}$  and  $\Delta T = 30^{\circ}\text{C}$ . External temperatures were produced with a set of Peltier cells and a radiator, while the internal conditions were generated with an electric resistance.

Physical strain was simulated by producing internal corresponding to different state conditions, presented in Table 1 (no strain to maximum strain). Thermal parameters between clothing layers were monitored using transducers.

### 3. Results and Discussions

Tables 1 presents the experimental results, showing the influence of the Peltier cells on the heat transferred from the body to the environment, in different strain conditions.

The experiments took into consideration two situations - when the external temperature decreased, the outside Peltier cells were used for heating, while when the external temperature increased, and the Peltier cells were used for cooling. Their energy contribution decreases with the physical effort made, as illustrated in Fig. 7.

If the outside temperature increases, energetic contribution Peltier cells which are used for cooling will grow proportionally to the physical effort made, as shown in Fig. 8.

**Table 1**  
*Peltier Cells for Energy Contribution to Maintain the Comfort*

Nr. crt.	State condition of the subject	Waste heat U [Kcal/h] or [W]	Text < 21°C			Text > 21°C		
			$\Delta T=10^{\circ}\text{C}$	$\Delta T=20^{\circ}\text{C}$	$\Delta T=30^{\circ}\text{C}$	$\Delta T=10^{\circ}\text{C}$	$\Delta T=20^{\circ}\text{C}$	$\Delta T=30^{\circ}\text{C}$
1	During sleep	40	35	47	64	6	10	14
2	Seated	50	33.5	46	62	7	10.4	15
3	Very easy work	75	30.5	43	58	8	10.7	16
4	Light work	100	28	39	54	8.5	11	18
5	Average work	150	22	32	44	9	13	19
6	Go at a walk	180	18	28	40	9.5	14	20.5
7	Hard work	300	7.5	14	24	10.5	16	26
8	Maximum effort	600				25	39	55

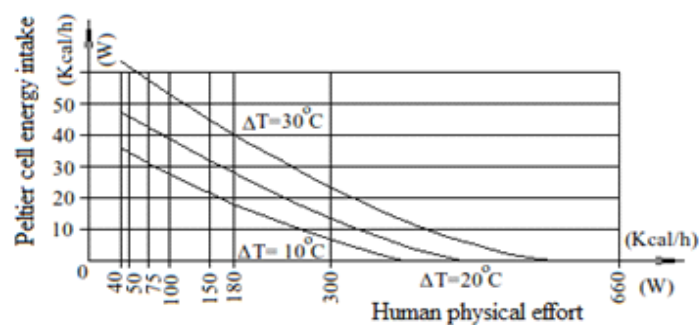


Fig. 7 – Diagram for energy consumption - outdoor ambient temperature below 21°C.

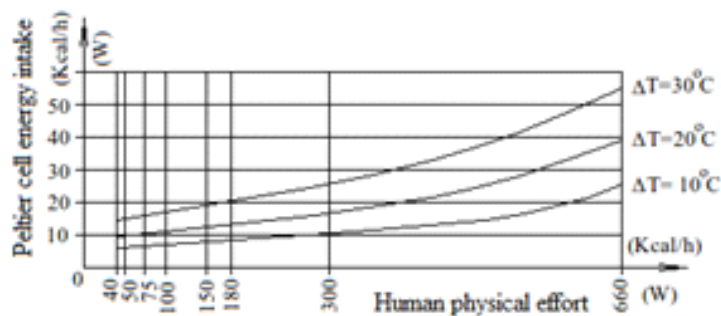


Fig. 8 – Diagram energy consumption - outdoor ambient temperature above 21°C.

Energy consumption for different states of physical effort were determined in reference to external temperature of 21°C as shown in Figs. 9 and 10.

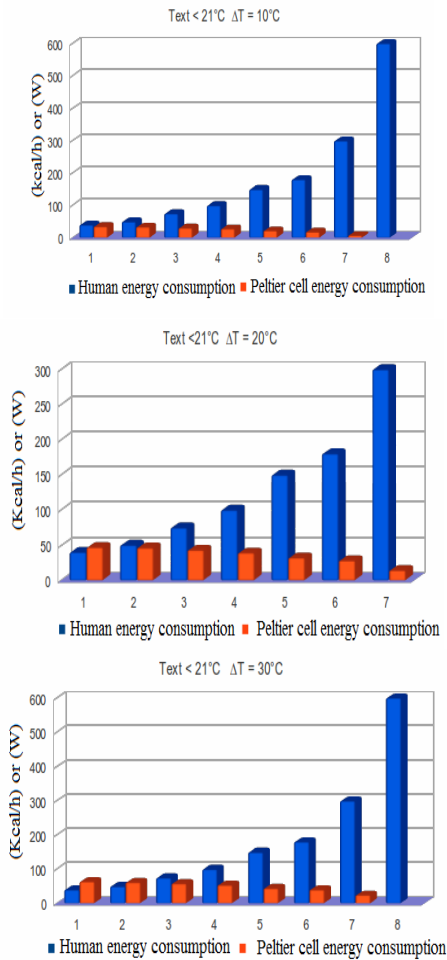


Fig. 9 – Comparative energy consumption for an external temperature below 21°C.

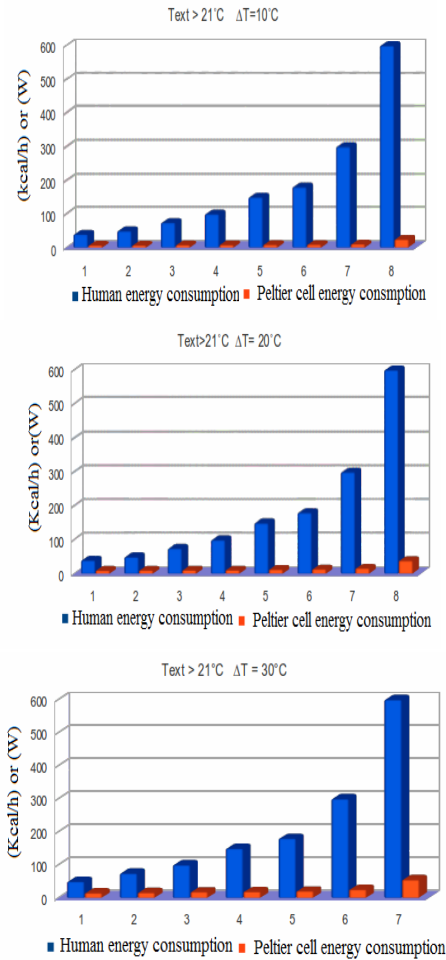


Fig. 10 – Comparative energy consumption for an external temperature above 21°C.

#### 4. Conclusions

The selection of Peltier cells with respect to the thermal transfer process consider, on one hand, the optimum difference between the body temperature (33°C) and environment temperature (the worst case scenario) and, on the other hand, the maximum thermal power.

As shown in experimental model represented in Fig. 3, the number of



Peltier cells depends on the number of layers included in the clothing ensemble. The experimental device designed for the study is actually a bioclimatic micro-room and will be used for further research, extending the options regarding the clothing structure, by changing the base material layer. This installation records the parameters influence on comfort indexes.

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## ROLUL CELULELOR PELTIER ÎN CONTROLUL MICROCLIMATULUI DIN PRODUSELE DE ÎMBRĂCĂMINTE

(Rezumat)

În prezent, problemele cu care se confruntă industria textilă sunt create de competiția puternică, una din soluțiile privind nivelul ridicat de competitivitate fiind identificarea unor nișe care să permită dezvoltarea unor noi oportunități. Multe companii textile au dezvoltat și îmbunătățit de-a lungul anilor produse și procese care în prezent nu mai sunt de ajuns. În acest context, după dezvoltarea textilelor tehnice și funcționale, cele mai recente dezvoltări se referă la textilele inteligente, un grup de produse cu numeroase aplicații inovative. Cercetări recente în acest domeniu au avut în

vedere atașarea unor sisteme electronice în produsele de îmbrăcăminte, pentru a proteja purtătorul de factorii climaterici extremi, precum temperaturile scăzute sau prea ridicate. Lucrarea pornește de la preocupările actuale din Uniunea Europeană privind cercetări originale referitoare la rolul celulelor Peltier în controlul schimburilor de căldură în produsele de îmbrăcăminte. Pornind de la prezentarea celulelor Peltier, lucrarea prezintă aportul energetic al acestor celule în ansamblul de îmbrăcăminte expuse temperaturilor extreme. Transferul energetic este exprimat ca un parametru de confort, și anume rezistența termică.

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## STUDY OF THE IMPACT BEHAVIOUR OF COMPOSITES MATERIALS WITH TEXTILE REINFORCEMENTS

BY

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**Abstract.** Composite materials are widely used because they have special properties. The advantage of composites is that monolithic materials have superior characteristics to a mass much smaller than them. In the paper has analyzed the behavior of composite materials with textile inserts and polyvinyl alcohol matrix.

**Key words:** composite, matrix, impact.

### 1. Introduction

Composite sandwich construction is widely used in different industries (ex. aerospace, marine, furniture, etc.) applications where there is need for lightweight structures with high in-plane and flexural stiffness (Vinson, 2001; Zenchert, 1995). These structures are known to be susceptible to impact damage by foreign objects (Cantwell & Morton, 1991; Daniel *et al.*, 2011). Sandwich composites are composed of layers with different compositions, which a thick layer of material filling. These structures are used to obtain compliance materials, which must simultaneously fulfill several conditions: mechanical and

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good chemical resistance, low specific weight, good thermal and acoustic insulation, impermeability to gases and water vapor.

The materials most used for sides are laminated composites and metals, and for core - metallic or nonmetallic honeycomb, foam cell, soft wood, etc. Typically, the faces are glued to the core with an adhesive. Intermediate phase that appears in any composite material, in the border areas of the base material and filler, significantly influence the overall performance and plays an important role in achieving effective material properties.

The nature and intensity of the interface connections are determined by the structure and surface characteristics of the reinforcement elements (roughness, surface area, porosity, the possible presence of active functional groups chemically) and certain structural characteristics of the matrix (chemical composition, macro-molecular conformation, etc.).

The interactions that determine the reinforcement-matrix interface fall into the following mechanisms: mechanical coupling or overlap micromechanics of the two components, coupling with the physical (van der Waals, dipole-dipole) or electrostatic attractions, covalent links, often involving the participation a coupling agent.

The coupling agents are used for improving the wetting of fibers by matrix (a phenomenon that increases the default area of contact between the two phases). To obtain optimum properties, especially in the mechanical strength is important how the mechanical stress on the surface spreads of matrix-fiber separation. A good stress transfer requires a large contact surface and a very good adhesion. A low adhesion between matrix and fibers leads to rapid propagation of cracks within the composite.

The impact of composite material with a body will generate crack propagation in the mass of material.

## 2. Materials and Methods

Textile products are used to obtain composite materials as reinforcement element (fibrous layers, fabrics, etc.). In the experiments has been study the possibility to achieve a composite material by overlapping layers of textile with variable number of different structures and consolidate them with glue - pressed at different values.

Samples were obtained using a matrix size of 175 x 126 mm, the fabric samples were used with the same dimensions. The characteristics of composites material are presented in Table 1.

The samples were obtained by using a mold with sizes of 175 x 126 mm, the woven fabric samples used has the same dimensions has been made between the manual mixture of PVA and wood chips, for samples CTRA40 and CTRA10, or fabric layer mixed with PVA for CTDB and CTD samples, as suggested in Fig. 1.

**Table 1**  
*The Characteristics of Composites Material*

No.	Composites coding	Fabric characteristics	Pressure [Mpa]	Ratio PAV/reinforcements layer
1	CTDB3	100% Flax warp Nm5/2, weft Nm 5/1 Pwarp = 72 ends/10 cm, Pweft = 62 ends/10 cm	0.065	5/1
2	CTDU3		0.065	3/1
3	CTRA40		0.065	5/1
4	CTRA10		0.065	4/1
5	CTD51		0.04	2/1
6	CTD52		0.196	2/1

The pressing was performed with a laboratory screw press for 10 min. In this period, the sample was dried at a temperature of 600C to prevent damage to the PAV matrix. The proportion of matrix and reinforcement material (fabric) varied between 2/1 and 5/1 depending on the type of composite material.

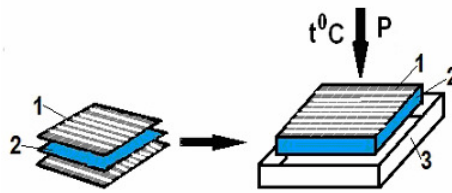


Fig. 2 – Method for obtaining the composite material.

Composite panels can be used as ambient objects so that the main request is to be subjected to the impact resistance. The phenomenon of impact depends on the speed with which the composite material body contact (low ( $v < 0.25$  km/s), medium ( $v = 0.25, \dots, 2$  km/s), ballistic ( $v = 2, \dots, 12$  km/s) and hyper speed ( $v > 12$  km/s)). In the study used to create velocity impact phenomenon was 0.00679 km/s, mass of impactor is 0.78 kg.

The impact force which determined the deformation of composite panel has been calculated using the scheme from Fig. 2.

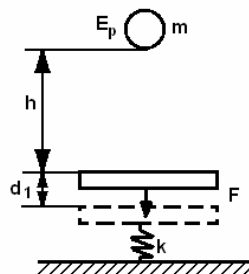


Fig. 2 – The principle of calculating impact force.

The hypothesis has been considered that the material deformation after impact deformation is similar to a resort. Relations of the calculation are:

1. Total energy:

$$E_p = G \cdot (h + d_1) \quad (1)$$

2. Mechanical work:

$$E_p = \int_0^{d_1} k \cdot x dx \quad (2)$$

3. Elastic constant of spring:

$$k = \frac{2 \cdot G \cdot h}{d_1^2} \quad (3)$$

4. Impact force:

$$F = k \cdot d_1 \quad (4)$$

Depending on the deformation produced by the impact was calculated body work needed for the deformation of composite material 1 mm, results are presented in the Table 2.

### 3. Results and Discussions

Composite samples were tested at the impact made by a small spherical body weight. Contact problem for finite thickness layers can be solved using numerical methods. The contact area is too small and we assumed the contact stresses of Hertzian type. The contact stresses take the shape of a semi-ellipse.



CTDB3



CTDU3

Fig. 3 – Composite material with 3 layers of fabrics.



CTRA40



CTRA10

Fig. 4 – Composite material with 2 layers of fabric and wood chips core.

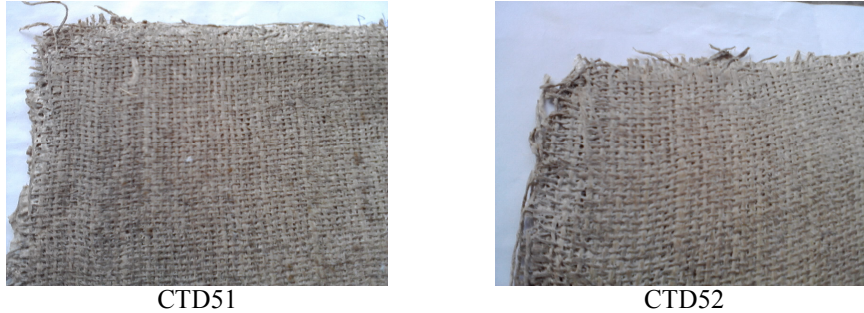


Fig. 5 – Composite material with 5 layers of woven and ratio 2/1.

The energy required for deformation of composite material 1 mm was determined by measuring the deformation depth produced by impact. The calculated values are presented in Table 2.

**Table 2**  
*The Characteristics of Composites Material*

Nr. crt.	Composites coding	Fabric characteristics	k [MPa]	F <sub>1mm</sub> [N]
1	CTDB3	100% Flax. Warp Nm 5/2. Weft Nm 5/1 Pu = 72 ends/10 cm. Pb = 62 ends/10 cm	6.509	6509.107
2	CTDU3		7.101	7100.552
3	CTRA40		3.377	3376.772
4	CTRA10		2.313	2312.747
5	CTD51		2.786	2786.275
6	CTD52		2.965	2965.034

Maximum force for deformation of the composite is recorded for CTDU3 sample, the composition has been realized by overlapping three thick fabrics, the warp direction, and a ratio 3/1 between the matrix and the support fabric and a medium pressure (0.065 MPa).

Minimum force is recorded for CTD51 and CTD52 samples to even if the pressure of achieving the composite was higher, because the fabric used as reinforcement material does not absorb enough matrix. During the forming process of the composite is found that much of the matrix is discharged from the mold. The remaining matrix is absorbed by the fabric and does not participate in strengthening the composite, Fig. 5.

#### 4. Conclusions

In this article is presented the effect of the impact of a spherical body on a composite sandwich. For the study, composite materials with PAV matrix and reinforcement material were produced. Based on information obtained from experiments, the influencing factors the ability of the composite deformation at low speed impact, are: the structure layers, the proportion of matrix/

reinforcement material, building pressure and temperature. In conclusion it can obtain composite plate, for reduced use of reusable materials in the textile industry. By adding a core of wood chips, one can replace some of the decorative tiles in furniture.

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### STUDIU ASUPRA COMPORTĂRII LA IMPACT AL MATERIALELOR COMPOZITE CU INSERȚII TEXTILE

(Rezumat)

Materialele compozite sunt folosite pe scară largă, deoarece acestea au proprietăți speciale. Avantajul este că materialele compozite, față de materialele monolitice, au caracteristici superioare la o masă mult mai mică decât acestea. În lucrare este analizat comportamentul materialelor compozite cu inserții textile și matrice din alcool polivinilic.



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## BIODEGRADABLE COMPOSITES WITH TEXTILE REINFORCEMENTS

BY

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**Abstract.** We presented a series of investigations on the structure and mechanical characteristics of biodegradable composite variants with insertions of textiles for furniture, decorative and ambient products. We investigated the properties of composite fiber inserts and bast fabrics reinforced with polyvinyl alcohol, protein material, polycaprolactone etc.

**Key words:** composites, biodegradable.

### 1. Introduction

Composite materials have evolved over time, due to interest in using waste from different manufacturing sectors, to diversify the raw materials to reduce material costs and improve performance of finished products.

In present, an important goal of obtaining and using composite materials is based on efforts to reduce environmental pollution by reducing energy consumption in manufacturing processes and by obtaining biodegradable products at the end of life.

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Composite materials are biodegradable and environmentally friendly and are considered a result of the rising interest appeared to improve quality of life in the context of sustainable development of society (Baillie, 2004). Technologies for composites (bio) degradable with textile inserts are included in the principles of sustainable development within the society. The intention is, on one hand, to develop the raw materials and on the other hand, to develop the products that will not adversely affect nature after completing the life cycle, thereby helping to reduce pollution.

Samples made within the frame of our research are layered (bio) degradable composites and are obtained from the consolidation of two or more layers of bast fabrics, reinforced through various polymer matrices. For structure symmetry of the laminated composite and for the uniformity of properties it is used an odd sequence of material layers oriented perpendicular to each other. Sometimes layered composites can be protected at surface with auxiliary layers such as foils, veneers, lacquers, other protective films etc.

In making the variants of composites with textile insertions it is important to establish the optimum parameters for obtaining the property of use and the aesthetic properties that enhance the quality of finished or semi-finished products (Iacob *et al.*, 2011; Mareş *et al.*, 2011a, Mareş *et al.*, 2011b). One of the priorities of the present research is the development of environmental products from (bio)-degradable composite materials, which have mechanical properties similar to conventional products and a lower level of costs compared to conventional products due to the use of textile materials from waste. In terms of structurally, (bio)-degradable composites are heterogeneous materials, made from at least two constituent phases with different properties. Composite material components are separated by well defined structural boundaries and material properties of composite components complement each other.

(Bio)degradable composites, obtained in the present research, may fall either in static structural materials (components that do not change their position or form during use) or non-structural materials (components that do not have to support heavy load during their operation, such as doors, decorative panels, wallpaper, etc). (Bio)-degradable products can have different destinations: furniture, decorative products, home utility products etc. A number of experimental composites are made from two or more layers, including at least one layer of textile insertion. Layer's strengthening is achieved through polymers that are biodegradable or potential (bio)-degradable. Products biodegradation is a complex process that is influenced by a number of factors, which assume that after completing the life cycle of products, these do not affect the natural environment in the degradation process. Thus, the end product life cycle is done slowly and completely, with the gentle return to the nature of the constituent materials of (bio)-degradable composites.

Biodegradation is influenced by many factors such as structure and chemical composition of constituent materials, the distribution of monomer units in the polymer chain, the type and concentration of particles that form the

matrix, molecular weight and its distribution in the structure of basic polymers matrix, structure and morphology of polymers (amorphous structure, semi-crystalline, micro), the presence of ionic groups in the composite matrix and at the textile insertion surface, composites manufacturing conditions, surface treatments of composite materials and products (varnish, etc), the mechanisms of hydrolysis, the mechanisms of hydrophilic, porosity of material in contact etc. The effect of all these variables are taken into account when evaluating existing products, namely the anticipation of the biodegradability of composites and environmental products in their design phase, by means of statistical analysis. Present research has strong character of novelty and originality, with the objective of achieving composite panels with textile inserts, with or without layers of wood, boards for furniture or other finished products to ambient destinations, for interior or exterior (doors, decorative panels, etc), as well as recreational objects (chess or backgammon boxes etc).

Obtaining of composites with textile inserts may involve recycling of manufacturing waste and waste from the textile industry and furniture industry, but also the raw materials. In addition, the development of environmental products from these materials may also have the aim to reduce consumption of wood in the manufacture of furniture and ambient decorative objects.

Due to their performance composites with textile inserts successfully replace traditional materials in various areas: the construction of automobiles, industrial equipment, building materials, furniture, biomaterials, packaging and consumer goods.

The advantages of polymer matrix composites results from three directions:

- superior physical-mechanical properties as a result of combining individual properties of material components (textile insertion and matrix);
- possibility to achieve a large number of parts, often with complex designs;
- moderate or low manufacturing cost.

The properties of polymer matrix and textile inserts, the proportion of both components and composite obtaining technology represent the most important factors which can control the material characteristics. Thus, there can be achieved low specific weight of textile composites, mechanical strength and high flexibility, good resistance to corrosion and soundproof properties (Schledjewski, 2006). If the constituent materials are chosen correctly, then these features may be added to a property, especially important after the period of use of the material, the biodegradability. Making a biodegradable composite means that at least one of the following conditions is fulfilled:

- use of biodegradable matrices;
- use fillers and reinforcing materials susceptible to degradation;
- the introduction of auxiliary constituents that trigger, maintain and stimulate biodegradation of stable components.

The method used to induce or increase the biodegradable character must

be selected so the material can compete in terms of physical and mechanical properties with conventional materials (Moise *et al.*, 2011). Like other physical and mechanical properties, biodegradability of composites can be adjusted on a relatively wide range of values, depending on the ratio of the two major components, matrix and reinforcement material/filler. In order to realize new composite with textile inserts, which meet the requirements of environmentally friendly products, there were tested materials with different compositions of the polymeric matrix and reinforcing material, composition set according to the protocol model shown in Fig. 1.

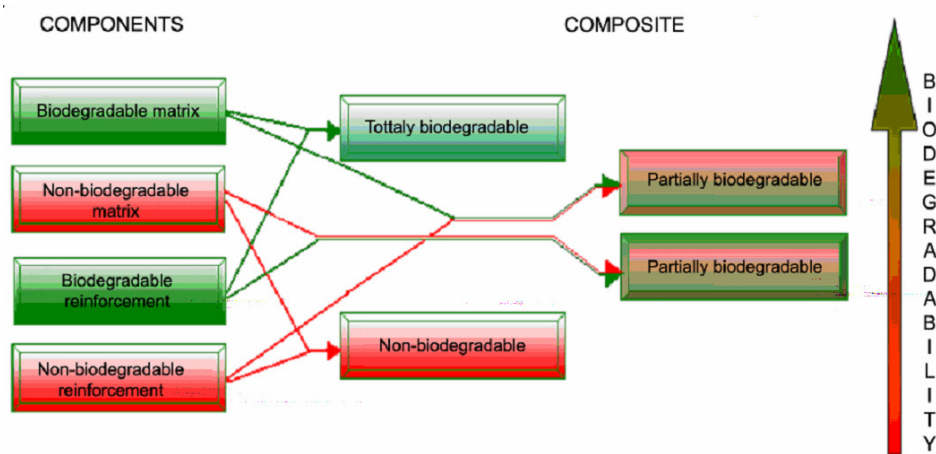


Fig. 1 – Principles of obtaining the biodegradability of composite materials.

Changing the matrix and the reinforcement material has allowed the obtaining of composites with biodegradation behavior which can cover a wide range of values.

In all types of composite reinforcement made materials are biodegradable and biodegradability regulation was achieved by using polymeric matrices different susceptibility to biodegradation. Composite variants primarily use biodegradable matrices of biodegradable natural polymers and synthetic polymers and additives in some cases non-biodegradable synthetic polymers.

## 2. Materials and Methods

The following constituents were used in the researches to obtain composites:

- jute fiber reinforcement material in others;
- flax and jute fabrics (variants V1, V2);
- biodegradable polymer matrix - vinyl polyhydric alcohols, protein material (animal glue);
- biodegradable - polystyrene / styrene.

For physical and mechanical characterization of composite materials obtained experimental determinations have been made to establish their following properties:

- composition, density and humidity (gravimetric method);
- mechanical properties (hardness, determined with Consistometer Hoppler, tensile strength, elongation at break, modulus of elasticity - the standard mechanical tests).

Composite experimental variants are presented in Table 1.

**Table 1**  
*Experimental Variants Composite for Fabric Reinforcements*

Experimental variants	Material description		Matrix composition/textile reinforcement, [%]
	Matrix	Reinforcement	
PAV-T0	PAV	Witness matrix PAV (without reinforcements)	100
PAV-T1		Fabric – 1 layer	70.59 / 29.41
PAV-T2		fabric – 2 layers	79.11 / 20.89
PS - T0	PS	Witness matrix PS (without reinforcements)	100
PS - T1		Fabric – 1 layer	71.13 / 28.87
PS - T2		fabric – 2 layers	74.53 / 25.47
PS - F		Jute fibers	86.13 / 13.69

### 3. Results and Discussions

Physical and mechanical properties of composites, flexibility and strength are important characteristics to the processing of composite materials to achieve the finished product and during its operation. Hardness was determined by Vickers method at room temperature, using as a cone of steel penetrator, while the use of force was between 60 sec and 30 sec, and the force applied on samples from 30 N and 60 N was. Averages are presented in Table 2.

**Table 2**  
*Structural Characteristics for Composites with Textile Reinforcements*

Experimental variants	Penetration [mm]		Hardness [N/cm <sup>2</sup> ]
	Determined values	Medium	
PAV-T0	1.04; 1.06; 1.01; 1.01	1.03	0.496
PAV-T1	0.85; 0.91; 0.88; 0.87; 0.89	0.88	0.643
PAV-T2	0.65; 0.70; 0.66; 0.64; 0.68	0.67	1.089
PS - T0	0.23; 0.27; 0.22; 0.24	0.24	3.874
PS - T1	0.24; 0.19; 0.20; 0.19; 0.22	0.21	4.462
PS - T2	0.14; 0.15; 0.14; 0.15; 0.15	0.15	6.122
PS - F	0.36; 0.40; 0.38; 0.41; 0.41	0.39	2.155

The analysis of experimental results shows that composites with textile inserts in the form of fabric are more resistant than the unfaced matrix polymer. On the other hand, composites with jute fiber inserts have a lower hardness compared to the hardness of the polymer matrix.

Matrices and biodegradable textile inserts were used in obtaining biodegradable composites with textile inserts, as shown in Table 3.

**Table 3**  
*Data Matrix Composites and Biodegradable Reinforcements*

Experimental variants	Matrix		Textile reinforcement fabrics V2 (layers)	Matrix composition/textile reinforcement, [%]
	Composition	Components report		
CC2	CA/A1/A2	77 / 10 / 13	2	64.7 / 35.3
CCP1	CA/A1/A3	80 / 10 / 10	4	64.0 / 36.0
CCP2	CA/A1/A4	88 / 10 / 2	4	67.1 / 32.9
CCP3	CA/A1/A4	82 / 10 / 8	4	68.9 / 31.1
CCP4	CA/A1/A2/A4	75 / 10 / 13 / 2	4	67.5 / 32.5
PCL1	PCL	–	4	55.49 / 44.51
PCL2	PCL/A4	75 / 25	4	60.22 / 39.78

Obs.: CA = Material protein (bone glue) PCL = Poly ( $\epsilon$ -caprolactone), A1 = Plasticizer; A2 = Material cellulosic fillers (wood dust), A3 = filler material (synthetic polymer biodegradable), A4 = cross linking agent (resin cetonform-aldehyde).

Table 4 shows mechanical characteristics of composite variants.

**Table 4**  
*Features of Matrix Composites Based on Protein Material*

Experimental variants	Tensile strength Rm [MPa]	Elongation Ar [%]	Elasticity modulus E [MPa]	
			Initial	Stabilized
CC2	8.10	4.22	895	112-126
CCP1	9.82	5.75	719	65-144
CCP2	7.12	3.15	624	108-205
CCP3	11.05	1.71	1210	375-431
CCP4	2.69	2.68	270	77-80

As can be seen from experimental data analysis, physical and mechanical characteristics depend on the nature and ratio of components and material technology that has been made. Thus, the mechanical strength of composites with matrix protein material depends not only on the nature of additives, but also their content. As expected, highly cross-linked matrix material has the highest values of tensile strength.

In terms of mechanical behavior, the composites investigated have very similar qualities, but the best properties of strength and stiffness (combined with ductility - elongation at break - lowest of all the variations of the composite)

were obtained for type protein matrix material that was treated with plasticizers and cross linking agents (CCP3 composite version). On the other hand, for composites with the highest value content of textile inserts (ranging from composite CCP1), achieved a relatively high mechanical strength combined with highest ductility (elongation at break higher, on average, over 3 times the category CCP3 composites, which showed the highest stiffness).

Depending on the pressure applied in the process of obtaining composite materials can be obtained with different density (loose or more compact structures in May). Experimental research shows that for composite variants analyzed in the application of pressure  $1500 \text{ N/cm}^2$  at a temperature of  $70^\circ\text{C}$  there is significant change in density composite material, as shown in Table 5. Variation in density composites, according to Table 5, shows that the composition of the matrix, the properties of a layered material can be controlled so as to obtain optimum characteristics with respect to its field of use.

**Table 5**  
*Density of Matrix Composites Based on Protein Material*

Experimental variants	Density $[\text{g/cm}^3]$		Material density modification [%]
	Before the pressing	After the pressing	
CC2	1.22	2.19	79.5
CCP1	0.94	1.65	75.5
CCP2	0.88	1.60	81.8
CCP3	0.74	1.08	45
CCP4	0.78	1.89	142.3

#### 4. Conclusions

Composite materials “environmentally compatible” must not be made entirely of natural resources. Even some synthetic materials can be valuable ecologically. Products made of composite materials (bio) degradable textile inserts have a number of indisputable advantages, but also some disadvantages. The main advantages are: low cost, renewable and recyclable resources, low specific gravity, and relatively good resistance to mechanical stress. In addition, during processing affect the technological equipment abrasion and corrosion, do not increase greenhouse gas  $\text{CO}_2$  type manufacturing processes, waste incineration, etc. do not generate. The main disadvantages of composite materials (bio) degradable are: absorb moisture, have low resistance to attack by microorganisms, it can withstand high temperatures are relatively uniform in terms of property, etc. The manufacturing of products made of composite materials with textile inserts is envisaged and following aspect: natural resources used in the composite structure are in principle renewable, but most of them are used for other purposes, leading to strong competition between different branches of industry. For example, the raw materials used to obtain the

textile inserts compete for their use of the textile, food, pulp, etc. biofuels industry. Because natural resources are limited, the manufacture of composites with textile inserts is used including waste production and recoverable and reusable materials. For each experimental variant, the selection of natural resources used is based on the composition and properties of finished products and that use of composite materials.

Sometimes it is necessary that compatibility exists between the polymer matrix textile inserts. Transformations and mechanical and chemical treatments that lead to changes in surface structure of fibers, yarns or fabrics used as inserts, are also necessary. These steps include processes and operations involving a fuel and energy consumption, emissions and certain other pollutants in water, air and soil. Getting involved should be added to the consumption matrix polymer (natural polymers, synthetic chemical materials added etc). All these aspects are taken into consideration to optimize energy consumption and limit environmental pollution in the process of obtaining composites. Consequently, the processes of making composite materials and finished products are gentler with the natural environment compared to conventional products.

Regarding the use by benefiting composite products (bio) degradable, it is necessary that they provide comparable satisfaction with the products produced using traditional materials. On the other hand, because in the structure of these composites are widely used natural polymers, which are less resistant to moisture, heat and microorganisms from traditional materials, it is possible that the product life cycle is relatively small, and their maintenance is more expensive.

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## COMPOZITE BIODEGRADABILE CU INSERȚII TEXTILE

(Rezumat)

Cercetările de față cuprind o serie de experimente privind structura și caracteristicile mecanice ale unor variante de materiale compozite biodegradabile cu structură multistrat, obținute din diverși polimeri și inserții textile sub formă de țesături. Pentru obținerea compozitelor biodegradabile s-au folosit polimeri biodegradabili sau parțial biodegradabili precum alcoolul polivinilic, polimeri de natură proteică, policaprolactona.



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## TEXTILES BEARING CYCLODEXTRINS - AN ALTERNATIVE TO CONVENTIONAL DRUG RELEASE SYSTEMS

BY

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**Abstract.** Textiles bearing cyclodextrins can be formed from conventional textile materials upon which cyclodextrin molecules have been fixed. The paper presents the method to attach the cyclodextrin molecules to the textile material which involves the simultaneous spinning and grafting of cyclodextrin into the fibres. The presence of the anchor groups in the cyclodextrin derivative allows the chemical fixation of cyclodextrin by the reaction of the fibre hydroxyl groups with the cyclodextrin chlorotriazinyl groups.

**Key words:** cyclodextrin, spinning, grafting, hemp fibres.

### 1. Introduction

Cyclodextrins are used in various areas, such as in medical applications, in textile or in agriculture, being included in diverse products like food or cosmetics (Del Valle, 2004; Hedges, 1998). The lack of toxicity and the water solubility determine the cyclodextrins, success in different applications from pharmaceuticals to cosmetics, to food, in chromatography and textile processes

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(Del Valle, 2004; Shafei *et al.*, 2010). The use of cyclodextrins in textile field is due to the large inclusion capacity of hydrophobic molecules. Cyclodextrins (CDs) are used as a reservoir of active substance, released slowly under the action of some stimulus. The using principle of these compounds is that of their grafting by some procedure on cellulose fabric. Due to their conformation, they are able to include in the hydrophobic interior a pharmaceutical product, which can be subsequently released in the presence of specific stimulus.

CDs are polysaccharides composed of 6 to 8 units ( $\alpha = 6$ ,  $\beta = 7$ ,  $\gamma = 8$ ) of D-glucose and they are formed during the enzymatic degradation of starch. D-glucose units covalently bind to the carbon atoms C1 and C4 (Buschmann *et al.*, 2001, Uyar *et al.*, 2009). Fig. 1 shows the structure of  $\beta$ -CD. Cyclodextrins are truncated cone shaped with hollow (Popa *et al.*, 1992), being described frequently as toroid macro cycles. The presence of hydroxyl groups at both bases of the truncated cone gives to CD a good solubility in aqueous solution. CD cavity is hydrophobic (coated with hydrogen atoms of groups C(3)H and C(5)H, and glycosidic oxygen atoms O(4)). As a result, the cavities form in solution a hydrophobic matrix in a hydrophilic environment (Cramer, 1954).

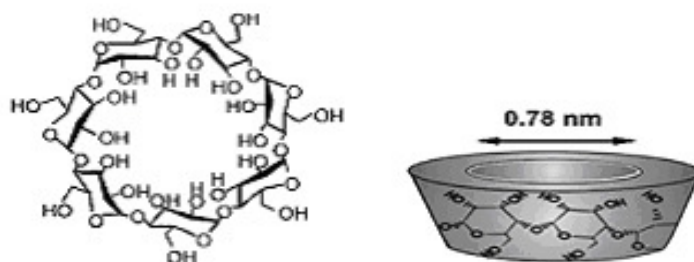


Fig. 1 – Structure and dimension of the inclusion cavity of the  $\beta$ -cyclodextrin (Buschmann *et al.*, 1998).

The cyclodextrin solubility in water of 1.85 g/100 mL  $H_2O$  and the cavity diameter of 7 Å may limit its applications (Szejtli, 1998). The form and the presence of internal hydrophobic cavities in CD cause special ability of these host species to include a wide variety of molecules and very different ions and to form stable inclusion complex (Lo Nostro *et al.*, 2003; Loftsson *et al.*, 2001). Cyclodextrins can include a variety of hydrophobic molecules or parts of them, inside their cavity, through non-covalent interactions, to form host - guest inclusion complex. CDs derivatives were developed to enhance the ability of inclusion and physico-chemical properties (Szejtli, 1998).

The key for cyclodextrins binding on different textile materials is the internal cavity. While the outer surface of the truncated cone is hydrophilic, the inner cavity of the CD is composed of hydrophobic parts. As a result, when CD aqueous solutions are mixed with polymers or hydrophobic organic molecules, it occurs a rapid process of inclusion (Becheri *et al.*, 2003). Cyclodextrins form

inclusion compounds with a wide range of guests, the main requirement being that these molecules fit dimensionally, all or at least partially, in the molecular cavity of the host. As a particularity, CD inclusion compounds can exist both in solution and in solid state (Szejtli, 1984). Since the CDs come into contact with the human skin, it is necessary to heed of their toxicological properties and their biocompatibility. Recent studies have shown no mutagenic, carcinogenic or teratogenic effects (Szejtli, 1988; Brewster, 1991). After the daily administration of 100-600 mg of  $\beta$ -CD to rats and dogs, for 6 months, there was no appearance of toxic symptoms (Duchene, 1998). CDs can be harmful to human body only in extremely high concentrations ([www.bioterapi.ro](http://www.bioterapi.ro)). The use of CD in textile industry has no pollutant effect on the wastewater. CDs are biodegradable and their biochemical oxygen consumption is similar to the other auxiliaries that are in use (Buschmann *et al.*, 1991).

It was shown that CDs finished fabrics through polycarboxylic acid are suitable in fragrance retention applications (Martel *et al.*, 2002). There were used fabrics of cotton, wool and polyester finished with the three types of cyclodextrin ( $\alpha$ ,  $\beta$ ,  $\gamma$ -CD) which were impregnated with six molecules of different fragrance, including menthol. It was noted that the intensity of fragrance materials treated with cyclodextrins are kept for one year, while for the untreated samples, retention of fragrance did not persist more than two weeks. Sustainability of the fragrance effect is directly dependent on the amount of cyclodextrin grafted on the fabric.

Other applications relate to towels which initiate the fragrance with hand skin friction with fabric or to fragrance developing bedding by direct contact between the skin and fabric which has CD groups grafted on the surface. Curtains finished with cyclodextrins can be used to improve the quality of air in rooms and offices (Buschmann *et al.*, 2001). Cyclodextrins "bin" can be used to capture unpleasant odour. Once filled it can be programmed (by setting release conditions: humidity, temperature, friction etc.) to release perfume or skin care components when it is in contact with moisture and then it can be filled again (CAVATEX). Resin finishing of cotton in the presence of cyclodextrin reduces the vapor pressure of organic molecules in sweat and thus it improves the odorant effect of textile materials (Ritter *et al.*, 2002). It is also possible to bind perfumes at textile finished with resin, with the aim of reducing the vapor pressure of these volatile perfumes. In this way, fragrances are released from textile materials for a longer period of time. Medical textiles with aromatherapeutic effect were produced in which the CD inclusion compounds are fixed on fabric by the traditional method. It was showed that the vapour pressure of fragrance in the fabric can last longer than 30 days (Wang, 2005). Permanent grafting of different chemicals causes specific performance and adds new properties to textile materials. Once chemically grafted on cellulosic substrates, compounds become supports for nanoparticles and active ingredients stabilization, being able to produce smart textiles and textile products with specific properties (Sawhney *et al.*, 2008).

## 2. Materials and Methods

A derivative of  $\beta$ -CD that is used in many applications is monochlorotriazinyl- $\beta$ -CD. Although it includes reactive halogen atoms, it does not cause mutagenicity, irritation and skin sensitization (Wacker-Chemie, 1996). In clinical trials conducted to establish the biocompatibility of textiles finished with this derivative were not detected skin irritations. Cyclodextrin derivative was introduced in 1996 as the first CD reagent deriva (Grigoriu, 2009; Denter *et al.*, 1997; Grechin *et al.*, 2007). Monochlorotriazinyl reactive groups of the derivative  $\beta$ -CD acts as an anchor group that reacts with hydroxyl groups of cellulose so that CD molecule is covalently bound on the surface of the fibre (Grigoriu, 2009).

In the experiments whose results are presented in this paper we used monochlorotriazinyl- $\beta$ -cyclodextrin product (MCT- $\beta$ -CD), under the trade name of CAVASOL<sup>®</sup> W7 MCT or CAVATEX W7 MCT, purchased from the WACKER Chemie AG Company. MCT- $\beta$ -CD is a reagent that can be applied on supports with nucleophilic groups such as -OH, -NHR or -SH (*e.g.* cellulosic fibres, protein fibres, polyamide, viscose, etc.), being an effective tool for surface modification of fibres by reactive chlorine atoms of triazinyl nucleus that can react with nucleophilic groups of cellulose fibres (-OH) (Fig. 2) (Grigoriu, 2009).

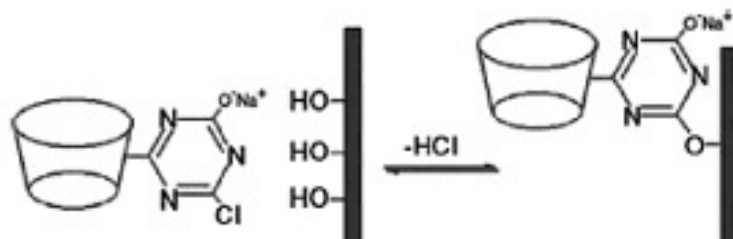


Fig. 2 – MCT- $\beta$ -CD grafting on cellulosic fibres (Grigoriu, 2009).

The performed experiments in this research have had as main objective, the analysis of the way in which the reactive cyclodextrin monochlorotriazinyl- $\beta$ -cyclodextrin (MCT- $\beta$ -CD) can be grafted on bast fibres simultaneously with the wet spinning. In the paper we present the analysis of the changes in the variation coefficients of the elongation of the yarns, which were spun from 100% hemp, according to the moistening time of the roving and the concentration of MCT- $\beta$ -CD in the tank of the wet spinning device. We have tested three types of yarns, yarns spun from bleached roving, yarns spun from alkaline treated roving and yarns spun from raw roving.

The mechanical characteristics of the processed yarns spun in different conditions (different moistening times and different concentration of MCT- $\beta$ -CD) where measured using a Tinius Olsen H5KT electronic dynamometer,

according to the ISO 2062 standardized methodology. Mathematical methods of dispersion analysis and regression and experimental programs were applied with the aim to assure optimum values of the dependent parameters.

### 3. Results and Discussions

One of the performed experiments intended to establish a multiple simultaneous correlation between the roving's moistening time in the tank of the wet spinning device ( $X_1$ ) and the concentration of MCT- $\beta$ -CD ( $X_2$ ) and the resultant characteristic, the variation coefficient of the elongation of the bleached hemp yarn which has a medium linear density of 72 tex ( $Y_1$ ). The regression equation resulted from testing the coefficients veracity and the accuracy of the model has the form.

$$Y_1 = 21.554 - 1.20717X_1 + 1.56648 X_2 + 3.97533 X_1^2 + 4.10533 X_2^2 + 6.48803 X_1X_2 \quad (1)$$

The answering surface in 3D space is presented in Fig. 3. It was confirmed the existence of a well-drawn surface with a minimum point, which corresponds to the minimizing of the resultant characteristic, the variation coefficient of the elongation of the bleached hemp yarn, fact which is evidently aimed. In Fig. 4 the outline curves of constant level of the resultant characteristic ( $Y_1$ ) are presented.

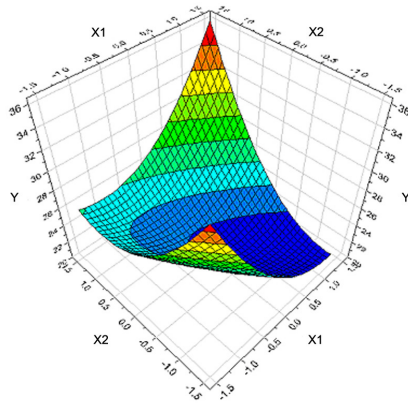


Fig. 3 – The answering surface in 3D space of the resultant characteristic ( $Y_1$ ), the variation coefficient of the elongation of the bleached hemp yarn, [%].

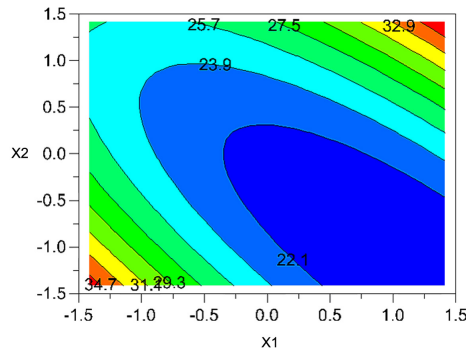


Fig. 4 – Outline curves of constant level of the resultant characteristic ( $Y_1$ ), the variation coefficient of the elongation of the bleached hemp yarn.

In Fig. 5 the variation of the resultant characteristic ( $Y_1$ ) depending on the soaking time of the roving (independent variable  $X_1$ ), for  $X_2 = X_{2c}$  is

presented. The same experiment was done in order to establish a multiple simultaneous correlation, between the roving's moistening time in the tank of the wet spinning device ( $X_1$ ) and the concentration of MCT- $\beta$ -CD ( $X_2$ ) and the resultant characteristic, the variation coefficient of the elongation of the alkaline treated hemp yarn which has a medium length density of 74 tex ( $Y_2$ ).

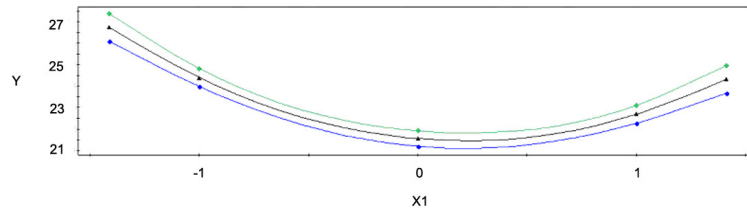


Fig. 5 – The variation of the resultant characteristic ( $Y_1$ ) depending on the soaking time of the bleached roving (independent variable  $X_1$ ), for  $X_2 = X_{2c}$ .

The regression equation resulted from testing the coefficients veracity and the accuracy of the model has the form:

$$Y_2 = 22.9499 - 0.0652588X_1 + 1.55897X_2 + 1.63019X_1^2 - 0.341775X_2^2 - 0.511675X_1X_2 \quad (2)$$

For the model expressed by the Eq. (2), the linear and square components were successively analyzed. The sign of the coefficient of the linear component for parameter  $X_1$  is negative; therefore the influence consists in the decreasing of the variation coefficient of the elongation. The effect is not major due to the low value of the coefficient. The presence of the square terms for both  $X_1$  and  $X_2$  has indicated the existence of a well outlined surface with a minimum point. There is a difference between the absolute values of the square terms' coefficients. The positive sign of the coefficient for variable  $X_1$  shows that the square term influences the resultant when increasing and the negative sign of the coefficient for the variable  $X_2$  shows the square term influences the resultant when decreasing. The coefficient of interaction corresponding to the term  $X_1X_2$  is present, meaning that the effect of the simultaneous variation of the two parameters is cumulative.

A third experiment aimed at establishing a multiple, simultaneous correlation between the soaking time of the scoured roving in the tank of the wet spinning machine, the concentration of MCT- $\beta$ -CD and the resultant characteristic, the variation coefficient of the elongation of the raw hemp yarn which has a medium linear density of 78 tex ( $Y_3$ ). The regression equation resulted after testing the correctness of coefficients and the adequacy of the mathematical model is the following.

$$Y_3 = 12.442 + 3.24125X_2 + 4.14388X_1^2 \quad (3)$$

The influence of the two parameters,  $X_1$  (the soaking time of the roving) and



$X_2$  (the concentration of MCT- $\beta$ -CD), on the variation coefficient of the elongation of the raw hemp yarn which has a medium linear density of 78 tex is rendered evident by the value of the coefficient of multiple correlation,  $R = 0.97579$ .

#### 4. Conclusions

The performed experiments have shown that the reactive cyclodextrin, monochlorotriazinyl- $\beta$ -cyclodextrin (MCT-  $\beta$ -CD) can be grafted on bast fibres simultaneously with the wet spinning.

There have been analyzed the changes in the variation coefficients of the elongation of the 100% hemp yarns, according to the moistening time of the roving ( $X_1$ ) and concentration of MCT- $\beta$ -CD in the tank of the wet spinning device ( $X_2$ ). The yarns were spun in from three variants of roving, bleached hemp roving, scoured hemp roving and raw roving.

From the technological point of view we were aiming to the minimizing of the variation coefficient of the yarn elongation. For the investigated experimental range, a minimum value of the variation coefficient of bleached yarn elongation of 20.35% was obtained for a soaking time of 40.88 sec and a concentration of 33.31 g/L MCT- $\beta$ -CD. For the other two types of yarns, spun from scoured and raw rovings, the passing time of the roving through the solution have to be in the interval of 22 and 24 sec, in order to obtain a minimum value of the variation coefficient of yarn elongation.

Once chemically grafted on hemp yarns, MCT-  $\beta$ -CD becomes a supports for nanoparticles and active ingredients of stabilization, being able to produce a nanometric finished textile material for medical applications by the inclusion of bioactive compounds (allantoin, eugenic acid, menthol) in the nanocavities of the reactive product.

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## TEXTILE PURTĂTOARE DE CICLODEXTRINE - O ALTERNATIVĂ LA SISTEMELE CONVENȚIONALE CU ELIBERARE DE MEDICAMENTE

(Rezumat)

Textilele purtătoare de ciclodextrine pot fi formate din materiale textile convenționale pe care au fost grefate moleculele de ciclodextrină. Lucrarea prezintă metoda de grefare a moleculelor de ciclodextrină pe materialul textil care presupune filarea simultană cu grefarea ciclodextrinei pe fibre. Prezența grupelor - ancorate în derivatul de ciclodextrina permite fixarea chimică a ciclodextrinelor prin reacția grupelor hidroxil ale fibrei cu grupele clorotriazinil ale ciclodextrinei.

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TEXTILE. PIELĂRIE

## DATABASE OF BIOCIDES USED IN TEXTILE AND LEATHER INDUSTRY

BY

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**Abstract.** The perfect technique to prevent the microbial degradation and to sterilize materials from textile and leather industry is to use chemicals with antimicrobial properties. These substances are called biocides and are designed to combat, neutralize and exert an effect of control on any harmful organism by chemical or biological means.

Besides the beneficial properties, biocides can manifest harmful effects on living organisms and the environment, reason for which is essential to know and have access to information about these produce before to use and sell them.

In order to minimize negative effects and make possible to obtain various information about the most used biocides in textiles and leather industry, we created and designed a database with online access.

The database allows the rapid processing and sorting of information, their extraction on various search criteria: chemical and commercial names, chemical and structural formulae, code numbers (CAS, EINECS, etc.), physical-chemical, toxicological and ecotoxicological properties, information on antimicrobial action on bacteria, yeasts, molds and the field of applicability, in which we specify the type of materials for which the biocide is used. Information is collected from various sources of references and has been structured under the

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form of files which can be downloaded from the database in pdf format or can be extracted according to certain specific fields.

**Key words:** database, biocides, textile, leather.

## 1. Introduction

The perfect technique to prevent the microbial degradation and to sterilize materials in the textile and leather industry is to use chemicals with antimicrobial properties. These substances are called biocides and are designed to combat, neutralize and exert an effective control on any harmful organism by chemical or biological means.

In the textile and leather industry, the biocides used in a preponderant way are organostannic compounds, chlorinated phenolic compounds, quaternary ammonium derivatives, halogenated organic compounds, surfactants, complexing agents etc. (Fig. 1) (Lacasse & Baumann, 2004).

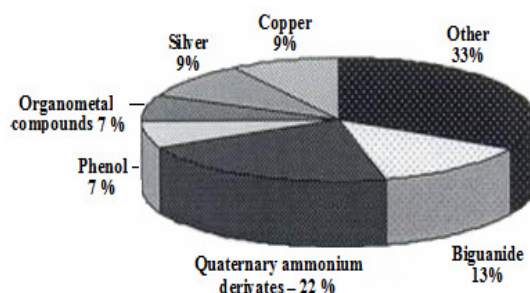


Fig. 1 – The main active components of biocides used in textile.

In the textile industry, the materials prone to attack of various microorganisms are plant fibers such as cotton, linen, jute and hemp, synthetic or artificial fibers derived from cellulose and therefore these must be treated antimicrobially. But, there are fibers made of synthetic polymers (polyamides, polyesters, polyethylene, polypropylene) that are naturally very resistant to the attack of microorganisms and do not require antimicrobial treatment (Carr, 1995).

Biocides used in textile and leather industry offer a wide range of benefits:

- Are effective against many organisms: bacteria, fungi, microfungi, algae, that destroy the textile and leather materials;
- Maintain the materials in good conditions of hygiene;
- Eliminate problems related to stains, odors;
- Protects the leather and textile materials against biodeterioration, ensuring their conservation;
- Avoid the decomposition of the textile and leather materials;
- Prevent the spread and transfer of pathogens;
- Provide a long life for the product;

- Do not damage the fabric properties;
- Are resistant to sunlight and leaching from the fabric;
- Are kept on textiles for a long time even after repeated washing and cleaning;
- Are not very expensive and are easy to use.

Besides the beneficial properties, biocides may manifest unwanted effects on the environment, living organisms, that's why it is necessary to have access to all information about these products.

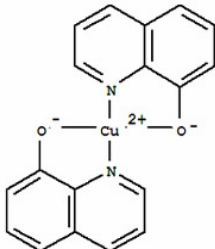
## 2. Experimental Work

In order to minimize the negative effects and obtain various information about most biocides used in textile and leather industry, we created and designed a database with online access. The database has been created in SQL language (Structured Query Language) and developed in MySQL. The design and interface of the web application have been realized using the language PHP (Hypertext Preprocessor) (Elmasri & Navathe, 2007; Doroş, 2010).


The information in the database on biocides were structured in the form of files that can be downloaded in .pdf format (Table 1) or can be processed, sorted and quickly extracted by different specific fields:

- chemical and commercial names;
- chemical and structural formulae;
- code numbers (CAS, EINECS, etc.);
- (eco)toxicological properties;
- information on the antimicrobial action on bacteria, fungi;
- the domain of application.

**Table 1**  
*File of 8-Hydroxyquinoline, Copper(II) Salt*

Substance Class	Derivatives of pyridine
Chemical name	8-hydroxyquinoline, copper(II) salt
Other names	Copper oxine; Oxine-Cu; 8-Hydroxyquinoline copper complex; Cupric 8-hydroxyquinolate; Copper 8-quinolinolate
Molecular Formula	$C_{18}H_{12}CuN_2O_2$
Structural Formula	
Codes	CAS-No.: 10380-28-6; EINECS-No.: 233-841-9
Chemical and physical properties	

**Table 1**  
*Continuation*

Substance Class	Derivatives of pyridine
Molecular mass, [g/mol]	351. 85
Appearance	Yellowish green crystal powder
Purity	99% min.
Density, [g/cm <sup>3</sup> ]	1.68
Boiling point, [°C]	267 at 760 mmHg
Melting point, [°C]	240
Flash Point, [°C]	143.1
Stability	Stable between pH 3 and 12, stable to light, heat resistant to 270°C
Solubility, [g/L]	insoluble in water, moderately soluble in xylene, trichloromethane
Toxicity/Ecotoxicity	
Copper oxine is irritating to eyes, respiratory system and skin.	
LD50 oral	4500 mg/kg rat
Symbols of danger alert	 <b>Xi: Irritant</b>
Antimicrobial efficacy	
Copper oxine is more effective than oxine and more active than zinc chelate. Spectrum of activity covering bacteria oxinei copper, yeast, fungi and algae.	
Applications in: textile and leather, wood, paper industries, agricultural chemicals.	
<p>Biological properties of copper compounds take important role in agriculture as a fungicide and as a biocide in anticorrosive paints for ship products and wood preservation. Copper oxine is a good antimycotic agent and is used in wood, plastic-rubber, leather, paper, used in formulating antiseptics, deodorants, antiperspirants, fungicides, pesticides, dyestuff etc. A very low level of copper is toxic to fungi and algae. Copper ions inhibit the metabolism of fungi when react with enzymes containing sulphur from plants. Copper compounds form a protective barrier on the plant surface and thereby prevent fungi from entering in the plant host. Because of its high efficacy against wood staining and mould-producing fungi, copper 8-quinolinolate is recommended for the protection of paper, textile, and plastic material.</p>	

Database on biocides used in the textile and leather industry is one relational and is based on the conceptual model of the application of analysis of biocides represented by Entity-Relationship diagram in Fig. 2 (Abb *et al.*, 2010; Roman *et al.*, 2011).

The symbols used in Entity-Relationship diagram have the following meanings:

- In rectangles are represented the substances that are included in the database as entities;
- With “#” are represented the specific attributes - a primary key, PK (plain): id\_toxicity, id\_code, id\_class etc.
- With “\*” are represented a series of mandatory attributes, eg name, file.pdf etc.
- With a circle “o” are represented a number of optional attributes for example: the details of a particular class of substances, the amount of substance that can have lethal toxic effect on an adult animal (LD<sub>50</sub>), Table 2 (Dănilă *et al.*, 1984).

**Table 2**  
*Hodge and Sterner Toxicity Scale*

The degree of toxicity	LD <sub>50</sub> oral for rats
highly toxic	under 1 mg/kg
very toxic	1 - 50 mg/kg
moderately toxic	50 - 500 mg/kg
low toxicity	500 - 5 000 mg/kg
practically non-toxic	5 000 - 15 000 mg/kg
relatively non-toxic	over 15 000 mg/kg

### 3. Conclusions

A database on biocides used in textile and leather industry represents an important tool in the evolution of technology, in the scientific research and for practical applications of protection against biodeterioration. It allows researchers, students etc. to have access to a wide range of information so that the documentation process and assimilation of new knowledge to be more productive and creative.

As the progress of science and technology determines the pace a society develops with, this database represents the starting point in grounding knowledge about biocides intended for the protection of textile and leather materials against biodeterioration.

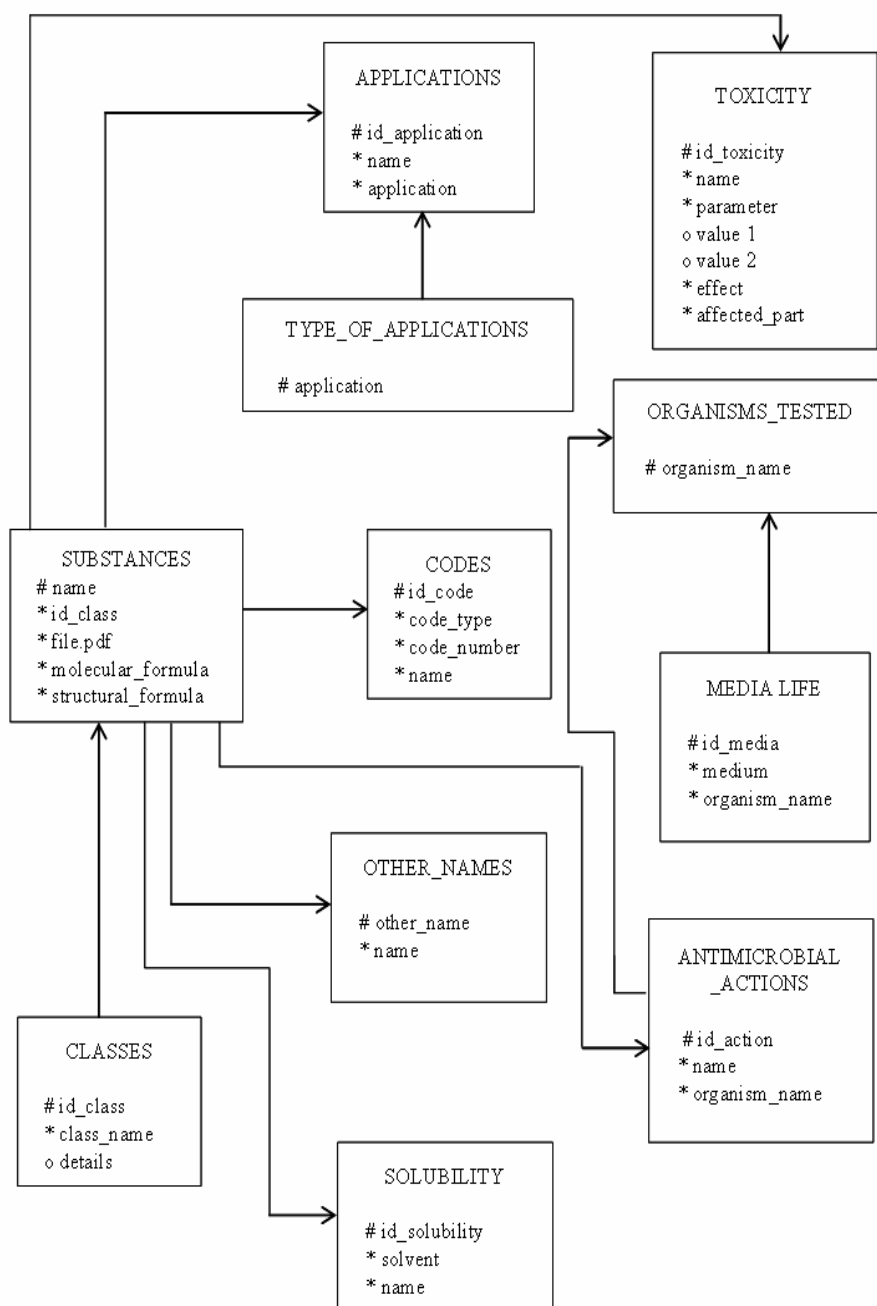


Fig. 2 – Entity Relationship Diagram.



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BAZA DE DATE PRIVIND BIOCIIZI UTILIZAȚI ÎN INDUSTRIA  
TEXTILĂ ȘI DE PIELĂRIE

(Rezumat)

Tehnica perfectă de a preveni degradarea microbiană și de a steriliza materialele din industria textilă și de pielărie este de a utiliza substanțe chimice cu proprietăți antimicrobiene. Aceste substanțe se numesc biocizi și au rolul de a combate, neutraliza și exercita un efect de control asupra oricărui organism dăunător, prin mijloace chimice sau biologice.

Pe lângă proprietățile benefice, biocizii pot manifesta efecte nocive asupra organismelor vii și a mediului înconjurător, motiv pentru care este esențial să știm și să avem acces la informațiile despre aceste produse înainte de a le folosi și comercializa.

Din dorința de a minimiza efectele negative și de a face posibilă obținerea de informații diverse despre biocizii cei mai utilizați din industria textilă și de pielărie am creat și proiectat o bază de date cu acces online.

Baza de date permite prelucrarea și sortarea rapidă a informațiilor, extragerea lor pe diferite criterii de căutare: denumire chimică și comercială, formulă chimică și structurală, numere de cod (CAS, EINECS etc), proprietăți fizico-chimice, toxicologice și ecotoxicologice; informații cu privire la acțiunea antimicrobiană asupra bacteriilor, levurilor, ciupercilor și domeniul de aplicabilitate, în care specificăm tipul de materiale pentru care biocidul este folosit. Informațiile sunt colectate din diverse surse bibliografice și au fost structurate sub forma unor fișe care pot fi descărcate din baza de date în format pdf. sau după anumite câmpuri specifice.

