

**NANOCOMPOSITE WOOD FIBROUS MATERIALS FOR ELECTROMAGNETIC
WAVE PROTECTION**

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Abstract

Recently, wood polymer nanocomposite materials with improved electroconductivity, microwave absorption capacity and with high quantity of wood component represent a great interest because of their unique properties and possibilities for obtaining of different technical articles and applications.

An original method for obtaining of copper – sulfide modified nanocomposite wood-fibrous materials has been developed. A mathematic modulation and optimization of the process is conducted using of carbamydeformaldehyde resin as bonding agent. Copper–sulfide modified nanocomposite wood-fibrous materials with high physical-mechanical indexes, high quantity of wood component and improved electroconductivity for electromagnetic wave protection are obtained.

Key words: *copper – sulfide wood nanocomposites, carbamydeformaldehyde resin, mathematic modulation and optimization, electroconductivity*

1. INTRODUCTION

Presently, metalcontaining nanomaterials and particularly the polymer nanocomposites are of great interest because of their unique physical and chemical properties and possibilities for different application (Wang, 1998).

Composites with improved characteristics – strength, thermal resistance and other specific properties – are produced by addition of mineral fillers, metals and fibers to polymers. In recent years a great progress is achieved in this direction by means of creating nanocomposite materials of better and novel properties with low filler percentage and many possibilities for new applications (Lio, 1993).

There is a large variety of new hybrid and metal polymer nanocomposite materials in which a strong interaction of the nanoparticles and the polymer chains is observed on the level of chemical bonding (Pomagailo, 2001)

The nanocomposites provide possibility to manufacture products of super conductivity properties. The researches in this line are very promising and could result in the development of new materials with unique properties (Wang, 2000, Wu 1989).

Great interest presents the researches related to production, characterization and adoption of electro-conductive polymers and fibrous materials with microwave absorption properties: Method for fiber modification by electro-conductive pigment on nano-cupro-sulphide polymer dispersion base and new electro-conductive polyacrylonitrile pigments with microwave absorption properties produced by chemical modification of deposition and coordinative bonding of copper complexes to polymer substance for microwave absorbers (Lekova, 1998).

During the past years there is an intensification of the researches related to the methods for copper sulphide electrolytic deposition on polyacrylonitrile in presence of complexing agents: with triethanolamine (Yen-Hung Chen, 2009) and with ethylenediamine tetra acetic acid (Yen-Hung Chen, 2010).

The researches show the actuality, importance and necessity to carry on thorough researches concerning production of new type of metal containing nano composite polymer materials with improved electrical conductivity and microwave adsorption capability for application in engineering and household as products for electromagnetic wave protection. In this respect, wood polymer plates present particular interest as construction material to be used in the furniture industry, building and packaging.

The purpose of the present research is the development of a continuous one-stage method for production of copper-sulphide wood-polymer nanocomposite materials and this method to be adoptable to the existing equipment and technology for wood fiber plates (WFP) production in LESOPLAST Inc. – Troyan.

The researches comprise:

- Experiments on development of appropriate and optimal method for wood fiber material modification to obtain copper-sulphide lignocellulose nanocomposites;
- Experiments on defining the technological stage and adequate equipment to obtain copper-sulphide wood fiber nanocomposite plate materials;
- modeling and optimization of the developed method in respect of compositions, proportion of the individual components and parameters by using of carbamideformaldehyde resin as bonding agent for the achievement of improved electrical conductivity (corresponding electromagnetic wave absorption), high content of wood component and with retaining the level of the initial standardized WFP characteristics.

2. MATERIALS AND METHOD

In present investigations are used

- wood fibers – high gain fibrous material (HFM), obtained in Lesoplast AD, Troyan by hydro heat treatment of wood chops for wood plates;
- carbamyl formaldehyde resin – content 0,1 % towards the fibers as binder of wood fibrous composites.

Before mathematic modeling and optimization of process for obtaining of copper–sulfide modified nanocomposite wood-fibrous materials, preliminary experiments for establishing of suitable technological modification stage have been carried out.

Experiments for establishing of technological stage

. * The obtained results on the stage, consists by treatment of technological chops in steaming chamber, showed that the modification process do not pass uniformly in the depth of wood and stayed on the surface or in the solution. This unevenness of modified and unmodified fibers in wood sample carpet will bring to bad results regarding electroconductivity. Therefore, the stage with steaming treatment of technological chops is not suitable as a modification method.

. * On the gluing stage of wood fibrous mass, the modification process is carries out in the volume of wood fibrous mass by homogenization $\text{CuSO}_4 \cdot 5\text{H}_2\text{O} : \text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ solution in proportion 1:2 with

fibrous suspension. Obtained samples do not possess good results regarding electro conductivity. This may be explained by considerable loss of modified reagents.

These previous results showed the advisability for conducting the treatment of wood fibers by surface modification with mixture of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}:\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ on the stages - gluing and formation of wood fibrous carpet.

Experiments for establishing of the treatment conditions and starting parameters

Previous experiments for obtaining of copper-sulfide wood fibrous composite specimens with carbamyldeformaldehyde resin as bonding agent with purpose an establishing suitable conditions and parameters have been conducted:

1. Constant parameters

- 1.1. Mole proportion $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}:\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O} = 1:1,75$;
- 1.2. Percent of modification agents in quantity 30 % towards absolute dry mass;
- 1.3. Duration - 10 minutes
- 1.4. Temperature for pressing – 178° C;
- 1.5. Duration of staying before pressing – 10 minutes.

2. Description of samples

The samples are surfacing treated with reagents before pressing.

- 2.1. Sample № 0 - wood fibrous mass;
- 2.2. Sample № 0' - wood fibrous mass only with modification reagents;
- 2.3. Sample № 1 - wood fibrous mass with modification reagents and 1% carbamyldeformaldehyde resin and 1 % paraffin emulsion towards absolute dry wood fibrous mass;
- 2.4. Sample № 2 - wood fibrous mass with modification reagents and 2% carbamyldeformaldehyde resin and 1 % paraffin emulsion towards absolute dry wood fibrous mass;
- 2.5. Sample № 3 - wood fibrous mass with modification reagents and 3% carbamyldeformaldehyde resin and 1 % paraffin emulsion towards absolute dry wood fibrous mass;

The specimens produced are characterized in respect of thickness, bending strength, swelling in water volume weight and volume electrical resistance.

The results (Table 1) show that:

- the reagent mixture improves the indexes of specimens: improving strength, decreasing swelling and lower electrical resistance (improved electro conductivity).
- the carbamyldeformaldehyde resin additive (CFR)- 1 to 3 % as bonding agent has an minimal effect on the indexes.

Table1. Indexes of composite specimens

N ^o	Thickness, mm	Bending strength, MPa	Swelling, %	Volume weight, kg/m ³	Volume résistance, Ω
0	2,7	28,76	74,04	1012	1,8142E ⁺¹¹
0'	2,6	29,91	50,00	1004	1,2141E ⁺⁹
1	2,7	28,79	48,15	1035	6,5312E ⁺⁹
2	2,7	28,52	48,15	974	2.1870E ⁺⁹
3	2,8	30,20	48,00	972	2,7632E ⁺⁹

On the basis of experimental results are established conditions and starting parameters, as following:

1. The quantity of reagent mixture $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}:\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ towards absolute dry wood fibrous mass – 25%, 35%, 45 % (step 10) - x_1
2. Mole proportion $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}:\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ - 1:1, 1:1,5, 1:2 (step 0,5) - x_2
3. Duration of staying before pressing - 7, 12, 17 minutes (step 5)- x_3

Constant parameters:

1. Temperature of pressing - $170 \div 175^\circ \text{C}$;
2. Duration of staying before pressing – 10 minutes.

The experiments are carried out with wood fibrous mass from production of Lesoplast AD- Troyan with fowling indexes: concentration – 3,75 %; grinding degree – 22DS; 15 % CFR and 10 % paraffin emulsion. The quantity of gluing substance is 1 % towards absolute dry wood fibrous mass. The experiments are conducted with specimens- sixes 30x30 cm. by surfacing treatment with the mixture of reagents. The obtained samples have 15 % wood fibrous material. The fowling process is pressing- 170°C , duration - 10 minutes.

The specimens are characterized in respect of electrical conductivity (volume electrical resistance by apparatus DC- MILLI- PICO- METER MV 40, VE KOMBINT PRACITRONIC, Dresden, as well as the bending strength by apparatus ZWICK Z0,5 Zwick & Co GmbH, standart EN 622-1. The swelling is determined according EN 622-2.

3. ANALYSIS AND DISCUSSION

In the present work, the modeling and optimization of the modification process of the wood nanocomposites are conducted with the fowling main performance characteristics (outputs):

- y_1 – bending strength on face, [MPa];
- y_2 – bending strength on sieve, [MPa];
- y_3 – swelling in water, [%];
- y_4 – logarithm of the volume resistivity, [Ω].

These outputs are depended by the influence of the three process parameters (factors): x_1 - % modification additive of two reagents towards absolute dry wood fibrous (WF) mass, x_2 - mole proportion $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}:\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$; x_3 - duration of staying before pressing [min.]. Changed limitations are: $25 \leq x_1 \leq 45$, $1 \leq x_2 \leq 2$ and $7 \leq x_3 \leq 17$. The weight of absolute dry WF mass at experiment is constantly, but the additives have different percent and mole proportions. By this reason WF mass and additives do not observed as three mutually dependent variable quantities.

The aim of the optimization problem is to find a great number of suitable combinations of two additives (factors x_1 and x_2), as a duration of staying (x_3) at which the bending strength (target function y_1 and y_2) is over 30 MPa and swelling under 37%.

The decision of the optimization problem

For the decision of so formulating task is selected a model based approach, especially regression model, which describes the relations of the outputs y_j , $j=1,2,3,4$ and factors x_1 , x_2 и x_3 . (11,12). Since in examining sphere of changing of factors there are not unsteady changing of initial quantities, the mathematical model must search in the class of polynomial models:

$$\hat{y}_{ju} = \sum_{i=1}^k b_i f_{iu} . \quad (1)$$

Where, $u = 1, 2, \dots, N$ is number of attempt, $i = 1, 2, \dots, k$ - numbers of coefficients in the model. $\mathbf{b} = [b_1, b_2, \dots, b_k]^T$ - k -verse vector of valuations of regression coefficients. f_{iu} - random, previous given of factors (x_1 , x_2 и x_3), $\hat{y}_{ju}$ - predict value of aim function. This function is received by replacement of values of f_{iu} for every experiment.

The organization of the experiment is very significant for the effectiveness of the investigation. In this case the experiment is conducted using optimal composition plan with 14 attempts ($N=14$ experiments).

In present work models of complete second order with 10 adequate for 4-th target functions coefficients are considered. In Table 2 the obtained regression coefficients are given. In Table 3 the obtained results from statistic analysis of the models are summarized. Q_{jr} - Means the residual sum of quadratics of corresponding models.

$$Q_{jr} = \sum_{u=1}^N (y_{ju} - \hat{y}_{ju})^2, \quad j = 1 \div 4 \quad (2)$$

Where, $\hat{y}_{ju}$ - is the predict value on j - model in u -attempt.

This predicted value characterizes the dispersion of experimental values of initial y_{ju} near to the predicted model values. The reason of the showing may be due of:

- a) non adequate of the model ;
- b) an influence of accident error.

The model may be suitable for using (prediction and optimization) if the influence of non adequate is commensurably or lower in comparison with the influence of accident error. This may be ascertained by the comparison of dispersions. The valuation of dispersion, connected with Q_{jr} is calculated.

$$s_{jr}^2 = Q_{jr} / \nu_{jr} , \quad (3)$$

Where $\nu_{jr} = N - k$ is the number of degrees of freedom. It is compared with the valuation of dispersion of random error S_{je}^2 . For valuation of the adequate of model [1] the repeated experiments n_j , $j=1,2,3,4$ at the same regime in centre of the plan $x_1=35$, $x_2=1.5$ и $x_3=12$ have been conducted. These experiments give the possibility to estimate the dispersion of the random error S_{je}^2 :

Table 2. Regression coefficients

Included function, f _{iu}	Coefficients in the model for y1	Coefficients in the model for y2	Coefficients in the model for y3	Coefficients in the model for y4
Free article	34.9091	16.7166	25.8138	10.4291
x_1	-1.3815	-1.1265	-1.2550	-0.0766
	2.0395	3.8955	2.9690	-0.1191
x_2	1.0430	0.1590	0.6000	0.2101
x_3	1.5494	-0.7319	0.4075	-0.0674
	0.9106	1.6544	1.2825	0.0181
$x_1 * x_2$	-0.5894	-1.2644	-0.9275	0.1218
$x_1 * x_3$	-3.0791	0.0684	-1.5087	0.0824
	-1.8091	3.8184	1.0113	0.2329
$x_2 * x_3$	-1.5466	0.6459	-0.4537	0.1613
x_1^2				
x_2^2				
x_3^2				

$$s_{je}^2 = \frac{1}{n_j - 1} \sum_{i=1}^{n_j} (y_{ji} - \bar{y}_j)^2, \quad j = 1 \div 4,$$

Where n_j is the number of observations, $\bar{y}_j$ - arithmetical mean. Number of freedom degrees is $\nu_{je} = n_j - 1$.

Table3. Level of significance

Model	$Q_r (v_r=4)$	s_e^2	v_e	F-calculated	F _{kp} ($\alpha = 0.05$)
Y1	13.04	1.76	5	1.85	5.19
Y2	21.52	6.97	5	1.29	6.26
Y3	10.51	7.49	2	2.85	6.94
Y4	0.273	0.402	17	1.70	2.96

The comparison is founded on the control of zero hypothesis $H_0: s_r^2 = s_e^2$, by F- criteria of Fisher. The Table3 shows the level of significance.

The searching of optimal regimes are realized by the method of scanning with constant step, which represents 5 % of observed field of changing of each factors.

Optimal regimes depending on special requirements for different applications of new copper – sulfide modified nanocomposite wood fibrous materials are developed.

The optimal regimes for reaching of minimal swelling; maximal strength on the face; and minimal electric resistance are given in following blocks N 1 to 4.

Block 1. Optimal regime for obtaining of minimal swelling						
Setting limitations: $y_1 > 30$; $y_2 > 25$; $y_3 < 30$						
X1	X2	X3	Y1	Y2	Y3	Y4
25.0000	1.9000	7.0000	31.238	27.210	29.227	3.696E+10
25.0000	1.9000	8.0000	31.727	26.476	29.106	3.694E+10
25.0000	1.9000	9.0000	32.093	25.794	28.948	3.804E+10
25.0000	1.9000	10.0000	32.334	25.163	28.754	4.036E+10
25.0000	2.0000	9.0000	31.610	28.246	29.935	4.357E+10
25.0000	2.0000	10.0000	31.828	27.565	29.704	4.674E+10
25.0000	2.0000	11.0000	31.922	26.935	29.437	5.165E+10
25.0000	2.0000	12.0000	31.893	26.357	29.133	5.880E+10
25.0000	2.0000	13.0000	31.739	25.831	28.793	6.896E+10
25.0000	2.0000	14.0000	31.462	25.357	28.416	8.331E+10
27.0000	1.9000	7.0000	32.136	26.512	29.328	3.224E+10
27.0000	1.9000	8.0000	32.662	25.844	29.258	3.228E+10
27.0000	1.9000	9.0000	33.063	25.228	29.151	3.329E+10
27.0000	2.0000	10.0000	32.897	27.036	29.975	4.071E+10
27.0000	2.0000	11.0000	33.028	26.473	29.759	4.507E+10
27.0000	2.0000	12.0000	33.035	25.961	29.506	5.139E+10
27.0000	2.0000	13.0000	32.918	25.501	29.218	6.037E+10

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27.0000	2.0000	14.0000	32.677	25.093	28.892	7.306E+10
29.0000	1.9000	7.0000	32.788	25.820	29.308	2.854E+10
29.0000	1.9000	8.0000	33.350	25.218	29.289	2.863E+10
29.0000	2.0000	11.0000	33.887	26.016	29.961	3.992E+10
29.0000	2.0000	12.0000	33.930	25.570	29.759	4.560E+10
29.0000	2.0000	13.0000	33.850	25.176	29.522	5.366E+10
31.0000	1.9000	7.0000	33.193	25.133	29.168	2.566E+10
31.0000	2.0000	12.0000	34.580	25.185	29.892	4.109E+10
35.0000	2.0000	7.0000	33.139	26.182	29.668	2.357E+10
35.0000	2.0000	8.0000	33.787	25.728	29.766	2.402E+10
35.0000	2.0000	9.0000	34.311	25.326	29.827	2.522E+10
37.0000	2.0000	7.0000	32.868	25.482	29.181	2.204E+10
37.0000	2.0000	8.0000	33.552	25.095	29.331	2.250E+10
Block 2. Optimal regime for obtaining of minimal swelling Setting limitations : y1>30; y2>15; y3<23						
X1	X2	X3	Y1	Y2	Y3	Y4
35.0000	1.2000	7.0000	30.091	15.482	22.786	4.0613E+10
37.0000	1.1000	10.0000	30.546	15.441	22.997	4.4816E+10
			30.196	15.034	22.615	3.7253E+10
37.0000	1.2000	8.0000	30.273	15.256	22.763	3.1891E+10
37.0000	1.3000	7.0000	30.141	15.498	22.823	4.5704E+10
39.0000	1.1000	11.0000	30.188	15.281	22.732	2.6212E+10
39.0000	1.4000	7.0000	30.089	15.010	22.550	3.7085E+10
41.0000	1.2000	11.0000	30.539	15.366	22.954	3.9113E+10
41.0000	1.2000	12.0000	30.232	15.146	22.688	3.365E+10
43.0000	1.3000	12.0000	30.665	15.029	22.845	2.6968E+10
43.0000	1.4000	11.0000	30.214	15.202	22.705	2.2073E+10
43.0000	1.5000	9.0000	30.011	15.618	22.811	3.3783E+10
45.0000	1.4000	13.0000				
Block 3. Optimal regimes for maximal strength on the face Setting limitations: y1>35; y2>22; y3<37						
X1	X2	X3	Y1	Y2	Y3	Y4
33.0000	1.9000	11.0000	35.148	22.884	29.023	2.8705E+10
33.0000	1.9000	12.0000	35.288	22.622	28.962	3.253E+10

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33.0000	1.9000	13.0000	35.304	22.411	28.864	3.7977E+10
33.0000	1.9000	14.0000	35.196	22.252	28.730	4.5673E+10
35.0000	1.9000	11.0000	35.207	22.473	28.846	2.678E+10
35.0000	1.9000	12.0000	35.383	22.277	28.836	3.04E+10
35.0000	1.9000	13.0000	35.435	22.132	28.790	3.5549E+10
			35.364	22.039	28.707	4.2825E+10
35.0000	1.9000	14.0000	35.140	24.431	29.794	3.4905E+10
35.0000	2.0000	12.0000	35.168	24.235	29.710	4.1278E+10
35.0000	2.0000	13.0000				
35.0000	2.0000	14.0000	35.074	24.092	29.590	5.0288E+10
37.0000	1.9000	11.0000				
37.0000	2.0000	12.0000	35.019	22.067	28.549	2.5367E+10
37.0000	2.0000	13.0000				
37.0000	2.0000	14.0000	35.050	24.062	29.564	3.2913E+10
			35.115	23.933	29.532	3.8988E+10
			35.057	23.855	29.463	4.7576E+10
Block 4. Optimal regimes for minimal electric resistance R: Setting limitations: $y_1 > 30$; $y_2 > 20$; $y_3 < 37$.						
X1	X2	X3	Y1	Y2	Y3	Y4
37.0000	1.8000	7.0000	32.850	21.033	26.943	1.9764E+10
37.0000	1.8000	8.0000	33.581	20.747	27.166	1.9732E+10
39.0000	1.8000	7.0000	32.208	20.397	26.303	1.8999E+10
39.0000	1.8000	8.0000	32.975	20.177	26.578	1.8999E+10
39.0000	1.8000	9.0000	33.619	20.008	26.816	1.9573E+10
39.0000	1.9000	7.0000	32.351	22.440	27.398	1.9515E+10
39.0000	1.9000	8.0000	33.095	22.169	27.636	1.9736E+10
41.0000	1.9000	7.0000	31.525	21.780	26.654	1.8929E+10
41.0000	1.9000	8.0000	32.305	21.576	26.943	1.9174E+10
43.0000	1.9000	7.0000	30.452	21.126	25.790	1.8641E+10
43.0000	1.9000	8.0000	31.269	20.988	26.130	1.8914E+10

43.0000	1.9000	9.0000	31.962	20.901	26.434	1.9771E+10
43.0000	2.0000	7.0000	30.575	23.415	26.998	1.9739E+10
45.0000	1.9000	9.0000	30.716	20.385	25.551	1.9834E+10

x – The volume electric resistance of non modified samples is from 6,6429E+11 to 8,9316E+11

The recommended conditions and parameters for achievement of maximal results are specified on the basis of data for the optimal regimes (blocks 1 to 4).

For minimal swelling (block 1) at setting limitations: $y_1 > 30$; $y_2 > 25$; $y_3 < 30$ the recommended conditions and parameters are:

Additive for modification: (25 %);

- mole proportion: $\text{CuSO}_4 \cdot 5\text{H}_2\text{O} : \text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ (1:2);

– duration of staying before pressing: (10 min.).

For minimal swelling (block 2) at setting limitations: $y_1 > 30$; $y_2 > 15$; $y_3 < 23$ the recommended conditions and parameters are: 37 %; 1:1, 3; 7 min;

For maximal strength on the face (block 3) at setting limitations: $y_1 > 35$; $y_2 > 22$; $y_3 < 37$ the recommended conditions and parameters are: 35 %; 1:1,9; 11 min.;

For minimal electric resistance R (block 4) at setting limitations: $y_1 > 30$; $y_2 > 20$; $y_3 < 37$ the recommended conditions and parameters are: 39 %; 1:1,8; 7 min.;

4. CONCLUSION

On the basis of present study, connected with obtaining and investigation of new copper sulfide modified nanocomposite wood fibrous materials for electromagnetic wave protection and mathematic modeling and optimization of process, using carbamide formaldehyde resin (CFS) as building substance, it is possible to give the following conclusions:

- It is established the advisability for conduction of modification of wood fibers by a surface treatment with $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ on the stages - gluing and formation of wood fibrous carpet;
- The developed modification method minimizes wastage of reagents and in the same time ensures the possibility for a full modification process;
- Optimal regimes for modification depending on special requirements of different applications of new copper – sulfide modified nanocomposite wood-fibrous materials have been developed.
- The copper sulfide modified nanocomposite wood-fibrous materials on the basis of carbamide formaldehyde resin as bonding substance with improved electroconductivity and corresponding microwave absorption ability at high content of wood component for special applications in the engineering and the popular costumes have been obtained.

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