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Polymer composites on the basis of lignocellulose containing copper sulfide for electromagnetic wave protection

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Composite materials are of fundamental importance in view of their unique properties and possibilities for applications. In this connection wood polymer micro- and nanocomposites for electromagnetic wave protection on the basis of lignocelluloses modified with Cu-S reduction systems and metal and metal oxides binding substances on the basis of recycled polyol from polyethylenterephtalate and isocyanates are highly effective new materials with special properties. An original method for *in situ* coordinative binding of cupro ions with lignocellulose using a three-component cuprous reduction system was developed. Binders from polyester polyols on the basis of waste polyethyleneterephtalate magnetic tapes after glycolysis, and polyisocyanate (p-MDI) were obtained. The reactions of complex formation as well the synthesis of metal and metal oxide binder systems for lignocellulose were investigated. Composites of modified flour and binders were produced by pressing. The optimum conditions - 110ºC, 20 min and 15000 kg/cm^2 pressing were established. Modified wood polymer composites possess good physical-mechanical characteristics, low electrical resistivity and high electromagnetic wave protection. Coordinative binding of copper ions with the oxygen atoms of cellulose OH groups and aromatic nucleus in the lignin macromolecule was observed. The new nanocomposites with microwave electromagnetic absorption properties could be used in radiotechnics, electronic industry, automobile building and others.

***Keywords**:* lignocellulose, wood, nanocomposites, cuprous sulfide, electromagnetic wave protection

INTRODUCTION

In recent years, increased use of wood polymer composites in technique and everyday life is observed. In this respect nanoscience and technology give unique possibilities for creating various combinations of new materials. The metal-containing nanocomposites and polymeric nanocomposites in particular, with their unique physical and chemical properties, as well as broad areas of application, have attracted great interest [1].

Composites based on polymers with conductive fillers are in the research focus of numerous studies as a part of a growing research trend. Polymers based on renewable materials with addition of chosen materials can be directly used as advanced materials [2].

Composites with improved parameters, including toughness, thermostability, and other specific properties, can be obtained by adding mineral fillers, metals, and fibers to polymers. Recent progress is being marked by the development of nanocomposite materials comprising a low percent of filler that have much larger intrinsic surface area, hence better parameters than conventional composites [3].

A great variety of inorganic layered materials can be used both as matrices or additives to polymers, with possibilities for obtaining a wide range of new hybrid nanocomposite materials [4].

An original method for obtaining metal-containing polymers by thermal decomposition of precursors (mainly metal carbonyls) in solution or melt has been published. A strong interaction between the nanoparticles and the polymer chain occurs in such nanocomposites at the level of chemical bonding. Electronically dense individual or aggregated metal-containing particles have been observed [5,6].

Recently, special attention has been paid to nanocomposites containing metal-organic derivatives on the basis of macromolecular metal complexes of ruthenium, palladium, platinum, etc. [7,8,9,10].

Nanocomposites allow the production of superconducting articles. It has been noted that the investigations in this field open possibilities of

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obtaining new materials with unique properties [10, 11, 12].

The method of direct precipitation of colloidal dispersed additive layers and their encapsulation into the polymer matrix has been recommended as one giving a lot of possibilities for the application of the obtained materials [13]. A new method for *in-situ* synthesis of precious nanoparticles using cellulose supports has been developed [14].

Methods for modification of fabrics with electroconductive pigments on the basis of copper sulfide polymer dispersion have been developed; these include new electro-conductive polyacrylonitrile (PAN) pigments with coordinative binding of nanostate copper sulfide into the PAN matrix for microwave protection [15].

Recently, investigations connected with copper sulfide coating on polyacrylonitrile with a chelating agent of triethanolamine [16] and ethylenediaminetetraacetic acid [17] by an electroless deposition method and its EMI shielding effectiveness have received great interest. The literature study showed that obtaining of new metal-containing nanocomposite polymeric materials with improved electroconductivity and microwave absorption ability is a question of increased interest and is very useful for the production of articles that can provide electromagnetic wave protection.

In this study, copper sulfide-containing nanolignocelluloses with improved electro-conductivity and microwave absorption ability were developed and on this basis polymer composites with special properties for electromagnetic wave protection were obtained.

The investigations aimed at:

- developing methods for obtaining of copper sulfide lignocellulose nanocomposites;
- establishing the probable mechanisms and schemes of delaying and subsequent inclusion of copper sulfides in the lignocellulose matrix;
- obtaining and characterization of recycled polyols on the basis of PET film tapes and corresponding binder substances;
- obtaining and characterization of wood polymer Cu(I) containing composites.

EXPERIMENTAL

Development of methods for obtaining of copper sulphide lignocellulose nano-composites

The development of methods for obtaining copper sulfide lignocellulose nano-composites is based on the fact that copper (Cu^{+1}) sulfide as an additive to polymers imparts high electroconductivity. The latter, as an indirect index for dielectric losses, shows that the corresponding new materials will possess microwave absorption properties. A higher effect could be achieved with nano copper sulfide, which is situated as a net in the lignocellulose matrix. In this sense the development of methods is based on performing chemical modification on different kinds of lignocellulose materials – wood flour (WF), wood fibrous materials (WFM), and waste cellulose fibers WCF) – with aqueous solutions of copper compounds and sulfur-containing reduction systems in suitable quantities and ratios under definite process conditions. Such conditions will give possibilities for a reduction of Cu^{+2} to Cu^{+1} ions to take place with subsequent coordinative precipitation in lignocellulose matrices.

In the present work the following lignocellulose materials were used:

- wood flour (WF);
- wood fibrous material (WFM), produced in Lesoplast AD, Troian (Bulgaria);
- waste cellulose fibers (WCF) by paper production.

Basic Variants for Chemical Modification Process on Lignocelluloses

By previous experiments (varying the kind and composition of components) two variants for experimentation were set:

- using a three-component system (cupric sulfate pentahydrate ($CuSO_4$. $5H_2O$); sodium thiosulfate pentahydrate ($N_2S_2O_3.5H_2O$); glyoxal (OCHCHO));
- using a two-component system (cupric sulfate pentahydrate ($CuSO_4.5H_2O$); sodium thiosulfate ($N_2S_2O_3.5H_2O$).
- The following indicators for optimization of the methods were used:
- amount of waste water;
- content of copper and sulfur in the waste water;
- content of copper and sulfur in the modified lignocelluloses.

IR spectra of non-modified and modified samples were recorded on a Perkin Elmer FTIR – GX apparatus, having a spectral range of 4000 to 350 cm^{-1} and a resolution of 4 cm^{-1}.

Modification with a Three-Component System

The three-component system comprised cupric sulfate pentahydrate ($CuSO_4.5H_2O$), sodium thiosulfate ($N_2S_2O_3.5H_2O$); and glyoxal (OCHCHO) can be characterized as follows:

Table 1. Copper-containing Lignocelluloses, Modified with a System of Three Components

№	Conditions of modification	Copper in sample, %	Sulfur in sample, %	Copper in filtrate, mg/l	Sulfur in filtrate, mg/l	Specific electric volume resistance, Ω.m
1.	Non - modified wood fibers (WF)	0.0006	0.33	-	-	8.25×10^8
2.	Non - modified waste cellulose fibers (WCF)	0.0007	0.25	-	-	5.7×10^7
3.	Non - modified wood flour (WF)	0.0009	>0.05	-	-	8.7×10^8
4.	Modified wood fibers (WF)	3.786	>0.05	362.0	103.0	1.65×10^8
5.	Modified waste cellulose fibers (WCF)	4.319	1.92	5.0	69.0	1.11×10^7
6.	Modified wood flour (WF)	5.058	1.79	1380.0	116.0	1.95×10^8

- the reduction process had a very strong effect on the wood fibrous materials (WFM), and therefore the sulfur content in the modified material was insignificant;
- on the waste cellulose fibers (WCF) the Cu^{+2} ions were reduced to Cu^{+1};
- on the modified wood flour (WF) the reduction yielded not only Cu_2S but also copper particles, hence the reduction process was most strongly expressed in that case.

Lower copper and sulfur contents in waste waters were observed with modified wood fibrous materials (WFM) in comparison with those of modified wood flour (WF).

The specific electric resistance decreased in the order 0.5 to 1.0 after modification of the three different lignocelluloses.

The results obtained with the three-component system cuprous sulfate ($CuSO_4.5H_2O$), sodium thiosulfate ($N_2S_2O_3.5H_2O$), and glyoxal (OCHCHO) unambiguously pointed to the possibility of obtaining Cu- containing lignocelluloses.

Establishment of probable mechanisms and schemes of delaying and subsequent connection of copper sulfide in the lignocellulose matrix

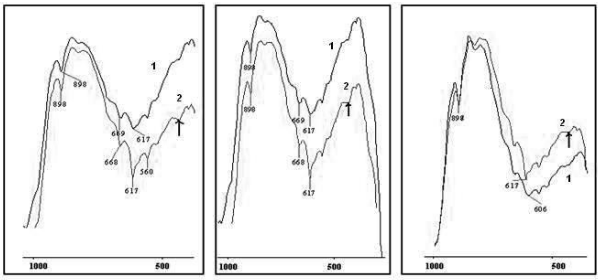
The scheme of the reduction process is as follows:

$Cu^{+2} + 1e^- \rightarrow Cu^{+1}$

$2S^{+2} – 2.2e^- \rightarrow 2S^{+4}$

$2CuSO_4 + 2Na_2S_2O_3 = Cu_2S + 2Na_2SO_4 + 3SO_2$

A peak at 400 cm^{-1}, representative of metal-oxygen ligands, was observed in the IR spectra of modified wood flour and of modified wood fibers (Fig. 1, a,b,c).



a **b** **c**

Fig. 1. IR spectrum of: a) non-modified (1) and modified (2) wood flour with system of three components (40% addition based on wood flour); b) non-modified (1) and modified (2) wood fibers with system of two components (30% added based on wood fibers); and c) non-modified (1) and modified (2) wood fibers with system of two components (40% added based on wood fibers).

These ligands are probably resulting from the coordinative binding of copper ions to oxygen atoms of cellulose -OH groups (Fig. 2) and aromatic nucleus in the lignin macromolecule (Fig. 3).

A similar model for Cu^{+2} complexes with the lignin model compound vanillin was given by Kozlevcar (2005). The binding of copper ions via methoxy oxygen atom and deprotonated hydroxy oxygen atom in nuclear species confirmed the binding role of copper to lignin (Zhang 2005). It is possible for lignocellulose materials to give evidence of physical adsorption to copper ions.

Obtaining and characterization of recycled polyols on the basis of PET film tapes and corresponding binder substances; and obtaining and characterization of wood polymer cuprous containing composites.

Obtaining of binder substances on the basis of PET magnetic tapes

The PET waste tapes were recycled by glycolysis using diethylene glycol, adipine acid and glycerol. The obtained polyols were characterized by hydroxyl number (DIN 53240); acid number and viscosity at 25ºC (DIN 3219).

The synthesis of the polyurethane binder was conducted on the basis of recycled polyols and dipheylmethane-4-4-diisocyanate (MDI) under conditions for obtaining of binders with free isocyanate groups. Such binders will be able to interact with the hydroxyl groups of lignocellulose materials. The reaction was conducted at an equivalent ratio 1:1 of the two components, at 80ºC in vacuo with subsequent drying at the same temperature for 24 hours with a view to promote the polymerization process. The obtained product was ground in a laboratory mill and was characterized by DSC, isocyanate content (DIN53185) and rheological characteristics.

Obtaining and methods for characterization of wood polymer composites (WPC)

Materials: Wood flour (BDS 3718) – conifer wood, particle size 140 μm, acidity 0,030 %, tarry substances 2%, specific volume 8 dm^3/kg, humidity 5%, cork content 0.34%;

Wood flour modified using the three-component system $CuSO_4$: $Na_2S_2O_3$: $C_2H_2O_2$ = 1.6:1.4:1, at a hydro module of soaking 1:10, 100°C temperature for 30 min. Laboratory-made WPC by pressing.

WPC were characterized with regard to: physical and mechanical properties such as tensile strength, elongation at break, modulus of elasticity and others (apparatus Zwick 2000, Zwick GmbH & Co. KG, Ulm, Germany) and electroconductivity by determination of specific electrical volume resistance (apparatus Teralin III, BDS HD 429 S1:2003).

The following parameters for determination of electromagnetic characteristics of wood polymer composites were used: complex dielectric permittivity (έ), reflection coefficient on module |Γ́| and damping coefficient α, dB/cm.

Results and discussion

Aromatic polyester polyols (APP)

The results of the characterization of the obtained APP are presented in Table 2.

Binder substances (BS)

The synthesis of a polyurethane binder was conducted on the basis of recycled polyols and diphenylmethane-4-4-diisocyanate (MDI) at an equivalent ratio 1:1 of the two components. The results from the DSC of BS are summarized in Table 3. On the basis of these results, BS number 4 is recommended for obtaining of WPC because the exothermic peak of 127.5ºC is at a lower temperature and with wider interval. Comparing this with other binder substances it is clear that there is a higher possibility for a reaction between the hydroxyl groups of lignocellulose materials and NCO groups of BS and therefore for obtaining of WPC with high physical-mechanical indexes.

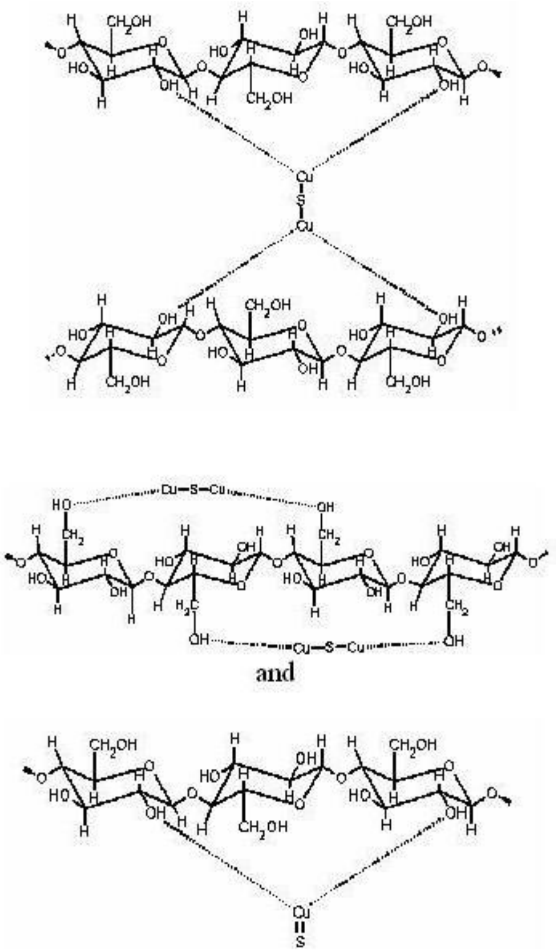


Fig.2. Copper-sulfide cellulose nanocomposites

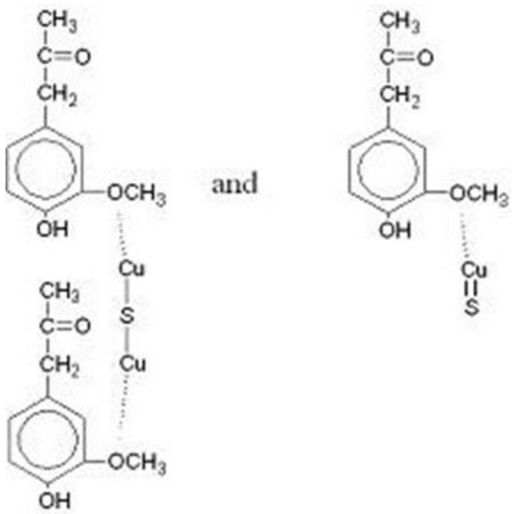


Fig.3. Copper-sulfide lignin nanocomposites

Table 2. Indexes of APP

Index	Measure	Recipe of APP			
		APP1	APP2	APP3	APP4
OH-number	mgKOH/g	416	341	326	325
acid number	mgKOH/g	0.36	0.85	0.60	0.79
Viscosity by oscillation	mPa.s	860	1160	1540	6360
Viscosity by rotation	mPa.s	760	1100	1500	5980

Table 3. Indexes of BS by DSC

Binder substance	Index		
	Temperature of glass., [°C]		Temperature of reaction ability, [°C]
	1	2	
BS №1	45.7	78.3	152.5
BS №2	52.4	67.9	158.7
BS №3	37.6	39.8	140.5
BS№4	37.2	76.4	127.5

Wood polymer composite materials

The parameters of the obtained WPC are given in Table 4, the physical-mechanical indexes of the WPS – on Figs. 4-7, and the specific electrical volume resistance – on Fig. 8.

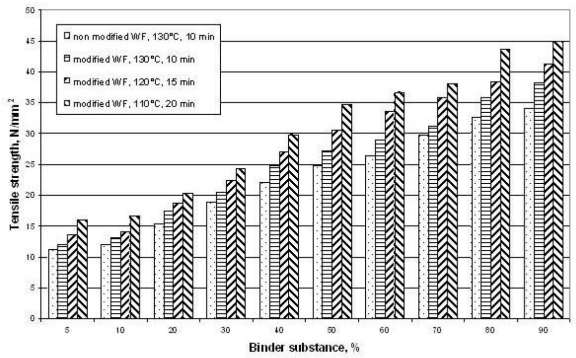


Fig. 4. Tensile strength as a function of BS%

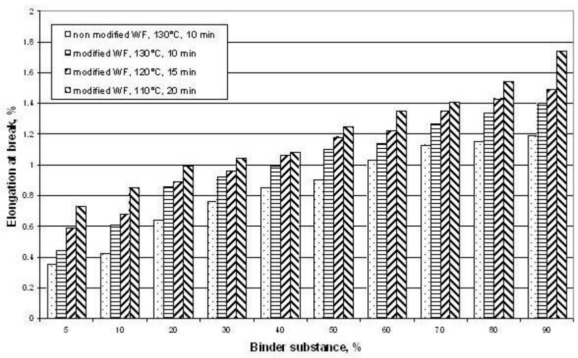


Fig.5. Elongation at break as a function of BS%

Series 1 includes WPC based on non-modified wood flour (WF) and BS (5% to 90%) after pressing at 130ºC for 10 min;

Series 2 includes WPC based on modified wood flour (WF) and BS (5% to 90%) after pressing at 130ºC for 10 min;

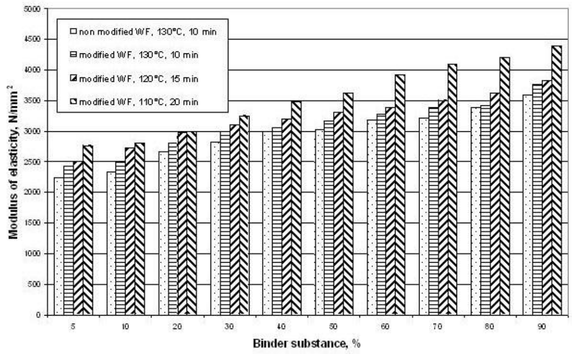


Fig. 6. Modulus of elasticity as a function of BS%

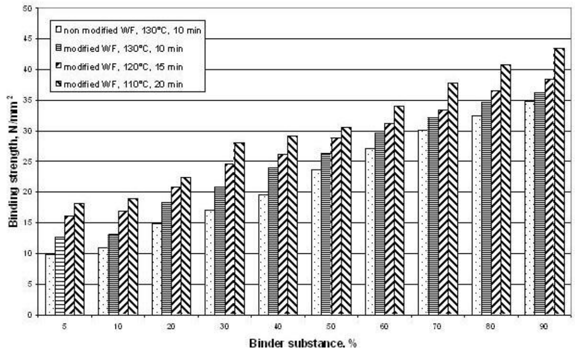


Fig. 7. Bending strength as a function of BS%

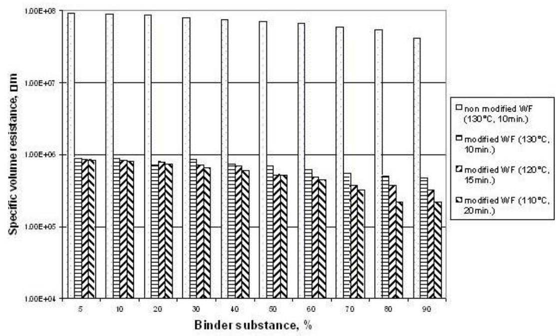


Fig. 8. Specific volume resistance as a function of BS%

Series 3 includes WPC based on modified wood flour (WF) and BS (5% to 90%) after pressing at 120ºC for 15 min;

Series 4 includes WPC based on modified wood flour (WF) and BS (5% to 90%) after pressing at 110ºC for 20 min;

The experimental data shown in Fig. 4 reveal the following correlations:

- the tensile strength of WPC based on non-modified WF after pressing at 130^{o}C for 10 min increases at a higher quantity of BS;
- the tensile strength of WPC based on modified WF for all variants of pressing exceeds that of WPC based on non-modified WF.

The highest values are achieved at 110 ^{o} C for 20 min and 90 % BS.

Table 4. Parameters of the obtained WPC

№	Sample	Components, %			Pressing conditions		№	Sample	Components, %		Pressing conditions	
		WF		BS	T	Duration			Modified WF	BS	T	Duration
		Non mod.	Mod.		(°C)	(min)					(°C)	(min)
1	M1P	95	–	5	130	10	21	M21P	95	5	120	15
2	M2P	90	–	10	130	10	22	M22P	90	10	120	15
3	M3P	80	–	20	130	10	23	M23P	80	20	120	15
4	M4P	70	–	30	130	10	24	M24P	70	30	120	15
5	M5P	60	–	40	130	10	25	M25P	60	40	120	15
6	M6P	50	–	50	130	10	26	M26P	50	50	120	15
7	M7P	40	–	60	130	10	27	M27P	40	60	120	15
8	M8P	30	–	70	130	10	28	M28P	30	70	120	15
9	M9P	20	–	80	130	10	29	M29P	20	80	120	15
10	M10P	10	–	90	130	10	30	M30P	10	90	110	15
11	M11P	–	95	5	130	10	31	M31P	95	5	110	20
12	M12P	–	90	10	130	10	32	M32P	90	10	110	20
13	M13P	–	80	20	130	10	33	M33P	80	20	110	20
14	M14P	–	70	30	130	10	34	M34P	70	30	110	20
15	M15P	–	60	40	130	10	35	M35P	60	40	110	20
16	M16P	–	50	50	130	10	36	M36P	50	50	110	20
17	M17P	–	40	60	130	10	37	M37P	40	60	110	20
18	M18P	–	30	70	130	10	38	M38P	30	70	110	20
19	M19P	–	20	80	130	10	39	M39P	20	80	110	20
20	M20P	–	10	90	130	10	40	M40P	10	90	110	20

The same correlations (Figs. 5-7) are observed for the indexes - elongation at break, modulus of elasticity and bending strength.

Differences however are observed between modified and non-modified WF. It is worth noting that the optimal indexes of elongation at break, modulus of elasticity and bending strength are observed with series 4 (WPC obtained at 110ºC for 10 min with 90% BS). Physical-mechanical indexes of WPC in all series increase upon increasing % BS quantity.

The experimental data shown in Fig. 8 reveal the following correlations:

- the specific electrical volume resistance insignificantly decreases with the increase in the % content of BS in series 1 with non-modified WF;

- for series 2, 3 and 4 the specific electrical volume resistance of modified WF significantly decreases in comparison with non-modified WF. The best results are achieved with series 4.

Highest electroconductivity (lowest values of specific electrical volume resistance) is achieved with WPC (obtained with modified WF 5 % and BS 90 %, after pressing at 110 ^{o}C for 20 min).

WPC (series 4) were subjected to electromagnetic wave characterization (microwave absorption properties – electromagnetic wave damping – from 9 to 6 dB and standing wave coefficient – from 2,57 to 1.13). These data show that WPC with a Cu-containing lignocellulose nanocomposite component and N-containing binder substance (BS) possess high electromagnetic wave absorption. The obtained results confirm the idea for coordinative binding of Cu_2S with the N-containing polymer components.

CONCLUSIONS

Treatment with a system of three components, comprising cupric sulfate pentahydrate ($CuSO_4.5H_2O$), sodium thiosulfate pentahydrate ($Na_2S_2O_3.5H_2O$), and glyoxal (OCHCHO) at ambient pressure and 90^{o} C is very effective for modification of waste cellulose fibers and to some extent wood flour.

IR spectral data confirmed the coordinative binding of copper ions with the oxygen atoms of OH groups in cellulose and in the aromatic nucleus of the lignin macromolecule.

A treatment for modification of wood flour with a three-component cuprous reduction system was conducted. Wood nanocomposites containing Cu-3.21% and S-1.07% were obtained.

Binder substances were prepared on the basis of recycled polyols from PET waste tapes and diphenylmethane-4-4-diisocyanate (p-MDI).

On the basis of non-modified and modified wood flour and binder substances Cu-containing nanocomposites with high electroconductivity and electromagnetic wave absorption were prepared.

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ПОЛИМЕРНИ КОМПОЗИТИ ЗА ЕЛЕКТРОМАГНИТНА ВЪЛНОВА ЗАЩИТА НА ОСНОВА НА ЛИГНОЦЕЛУЛОЗА СЪДЪРЖАЩА МЕДЕН СУЛФИД

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(Резюме)

Композитните материали предизвикват голям интерес поради техните уникални свойства и възможности за приложение. В тази връзка дървесноплимерните микро- и нанокомпозити за електромагнитна вълнова защита на основата на лигноцелулози, модифицирани с Cu -S системи и метал оксидни свързващи вещества на база рециклирани полиоли от полиетилен терефталат и изоцианати, са високо ефективни нови материали със специфични свойства. Разработен е оригинален метод, основаващ се на кординативно свързване in situ на купро йони с лигноцелулоза чрез трикомпонентна редукционна система.

Получени са свързващи вещества от полиоли, на основата на отпадни полиетилен терефталатни магнитни ленти чрез гликолиза, и полиизоцианат (p-MDI). Изследвани са реакциите на комплексообразуване и тези на синтез на металоксидните свързващи системи за лигноцелулози.

Получени са композити от модифицираното брашно и свързващите вещества чрез пресоване. Установени са оптималните условия на пресоване - 110^{o} C, 20 минути и 1500 kg/cm^2 Модифицираните дървесноплимерни композити притежават високи физикомеханични показатели, ниско електрическо съпротивление и висока електромагнитна вълнова защита.

Установено е кординативно свързване на медните йони с кислородните атоми на целулозните OH групи и ароматните ядра в макромолекулите на лигнина.

Новите нанокомпозити с висока електромагнитна вълнова абсорбция могат да бъдат използвани в електротехниката, електрониката, автомобилостроенето и в много други отрасли на техниката и бита.