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(54) A METHOD OF PRODUCING A PLATE COMPOSITE WITH THE USE OF CELLULOSE AND CARBON PARTICLES WITH INCREASED FIRE RESISTANCE

(57) A method of producing a plate composite with the use of cellulose and carbon particles with increased fire resistance is that

1.1. particles rich in elemental carbon, i.e. with a carbon content >90%, selected from expanding graphite (EG) and carbon nanotubes (CNT), are dispersed in water in weight ratios ranging from 0.2:1 to 1:0.2. in an aqueous system, the weight ratio of carbon particles to water is from 1:1 to 100:1.

1.2. a cellulose pulp with a concentration of 1-25% in water is produced

1.3. a dispersed suspension of carbon particles is introduced into the cellulose pulp.

1.4. a composite is formed on a paper machine

1.5. the composite is dried and pressed

wherein the EG/CNT dispersion is kept in water for a period of 24 hours, and resulting particle dispersion is

homogenized for 30-45 s by means of a homogenizer with a rotation speed from 400 to 4000 rpm until a homogeneous water dispersion is obtained and the homogeneous the particle dispersion is added to the pulp produced in parallel in an amount of 1-75% by weight based on the total mass of the product and the mixture is further grounded for 10 s in the refiner, and cellulose pulp is produced in such a way that the cellulose in the form of sheets is initially comminuted into particles size 1-4 cm², soaked for 24 hours in deionized water, the weight ratio of cellulose to water is 1:10, and after 24 hours the cellulose is defibrated in a defiberizer and water is added until the total cellulose to water ratio is at the level of 1:50, then the homogeneous pulp is transferred to a sheet forming apparatus and diluted with water to a mass concentration ranging from 2-20%, and cellulose-carbon sheets are formed.

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Description

[0001] The present invention relates to a method of producing of a composite material made of cellulose and carbon particles, resulting in a plate composite with increased fire resistance.

- ⁵ **[0002]** The present invention may be used in the construction industry (as a cladding material), vehicular, water and air transportation (e.g., building up railroad compartments, furnishing tourist vehicles, boats, yachts, aircraft), as well as a specialized packaging material. Composite may be used specifically as a cladding material due to its unique properties (e.g. low density, small cross-sectional dimensions, barrier to fire, water, sound).
- [0003] From the description of CN205950916 (U), a solution of layered elements is known, where a fire-resistant plywood was produced, in which the outer layers are made of fire-resistant impregnated wood veneers and the inner layer is made of fiberboard. In this solution, aluminum hydroxide was introduced into the inner layer in the form of granules. The layers were glued together using fire-retardant amine resin. According to the description, the layers can also be made up of paper made fire-resistant with melamine.
- [0004] Utility model CN205369573 (U) discloses a fire-resistant board with a three-layer construction, including a resistant inner layer. The layers between each other are glued together by means of an glue. The outer layers may can be cladded on both sides.

[0005] US 2019/0016958 A1 solution is known concerning the use of expanded graphite for the reduction of flammability, specifically for its use as a flame retardant agent, for materials and/or products consisting of, or comprising, wood fibers, fiber cellulose, wood powder, cellulose powder, wood granules, cellulose granules and/or polyolefin-based ma-

- 20 terials. Moreover, it relates to materials and/or products, consisting of, or comprising wood fibers, cellulose fibers, wood powder, cellulose powder, wood pellets, cellulose pellets and/or polyolefin-based materials. To reduce the flammability in materials and/or products, expanded graphite is embedded. The invention also relates to such an agent, specifically a fire retardant agent, wherein expanded graphite is used alone or in combination with a boric acid/borax/alkali salt mixture. Specifically favorable areas of application are binders, adhesives and/or materials, products and/or materials,
- ²⁵ products and/or intermediates containing in specifically polyolefin-based materials, preferably for damping (walls, floors, ceilings) and/or for fixing floors and walls.

[0006] A method of producing a plate composite with the use of cellulose and carbon particles with increased fire resistance is that

- 30 1. Particles rich in elemental carbon, i.e. with a carbon content >90%, selected from expanded graphite (EG) and carbon nanotubes (CNT), are dispersed in water in weight ratios ranging from 0.2:1 to 1:0.2. in an aqueous system. The weight ratio of carbon particles to water is from 1:1 to 100:1.
 - 2. Cellulose pulp with a concentration of 1-25% in water is produced
 - 3. A dispersed suspension of carbon particles is introduced into the cellulose pulp.
- 4. A composite is formed on a paper machine
 - 5. The composite is dried and pressed

[0007] Wherein in the first step an aqueous dispersion of elemental carbon-rich particles (>90%) is produced, so that particles of expanded graphite (EG) and carbon nanotubes (CNT) are mixed, in weight ratios in the range of 0.2:1 to 1 :0.2. in an aqueous system. The weight ratio of carbon particles to water ranges from 1:1 to 100:1, the EG/CNT dispersion is maintained in water for a period of 24 hours, and then the resulting particle dispersion is homogenized for 30-45 seconds using a homogenizer with a speed from 400 to 4000 rpm until a homogeneous water dispersion is obtained. The resulting homogeneous dispersion of particles is added to the simultaneously produced pulp in an amount

of 1-75% by mass in relation to the total mass of the product and the mixture is further grounded for 10 s in the refiner.
 [0008] The size of EG particles is in the range of 10 - 1000 micrometers, and the average length of CNT is 1.5 μm, and the diameter is 9.5 *10-9 m.

[0009] Wherein the pulp is produced in such a way that cellulose, in the form of sheets, is initially comminuted into particles size of 1-4 cm², soaked in deionized water for 24 hours, the weight ratio of cellulose to water is 1:10, and after 24 hours the cellulose is defibered in a defiberizer and water is added until the total cellulose to water ratio is at the level of 1:50

50 of 1:50

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[0010] The homogeneous pulp is transferred to a sheet-forming apparatus and diluted with water to a mass concentration in the range of 2-20%, specifically 7.5%, and cellulose-carbon sheets are formed. Sheet forming is advantageously preceded by intensive mixing of the dispersion by the barbotage method, i.e., by passing air through the forming sieve from below at a pressure of 0.2-0.4 bar, and then the pulp is filtered out on a sieve with a mesh size of max. 0.25 cm.

⁵⁵ The formed sheets are then transferred to a dryer, where simultaneously excess water is sucked out and the sheet is pressed at a maximum temperature of 97°C in time up to 600 s at a pressure of max. -97kPa.
[00111] The resulting composite material based on cellulose fibers incrusted with carbon particles allows to achieve

[0011] The resulting composite material based on cellulose fibers incrusted with carbon particles allows to achieve low cross-sectional dimensions and low density while maintaining high fire resistance qualities. These features are highly

desirable for specialized applications.

[0012] The composite can be a single-layer material (photo 1.) or a multi-layer material, consisting of at least two layers, where at least one layer is the said composite material, and the layers are joined by any known binder or biobinder. The composite material can also constitute inner layers, outer layers or both at the same time. The composite material

⁵ can also be combined with other layers made of other or the same materials, but characterized by different carbon to cellulose particle ratios (photo. 3). [0013] The aim of the invention is, therefore, a composite in the form of a layer, as a finished product as such, or a system of layers made of cellulose incrusted with expanded graphite (EG) and carbon nanotubes (CNT), combined to form a plate-like composite material in which the cellulose fibers provide mechanical strength to the product and the

¹⁰ carbon particles protect it from fire.

[0014] Table 1 shows examples of cellulose material variants with different carbon particle contents. It was unexpectedly found that saturating the hydrophobic carbon particles with water, provided full homogeneity and uniform distribution in the cellulose mass, which in subsequent steps of the production of the composite material translated into the fire-resistant properties of the final product.

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Table 1.

Share of individual components in samples of cellulose-carbon layers

20	variant	cellulose mass [g]	EG mass [g]	CNT mass [g]	EG/CNT mass [g]	relative humidity [%]	absolute humidity [%]	weight [g/m²]	HRR peak value (MLC) [kW/m ²]	loss in mass (MFT) [%]
25	С	10	0	0	0	3.81	3.67	30.8	137.04	98.80
	СС	5	0	0	0	1.16	1.15	15.6	48.44	98.99
	Α	10	5	0	0	2.31	2.26	45.0	81.30	69.97
30	В	10	3.75	0	0	2.08	2.03	52.5	70.63	55.10
	D	10	10	0	0	2.24	2.19	58.6	59.48	14.48
	E	10	15	0	0	1.64	1.61	74.1	46.07	7.91

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1.44

4.19

1.67

0.70

87.1

22.4

29.3

44.1

6.36

76.76

47.25

10.06

38.06

60.39

44.44

30.73

1.47

4.37

1.70

0.71

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	J	10	0	0	2.5/2.5	2.48	2.42	48.4	98.29	50.17
10	к	10	0	0	3.75/3.75	2.54	2.48	57.0	84.37	46.48
	L	10	0	0	5.0/5.0	2.32	2.27	63.3	68.93	45.59
	LL	10	0	0	7.5/7.5	2.05	2.01	84.7	36.43	6.84
	M	10	0	0	10.0/10.0	1.54	1.52	103.4	22.82	5.32
	N	5	0	0	1.25/1.25	1.57	1.55	22.7	64.83	53.36
15	0	5	0	0	2.5/2.5	1.05	1.04	30.4	58.01	53.36
	Р	5	0	0	5.0/5.0	1.97	1.93	44.5	34.72	7.15
	R	5	0	5	0	3.48	3.36	22.7	60.25	71.65
	S	5	0	5	0	1.74	1.71	31.8	63.35	10.06
20	Т	5	0	10	0	1.48	1.46	49.5	59.38	50.17
	V	10	0	5	0	2.44	2.38	49.5	126.56	61.78
	W	10	0	3.75	0	1.91	1.88	57.7	119.66	50.20
25	X	10	0	10	0	1.89	1.86	74.9	113.54	47.99
	Y	10	0	15	0	1.87	1.83	93.2	94.52	29.26
	Z	10	0	20	0	1.64	1.62	111.9	88.05	27.44
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COMBUSTIBLE PROPERTIES OF THE COMPOSITE COMPONENT AND THE COMPOSITE AS THE FINAL MATE-RIAL

Combustion properties of plate composite determined by the mini fire tube (MFT) method

[0015] The lowest value of mass loss (ML) and the exhaust gas temperature was shown by the cellulose-carbon layer 35 formed in the system: cellulose:EG:CNT (M), with a maintained ratio of respectively: 1:1:1. The mass loss and exhaust gas temperature at the exit of the pipe were respectively: 5.3% and 115°C. In the other variants, the mass losses were higher (6-70%), as were the exhaust gas temperatures (96-315°C). Control samples comprising 5 and 10 g of cellulose showed the highest mass loss (about 99%). The exhaust gas temperature at the exit of the pipe reached a the highest value of about 400°C.

40 [0016] It was unexpectedly found that the presence of both types of carbon particles (EG and CNT) in the cellulose layer translated into a synergistic effect in terms of fire protection.

Combustion properties of plate composite determined by the oxygen index (OI) method

45 [0017] Layer samples made of cellulose encrusted with EG and CNT tested by the oxygen index (OI) method showed high fire resistance. The maximum oxygen index value for the sample of cellulose encrusted with EG (at a cellulose to EG ratio of 1:2) was 75%. The lowest value (22.5%) was shown by a sample of cellulose encrusted with EG and EG/CNT in a 2:1 ratio. The control samples showed an OI value of 20%, making the latter qualify as combustible.

50 Combustion properties of plate composite determined by mass loss calorimeter (MLC) method

[0018] Samples of cellulose encrusted with CNT (in a 2:1 ratio) tested by the MLC method achieved the highest HRR of about 126kW/m², while the control sample showed an HRR at the level of about 137kW/m². The cellulose sample encrusted with EG or EG/CNT (with a cellulose to carbon particle ratio of 1:2) showed the lowest HRR, regardless of the mass of the cellulose (5 or 10g). The values were as follows: 35 and 38 or 23 and 31 kW/m² for 5g cellulose, respectively: EG (1:2) and cellulose 10g: EG/CNT (1:2).

[0019] A 10g cellulose sample with CNT (cellulose to carbon particle ratio of 1:2) showed the longest time to ignition - 18 seconds. The cellulose sample encrusted with EG and CNT particles (5g cellulose: EG/CNT, 2:1) ignited the fastest, i.e. after about 3s.

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[0020] Cellulose encrusted with carbon particles in a 1:1.5 or 1:2 ratio, where the carbon particles were an EG/CNT system in a 1:1 ratio, showed high fire resistance properties.

[0021] The results confirmed the non-obviousness resulting from the MFT analysis, where a synergy effect was found in the case of cellulose encrusted with EG and CNT in the ratio of 1:0.75:0.75 and 1:1:1.

Combustion properties of layered composite determined by mass loss calorimeter (MLC) method

[0022] The three-layer composite, in which the core was a layer comprising different amounts of EG: the cellulose 10 mass ratio to EG was 1:5 (8 BV), showed the best results, i.e., the maximum peak HRR was 212 kW/m² at 290s, while the control variant (F) reached a value of 282 kW/m² at 260s.

[0023] The three-layer composite, in which the core constituted a layer of cellulose encrusted with EG/CNT in a 1:1:1 ratio to cellulose (10EG10CNT10C), characterized by the highest fire resistance among the materials tested. The maximum value of the heat release rate (HRR) was 154.07 kW/m², while the HRR peak of the composite containing only EG was 282 kW/m²

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Example 0

[0024] A method of producing a plate composite with the use of cellulose and carbon particles with increased fire 20 resistance is that

1. Particles rich in elemental carbon (>90%) are dispersed in water, wherein the particles rich in elemental carbon being a mixture of expanding graphite (EG) and carbon nanotubes (CNT), in weight ratios in the range of 0.2:1 in an aqueous system. The weight ratio of carbon particles to water is from 1:1.

- 25 2. Cellulose pulp with a concentration of 1% in water is produced
 - 3. A dispersed suspension of carbon particles is introduced into the cellulose pulp.
 - 4. A composite is formed on a paper machine
 - 5. The composite is dried and pressed
- 30 [0025] Wherein in the first step an aqueous dispersion of elemental carbon-rich particles (>90%) is produced, so that particles of expanded graphite (EG) and carbon nanotubes (CNT) are mixed, in weight ratios in the range of 0.2:1 in an aqueous system. The weight ratio of carbon particles to water is 1:1, the EG/CNT dispersion is maintained in water for a period of 24 hours, and then the resulting particle dispersion is homogenized for 30-45 seconds using a homogenizer with a speed from 400 to 4000 rpm until a homogeneous water dispersion is obtained. The resulting homogeneous

35 dispersion of particles is added to the simultaneously produced cellulose pulp in an amount of 75% by weight in relation to the total mass of the product and the mixture is further grounded for 10 s in the refiner. [0026] The size of EG particles is in the range of 10 - 1000 micrometers, and the average length of CNT is 1.5 μ m, and the diameter is 9.5 *10-9 m.

[0027] Wherein the pulp is produced in such a way that cellulose, in the form of sheets, is initially comminuted into 40 particles size of 1-4 cm², soaked in deionized water for 24 hours, the weight ratio of cellulose to water is 1:10, and after 24 hours the cellulose is defibered in a defiberizer and water is added until the total cellulose to water ratio is at the level of 1:50

[0028] The homogeneous pulp is transferred to a sheet forming apparatus and diluted with water to a mass concentration of 7.5%, and cellulose-carbon sheets are formed. Sheet forming is advantageously preceded by intensive mixing

- 45 of the dispersion by the barbotage method, i.e., by passing air through the forming sieve from below at a pressure of 0.2-0.4 bar, and then the pulp is filtered out on a sieve with a mesh size of max. 0.25 cm. The formed sheets are then transferred to a dryer, where simultaneously excess water is sucked out and the sheet is pressed at a maximum temperature of 97°C in time up to 600 s at a pressure of max. -97kPa.
- 50 Example 0'

[0029] A method of producing a plate composite with the use of cellulose and carbon particles with increased fire resistance is that

55 1. Particles rich in elemental carbon (>90%) are dispersed in water, wherein the particles rich in elemental carbon being a mixture of expanding graphite (EG) and carbon nanotubes (CNT), in weight ratios in the range of 1:0.2 in an aqueous system. The weight ratio of carbon particles to water is 100:1. 2. Cellulose pulp with a concentration of 25% in water is produced

EP 4 389 969 A1

- 3. A dispersed suspension of carbon particles is introduced into the cellulose pulp.
- 4. A composite is formed on a paper machine
- 5. The composite is dried and pressed
- ⁵ **[0030]** Wherein the first step an aqueous dispersion of elemental carbon-rich particles (>90%) is produced, so that particles of expanded graphite (EG) and carbon nanotubes (CNT) are mixed, in weight ratios in the range of 1:0.2 in an aqueous system. The weight ratio of carbon particles to water is 100:1, the EG/CNT dispersion is maintained in water for a period of 24 hours, and then the resulting particle dispersion is homogenized for 30-45 seconds using a homogenizer with a speed from 400 to 4000 rpm until a homogeneous water dispersion is obtained. The resulting homogeneous
- ¹⁰ dispersion of particles is added to the simultaneously produced pulp in an amount of 1% by weight in relation to the total mass of the product and the mixture is ground for 10 s in the refiner.
 [0031] The size of EG particles is in the range of 10 1000 micrometers, and the average length of CNT is 1.5 μm, and the diameter is 9.5 *10-9 m.

[0032] Wherein the cellulose pulp is produced in such a way that cellulose, in the form of sheets, is initially comminuted

¹⁵ into particles size of 1-4 cm² size, soaked in deionized water for 24 hours, the weight ratio of cellulose to water is 1:10, and after 24 hours the cellulose is defibered in a defiberizer and water is added until the total cellulose to water ratio is at the level of 1:50

[0033] The homogeneous pulp is transferred to a sheet forming apparatus and diluted with water to a mass concentration in the range of 7.5%, and cellulose-carbon sheets are formed. Sheet forming is advantageously preceded by

intensive mixing of the dispersion by the barbotage method, i.e., by passing air through the forming sieve from below at a pressure of 0.2-0.4 bar, and then the pulp is filtered out on a sieve with a mesh size of max. 0.25 cm. The formed sheets are then transferred to a dryer, where simultaneously excess water is sucked out and the sheet is pressed at a maximum temperature of 97°C in time up to 600 s at a pressure of max. -97kPa.

²⁵ EXEMPLARY EMBODIMENTS OF COMPOSITE LAYER (EXAMPLE 1-6) AND A COMPOSITE LAYER (EXAMPLE 7-19)

Example 1

- 30 [0034] 10 g of EG and 10 g of CNT were added to 10 g of comminuted and pre-milled cellulose. Obtaining the material was done as in examples 0 and 0'. The results obtained by the MFT method for mass loss were 5.32% in 60 s of measurement. The exhaust gas temperature for this variant was also comparable to the other examples, reaching 114.87°C in 60 s of measurement. Analysis of the results obtained by the LOI method indicates very high effectiveness of the material obtained. The oxygen index value was 58.97%, a nearly 3-fold increase compared to the control variant,
- ³⁵ which classifies the material as non-inflammable. Tests using MLC show that the average HRR peak was 22.81 kW/m², the time to ignition was 15.67s. For variants containing 10 g of cellulose and 20 g of EG and 10 g of cellulose and 20 g of CNT, the values of mass loss and exhaust gas temperatures measured by the MFT method were 6.35% (111,36°C) and 27.44% (211,32°C), respectively. Also, the HRR peak values in both cases were higher than for the variant containing 10 g of cellulose and 20 g of carbon additives (at a 1:1 ratio of EG to CNT). The values of these peaks were 38.06 kW/m²
- ⁴⁰ and 88.05 kW/m², respectively. These values demonstrate the positive interaction of the carbon additives used.

Example 2

- [0035] 7.5 g of EG and 7.5 g of CNT were added to 10 g of comminuted and pre-milled cellulose. Obtaining the material was done as in examples 0 and 0'. Using the test method as in example one, high efficiency was achieved. The results obtained by the MFT method for mass loss were 6.84% in 60 s of measurement. The exhaust gas temperature for this variant was significantly lower compared to example 1, and was 98.99°C at 31 s of measurement. Analysis of the results obtained by the LOI method indicates very high effectiveness of the material obtained. The oxygen index value was 46.15%, classifying the material as non-inflammable. Tests using MLC show that the HRR peak value was 36.43 kW/m²,
- 50 the time to ignition was 9.33 seconds. For variants containing the same amount of cellulose and the same amount of single carbon additives, for the variant containing 15 g EG, the mass loss measured by the MLC method was 7.91% reaching a maximum exhaust gas temperature of 102.6°C. For the variant containing 15 g of CNT, the values were 29.26% and 202.09°C, respectively. The LOI values also classified these materials as non-inflammable. The HRR peak values for each additive were higher than for the variant containing 15 g of carbon additives (1:1 ratio) and amounted
- ⁵⁵ to 46.07 kW/m² for the variant of 10 g cellulose with 15 g EG and 94.52 kW/m² for the variant of 10 g cellulose and 15 g CNT.

Example 3

[0036] A three-layer plywood containing a sheet of modified cellulose material with EG and cellulose in a 1:1 ratio as the inner layer was produced. The outer layers were face veneers made of Scots pine. Tests using MLC show the maximum HRR peak value was 214.40 kW/m² at 335 seconds, the time to ignition was 71 s. For the control test containing two layers of veneers without the middle layer and glued with the same resin, the HRR peak value was 330.96 kW /m² at 210 seconds, time to ignition was 54 s.

Example 4

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[0037] A two-layer plywood was produced containing a sheet of modified cellulose material with EG, CNT and cellulose in a 1:1:1 ratio as one outer layer. The second outer layer was a venner of common pine wood. Tests using MLC show, the maximum HRR peak value was 41.27 280kW/m² at 280 seconds, the time to ignition was 207 seconds.

15 Example 5

[0038] A three-layer plywood was produced containing a sheet of modified cellulose material with EG, CNT and cellulose in a 1:1:1 ratio as the inner layer. The outer layers were face veneers made of Scots pine. Tests using MLC show, the maximum HRR peak value was 154.07 kW/m² at 160 seconds, the time to ignition was 48 seconds. The control test was the same as in Example 7.

Example 6

[0039] A three-layer plywood was produced containing a sheet of modified cellulose material with EG and cellulose in a 1:1 ratio as the inner layer. The outer layers were face veneers made of Scots pine. The sheet contained half as many components as example 7. Tests using MLC show, the maximum HRR peak value was 194.35 kW/m² at 265 seconds, the time to ignition was 51s. The control sample was the same as in example 7.

Example 7

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[0040] A three-layer plywood was produced containing a sheet of modified cellulose material produced on an industrial scale (based on the description of composite layer manufacturing) with EG and cellulose in a ratio of about 1:5 as the inner layer. The outer layers were face veneers made of Scots pine. Tests using MLC show, the maximum HRR peak value was 241.88 kW/m² at 285 seconds, the time to ignition was 113 s. The control test was the same as in example 7.

Example 8

[0041] A four-layer plywood was produced containing sheets of modified cellulose material produced on an industrial scale (based on the description of composite layer manufacturing) with EG and cellulose in a ratio of about 1:5 as the two inner layers. The outer layers were face veneers made of Scots pine. Tests using MLC show, the maximum HRR peak value was 231.19 kW/m² at 345 seconds, the time to ignition was 52 s. The control test was the same as in example 7.

Example 9

⁴⁵ **[0042]** A two-layer plywood was produced containing a sheet of modified cellulose material produced on an industrial scale (based on the description of composite layer manufacturing) with EG and cellulose in a ratio of about 1:5 as the outer layer. The second outer layer was a venner of common pine wood. Tests using MLC show, the maximum HRR peak value was 163.01 kW/m² at 190 seconds, the time to ignition was 51 s.

50 Example 10

[0043] A three-layer plywood was produced containing a sheet of modified cellulose material produced on an industrial scale (based on the description of composite layer manufacturing) with EG and cellulose in a ratio of about 1:5 as the inner layer. The outer layers were face veneers made of Scots pine. Tests using MLC show, the maximum HRR peak

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inner layer. The outer layers were face veneers made of Scots pine. Tests using MLC show, the maximum HRR peak value was 338.37 kW/m² at 255 seconds, the time to ignition was 83 s. For the control test containing two layers of veneers without a middle layer and glued with the same starch adhesive, the HRR peak value was 229.71 kW/m² at 295 seconds, the time to ignition was 82 s.

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Example 11

[0044] A three-layer plywood was produced containing a sheet of modified cellulose material produced on an industrial scale (based on the description of composite layer manufacturing) with EG and cellulose in a ratio of about 1:5 as the inner layer. The outer layers were face veneers made of Scots pine. Tests using MLC show, the maximum HRR peak value was 212.31 kW/m² at 290 seconds, the time to ignition was 81 s. The control test was the same as in example 17.

Example 12

¹⁰ **[0045]** A three-layer plywood was produced containing a sheet of modified cellulose material produced on an industrial scale (based on the description of composite layer manufacturing) with EG and cellulose in a ratio of about 1:7.5 as the inner layer. The outer layers were face veneers made of Scots pine. Tests using MLC show, the maximum HRR peak value was 260.95 kW/m² at 360 seconds, the time to ignition was 70 s. The control test was the same as in example 17.

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Claims

- 1. A method of producing a plate composite with the use of cellulose and carbon particles with increased fire resistance characterized in that
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1.1. particles rich in elemental carbon, i.e. with a carbon content >90%, selected from expanding graphite (EG) and carbon nanotubes (CNT), are dispersed in water in weight ratios ranging from 0.2:1 to 1:0.2. in an aqueous system, the weight ratio of carbon particles to water is from 1:1 to 100:1.

- 1.2. cellulose pulp with a concentration of 1-25% in water is produced
- 1.3. a dispersed suspension of carbon particles is introduced into the cellulose pulp.
 - 1.4. a composite is formed on a paper machine
 - 1.5. the composite is dried and pressed

wherein the EG/CNT dispersion is kept in water for a period of 24 hours, and further the resulting particle dispersion is homogenized for 30-45 s by means of a homogenizer with a rotation speed from 400 to 4000 rpm until a homogeneous water dispersion is obtained and the homogeneous particle dispersion is added to the cellulose pulp produced in parallel in an amount of 1-75% by weight based on the total mass of the product and the mixture is further grounded for 10 s in the refiner, and the cellulose pulp is produced in such a way that the cellulose in the form of sheets is initially comminuted into particles size 1-4 cm², soaked for 24 hours in deionized water, the weight

- ³⁵ ratio of cellulose to water is 1:10, and after 24 hours the cellulose is defibrated in a defiberizer and water is added until the total cellulose to water ratio is at the level of 1:50, then the homogeneous mass is transferred to a sheet forming apparatus and diluted with water to a mass concentration ranging from 2-20%, and cellulose-carbon sheets are formed.
- 40 2. A Method according to claim 1, characterized in that the EG particle size is in the range of 10 1000 micrometers,
 - **3.** A method according to claim 1, **characterized in that** the average length of the CNT is 1.5 μm and the diameter is 9.5 *10-9 m.
- **45 4.** A Method according to claim 1, or 2 or 3, **characterized in that** the cellulose pulp is diluted with water in an amount of 7.5%,
- 5. A method according to claim 1, or 2, or 3, or 4, characterized in that the forming of the sheets is preceded by intensive mixing of the dispersion by the barbotage method, i.e., by passing air through the forming sieve from underneath at a pressure of 0.2-0.4 bar, and then the pulp is filtered out on a sieve with a mesh size of max. 0.25 mm, and then the formed sheets are then transferred to a dryer, where simultaneously excess water is sucked out and the sheet is pressed at a maximum temperature of 97°C in time up to 600 s at a pressure of max. -97kPa.

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PHOTOGRAPHIC DOCUMENTATION



Photo 1. Cellulose-carbon layer as a product in itself



Photo 2. So-called three-layer plywood with outer layers of wood face veneers and a cellulose-carbon core



Photo 3 So-called five-layer plywood with outer layers of cellulose-carbon material and three inner layers of carbon-cellulose material





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EUROPEAN SEARCH REPORT

Application Number

EP 23 20 9927

		DOCUMENTS CONSID	ERED TO BE RELEVANT		
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