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(54) Title: METHOD FOR EXTRACTING AND RECYCLING WASTE CHEMICALLY TREATED WOOD

(57) Abstract: A continuous process for the extraction, decontamination and defibration of waste creosote-, chromated copper arsenate- or pentachlorophenol-treated wood has been developed, which is based on a one-step chemical-thermal-mechanical treatment that allows the production of fibres suitable for the formation of lignocellulosic composite materials.

**METHOD FOR EXTRACTING AND RECYCLING
WASTE CHEMICALLY TREATED WOOD**

5 This invention relates to the extraction, decontamination and defibration of waste chemically treated (contaminated) wood with a one-step process that yields fibres suitable for the production of lignocellulosic composite materials.

10

 By chemically treated wood is meant exterior-use wood which has been impregnated in the past with chemicals (i.e. preservatives) - some of them being hazardous - such as creosote, salts of chromium, copper and arsenic
15 (CCA), pentachlorophenol (PCP) and others. Such a chemical treatment is carried out in order to protect wood and make it durable against long term deterioration, weathering and natural ageing. In general, chemically treated (contaminated) wood has been used in the
20 applications of construction and garden timber, electricity poles, telecommunication poles, railway sleepers, posts, etc.

 Gilbert et al. (U.S. 5.262.004, 1993) developed a
25 method for extracting chemical preservatives from treated wood by chipping, impregnating with alkali, treating with saturated steam, explosive decompression and refining in a crusher permitting the grinding of wood. This technology never reached to the pilot-scale level due to
30 its multiple-step operation and complexity.

 Levien et al. (U.S. 5.364.475, 1994) invented a process based on a supercritical fluid extraction for recycling waste treated wood. Such wood is chipped and stripped so that the core can separately be processed
35 since it contains less or no contaminants. The rest of it

is subjected to a supercritical fluid (SCF) treatment at high and moderate temperatures. Main disadvantage of the process is the use of carbon dioxide and methanol as modifiers, while the equipment required is very
5 expensive.

Fransham et al. (U.S. 5.378.323, 1995) developed a method and apparatus for removing oil- and tar- based wood preservatives from sawdust. The efficiency of the method is questionable, while it requires high energy
10 consumption for shaving and dusting the treated wood.

Korfiatis and Pal (U.S. 5.629.199, 1997) claimed a sonically enhanced method for removing creosote and PCP from treated wood products. In this process, treated wood is disintegrated to produce chips that are then contacted
15 with an organic solvent, preferably methanol, and sonicated to extract the contaminants. The efficiency of the process is limited to an extraction degree of approx. 94-96% which is obtained after long treatment times (10-12 hours).

20 Ruddick and Cui (U.S. 5.476.975, 1995) developed a method for extracting organic toxic contaminants such as PCP, polychlorinated dibenzo-p-dioxins, etc. from wood with a supercritical fluid such as carbon dioxide and use of an entrainer such as methanol or ethanol. The process
25 requires times of 4-5 hours for a relatively efficient extraction and applies very high pressures.

Portier et al. (Microbial-assisted remediation of creosote- and pentachlorophenol- treated wood products, Journal of Industrial Microbiology, Vol.17, 1996, p. 1-5)
30 reported on a new lab scale recycling method for waste creosote- and pentachlorophenol- treated wood, that consists of an extraction with methanol and a bio-

polishing with a microbial consortium containing specific adapted strains.

Michanickl and Boehme (U.S. 5.804.035, 1998) developed a process for recycling waste particleboards and fibreboards. In this process, which is a discontinuous process, chipped waste boards are treated with steam and additives such as urea and dilute mineral acids at temperatures around 120°C. The chipped boards disintegrate completely. Thereafter, this treated material is dried in a conventional dryer and processed as usual for the production of particleboards. No experience exists on using waste treated wood. The process is used today industrially to recycle only waste urea-formaldehyde bonded particleboards.

In the present application, it is described a method for extracting, decontaminating and defibrating waste chemically treated wood of different types by subjecting it to a chemical-thermal treatment at from 40 to 100°C accompanied by a mechanical treatment with high shear forces which defibrate the wood. The resulting clean and extracted fibres can be formed into composites, for example, dry process fibreboard, wet process low density fibreboard (softboard), wet process high density fibreboard (hardboard), special particleboard having such recycled fibres in the core layer, and other moulded products, by bonding, if necessary, with conventional synthetic resins. The extent of the high shear treatment, temperature and composition of chemical reagents required for the efficient extraction depend on the type of contaminant, degree of contamination, type of wood as well as on the lignocellulosic composite material to be produced. The lignocellulosic composites are bonded, if necessary, with synthetic adhesives such as urea-formaldehyde resins (UF),

melamine-urea-formaldehyde resins (MUF), phenol-formaldehyde resins (PF), melamine-urea-phenol-formaldehyde resins (MUPF), melamine-formaldehyde resins (MF), isocyanate binders (PMDI) and others.

5

Accordingly to the invention, therefore, there is provided a one-step method for extracting and decontaminating waste chemically treated wood such as creosote-, CCA-, PCP- treated etc., and simultaneously
10 defibrating it to fibres suitable for the manufacture of lignocellulosic composite materials. The treatment is carried out in alkaline conditions at 40° to 100°C accompanied by the application of high shear forces on the wood in a low pressure environment.

15

The method involves the treatment of waste chemically treated wood at from 40°C to 100°C. Most preferably the range is between 60°C to 80°C.

20 A typical operating pressure in the system would be from 1 to 2 atm.

The present method can be carried out in any high shear device at shearing intensities in the range of 250-
25 300 kWh/ton of dry wood. The treatment according to the invention is thus conducted in a high shear-mixing device operated at low pressure close to the atmospheric (1 atm) under specific chemical, thermal and mechanical conditions. Use of a conventional twin-screw extruder device in
30 accordance with a preferred embodiment of the invention provides the requisite high shear application.

The high shear forces to be applied depend on the wood material and the types of chemicals added to the substrate.
35 Moreover, dilute alkali like sodium hydroxide or sodium

5 sulphite along with special emulsifiers like o,m,p, dodecyl sulfonic acid, or additives of chelatant, or sequestrants like ethylene diamine tetracetate are usually applied. In overall, the aforesaid reagents are used in the form of water solution or suspension in quantities between 0.01-5% based on dry wood.

Following the defibration, and for dry processes, the fibres produced can be washed, neutralised (if necessary),
10 blended with a synthetic resin and additives, and finally dried using conventional air dryers (e.g. flash dryers) in a continuous process. From then onwards, the dried fibres follow the conventional procedure as for the production of medium density fibreboards. Alternatively, and for the
15 production of wet process fibreboards, the known conventional processes can be similarly applied, following the process in the high shear device.

The starting materials (wood chips) can be obtained by
20 mechanically disintegrating waste chemically treated wood, i.e. timbers, poles, posts, in conventional hammermills.

The invention is illustrated by the examples which follow, given without limitation:

25

EXAMPLE 1

A CCA-treated post was disintegrated in chips by utilising a conventional hammermill. 10 kg of such chips
30 were introduced in a lab scale twin-screw extruder, operated at atmospheric pressure, extracted and defibrated using an alkaline system of 0.4% sodium hydroxide (on dry wood) and 0.2% ethylene diamine tetracetate (on dry wood) at 80°C. This alkaline
35 treatment was very efficient and the extraction of

contaminated wood in the extruder removed 99.8% of copper and chromium and 99.9% of arsenic.

EXAMPLE 2

5

A waste creosote-treated electricity pole of an approx. 25% degree of contamination (on the dry basis) was disintegrated in chips by utilising a conventional lab chipper. 10 kg of such chips were introduced in the mentioned extruder, extracted, decontaminated and defibrated using an alkaline system of 0.4% sodium hydroxide (on dry wood) and 0.1% o,m,p dodecyl sulfonic acid (on dry wood) at 80°C. This alkaline extraction removed approx. 99% of all creosote compounds.

15

EXAMPLE 3

The fibres produced from Example 2 were dried and used for the production of lab-scale MDF fibreboards of 16mm thickness after mixing with a conventional UF resin. The resin level employed was 12%, the pressing temperature was 200°C, the pressing time was 15sec/mm and the press pressure was 35Kg/cm². Three replicate boards were produced and their properties were subsequently determined. The values of board properties are presented in the Table below.

The formaldehyde (HCHO) emission was determined by using the Perforator method. As it can be seen from the results of this test, the decontaminated fibres generated from waste creosote-treated wood gave MDF boards with quite high internal bond and bending strength properties. The quality of the boards produced in overall was very satisfactory.

35

Lab MDF boards	1	2	3
Internal bond (IB) strength, N/mm ²	0.60	0.57	0.55
Modulus of rupture (MOR), N/mm ²	20.3	21.0	19.7
24h swell, %	13.5	14.0	14.5
HCHO, mg/100g board	10.2	9.5	9.6

CLAIMS:

1. A process for extracting and decontaminating waste
chemically treated wood and simultaneously defibrating
5 it to fibres suitable for the manufacture of
lignocellulosic composite materials, in which waste
chemically treated exterior-use wood is subjected in
particle form to a combined chemical-thermal-mechanical
treatment in an aqueous alkaline system, in the
10 presence of a chemical additive which is selected from
dilute bases, emulsifiers and decontamination /
disintegration agents, at 40° to 100°C under a high-
shear force at intensities of 250-300 kWh/ton dry wood,
the system operating at a low pressure close to the
15 atmospheric.
2. A process according to claim 1, wherein the waste
chemically treated wood comprises creosote-, chromated
copper arsenate- and pentachlorophenol-treated wood.
20
3. A process according to any one of claims 1 and 2 in
which the chemical-thermal-mechanical treatment is
carried out in a twin-screw extruder.
- 25 4. A process according to any one of claims 1 to 3 in
which waste chemically treated wood is mechanically
disintegrated into chips before the chemical-thermal-
mechanical treatment.
- 30 5. A process according to any one of claims 1 to 3,
wherein the waste chemically treated wood is extracted,
decontaminated and defibrated in the presence of 0.01-
5% by weight sodium hydroxide.

6. A process according to any one of claims 1 to 3, wherein the waste chemically treated wood is extracted, decontaminated and defibrated in the presence of 0.01-5% by weight sodium sulphite.

5

7. A process according to any one of claims 1 to 3, wherein the waste chemically treated wood is extracted, decontaminated and defibrated in the presence of o,m,p dodecyl sulfonic acid as an emulsifier.

10

8. A process according to any one of claims 1 to 3, wherein the waste chemically treated wood is extracted, decontaminated and defibrated in the presence of additives of chelatant.

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9. A process according to any one of claims 1 to 3, wherein the waste chemically treated wood is extracted, decontaminated and defibrated in the presence of ethylene diamine tetracetate as sequestrant.

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GR 00/00020

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 B27K3/00 A62D3/00 B27N1/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B27K A62D B27L B27N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

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Further documents are listed in the continuation of box C. Patent family members are listed in annex.

° Special categories of cited documents :

<p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p>	<p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p>
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Date of the actual completion of the international search 7 September 2000	Date of mailing of the international search report 19/09/2000
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Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Dalkafouki, A
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INTERNATIONAL SEARCH REPORT

International Application No PCT/GR 00/00020

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
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