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THE INTERNATIONAL RESEARCH GROUP ON WOOD PROTECTION

Section 5

Environmental aspects

Environmental characterisation of retification process by-products (liquid and gaseous wastes)

by

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ABSTRACT

In order to reduce environmental risks during the service life of the treated wood and to find new alternative developments on the durability of wood, some research and technology development have been made on thermal treatment. The retification process is one of these processes. The retification process induces chemical modification of the lignin and cellulosic components and modifies the intrinsic properties of wood : efficient increases the durability against fungi and insects, increases of the dimensional stability, decrease of the mechanical properties. The interest of this process is to reduce the environmental impact during the service life. In order to confirm the high interest of this process for the reduction of the environmental impact, an environmental characterisation of wastes on pilot plant have been carried out. Chemical analysis on gaseous and liquid effluents have been performed. An energetic assessment has been realised. The results indicate the high interest of this process in terms of possible biodegradable wastes and chemical valorisation interest, interest on energetic consumption in comparison with other wood processing treatment, interest on using retification treated wood in flooring according to indoor air quality requirements.

Keywords : wood, thermal treatment, wastes, liquid, gaseous, volatile organic compounds, process, energy

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1- INTRODUCTION AND OBJECTIVES

Thermal treatment of wood by the retification process is known to be efficient to increase the durability of wood against fungi and insects except termites and dimensional stability. The development of this technology is growing up and some applications are very interesting as cladding and decking for example. Previous studies on maritime pine have demonstrated that with increase of the durability. The use for structural components in building is not recommended considering the decrease of the mechanical properties.

On another hand, this process is really innovative considering the increase of the intrinsic properties and an complementary qualification on the environment impacts applied on the process and the treated wood will be useful for a complete overview.

In order to complete the efficiency of this innovative process and to have an environmental assessment of the retification process, the general objectives of this research are :

- to assess the environmental impact of an innovative thermal treatment
- to assess the ecotoxicity of liquid wastes and to assess the VOC emission amounts
- to contribute to disseminate information on environmental and regulatory issues

By-products as liquids and gaseous wastes have been collected during the treatment of 8 cubic meter (two batches by tested wood specie) in the Pilot Plant (HTT, Now Process; south of France, Soustons). The chemical analysis and the ecotoxicity assessment have been carried out then in three laboratories.

Two tested wood species have been studied : poplar and maritime pine



Figure 1 : HTT Plant – NOW Process (retification)

The methodology is defined to assess the environmental impacts of this innovative process by the following studies

Concerning the process : - Collect liquid and gaseous wastes samples on site during the thermal treatment (on two loads)

- Determination of chemical compounds produced during the thermal process by chemical analysis
- Chemical and Ecotoxicological Characterisations of liquid wastes
- Gaseous effluent measurement
- Energetic consumption measurements, comparison with others processes.

- Definition of possible value products (from liquid wastes) obtained by hydrodistillation
- Concerning the thermal treated wood
- Chemical analysis : HAP detection
- VOC characterisation (emitted by wood) (according XP ENV 13419-1 standard)

2- RESULTS AND DISCUSSION

2.1-INTRODUCTION

The experiments based on the use of TG-DSC FTIR technique can allow to improve the chemical modification understanding generated by thermal treatment. Some major chemicals are produced as carbon dioxide, carbon monoxide, acetic acid. It is observed an heterogeneous behaviour of resinous wood species (maritime pine) instead of poplar specie. It could be also observed the effect of hemicelluloses on the generated chemical compounds.

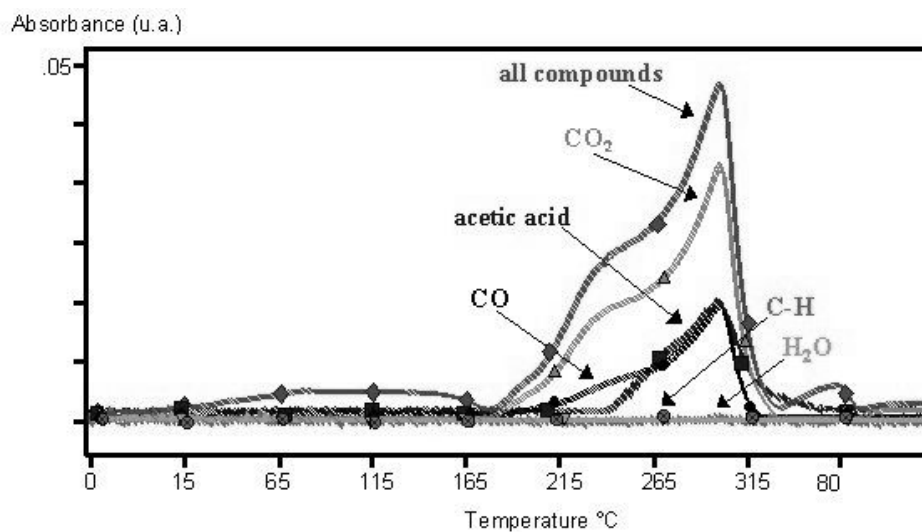


Figure 2 : Chemicals produced during the poplar pyrolysis (TG-DSC analysis)

The chemical analysis indicates that some chemicals as water, acetic acid, furfural, methanol, carbonyl compounds, carboxylic compounds and phenols are produced during the thermal process and are concentrated in liquid wastes (after cooling by water spray).

The chemical content of liquid wastes is dependant of the wood species and the temperature stage. The amount of produced chemical compounds increase during the retification step and during the cooling step. Few amount compounds are produced during the drying step. It is observed an high DBO/DCO levels (oxygen biological demand and oxygen chemical demand) of generated acidic liquid wastes.

2.2- CHEMICAL ANALYSIS OF LIQUID WASTES

Liquid wastes samples have been collected during two campaigns of treatment made on two wood species on the thermal treatment site (HTT in south of France), representative of other sites. The tested wood species are maritime pine and poplar (two loads of 8 m3 each).

The wastes have been collected during (1) the total period of the treatment (Pt) and fractionated time periods (four time periods : P0 and P2 as drying steps, P3 as retification step and P4 as cooling step).

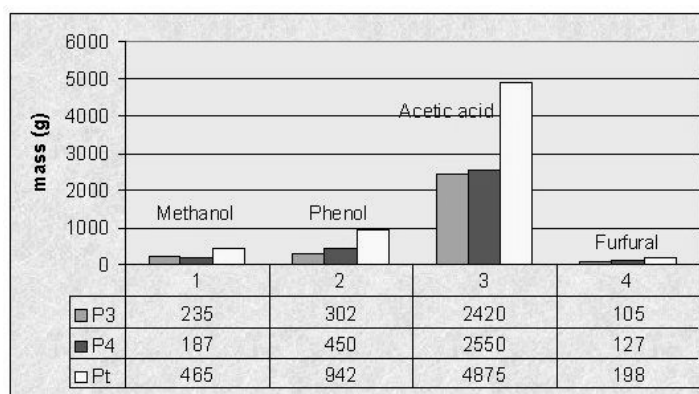
The results concerning the liquid waste production for each thermally treated wood species (for all process steps (PT) and for each process steps (P0 to P4)) are summarized in the Table

1. The liquid generated by the thermal treatment during these four steps including the cooling step produce around 500 liters if liquid for one treatment of 8 cubic meter of wood. The P3 (retification step) produce collected liquids for each time period are analysed by GC-MS and results are indicated in the Graphs. Some chemicals have been detected : terpenes, carbonyl compounds, esters, carboxylic acids, furans, phenolic compounds). Four chemical compounds

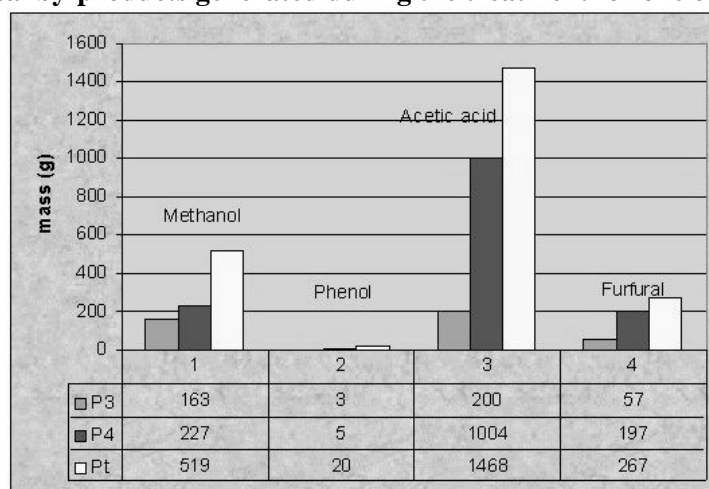
**Table 1 : liquid waste production
(expressed in Kg for one treatment of 8 cubic meter of wood)**

| phase P | liquid waste (Kg) poplar | liquid waste (Kg) maritime pine |
|---------|--------------------------|---------------------------------|
| P0 | 0,9 | 1,1 |
| P1 | 82,7 | 127,3 |
| P2 | 159,3 | 314,2 |
| P3 | 117,6 | 75,6 |
| P4 | 106,4 | 84,4 |
| Total | 467,1 | 602,6 |
| PT | 377,9 | 562 |

have detected in large amounts. The P3 and P4 steps produce 80-91% of the total amount of the chemicals produced.



Graph 1 : Chemical by-products generated during the treatment for one cubic meter (poplar)



Graph 2 : Chemical by-products generated during the treatment for one cubic meter (maritime pine)

The results show that the treatment of poplar produce more chemicals (factor 3x) than the treatment of maritime pine, with respectively 6 480 g of major chemical compounds for one cubic meter of poplar and 2 294 g of major chemical compounds for one cubic meter of maritime pine (based on Pt).

For poplar : Acetic acid represents more than 75% of the total amounts produced. Furfural and acetic acid represent more than 80% of the total produced compounds. Phenol is more produced during the treatment of poplar. During the drying steps (P1 and P2), terpenes, furanic compounds and other phenolic compounds are produced.

For maritime pine : Acetic acid represents more than 64% of the total amount produced. Terpenes (α -terpineol) are produced during the steps P2 and P3.

The level of methanol is quite similar for the two species

2.3- ECOTOXICOLOGICAL ASSESSMENT OF PRODUCED LIQUID WASTES

Measurements have been made according to the following listed methods.

- Determination of DBO₅/DCO (NF EN 1899-1) : oxygen demand expressed in mg/l for bacteria to eliminate organic compounds. The DBO₅ is indicated for a biological oxidation in 5 days.
- Determination DCO (NF T 90-101) : chemical oxygen demand expressed in mg/l by oxidant of organic compounds in water.
- Determination of hydrocarbons according NF T 90-114
- Determination of insoluble organic compounds in water (MES) according NF T 90-105-2
- Assessment of the Ecotoxicity against *Daphnia magna* (expressed in CE50) according NF EN ISO—Physical parameters : pH, ratio DBO/DCO.

The results of the Ecotoxicity assessment are summarized in the Tables 2 and 3.

The main results can be indicated as follows.

- High acidity of produced liquid with a pH comprising between 2 and 3.
- High content of organic compounds in the Steps 3 and 4 of the process (retification phase and cooling) with a generation of 91-97 % of the total DBO and DCO.
- High level of ecotoxicity (*Daphnia magna*)
- Measured DBO and DCO levels higher than levels for sending directly liquid wastes to a purification station.
 - factor 180 for the treatment of Poplar
 - factor 35 for the treatment of Maritime pine
- Possible biodegradation by bacteria : ratio DBO/DCO > 0.6 for the two wood treatments

Physico-chemical characterisation and ecotoxicity assessment

| Poplar | Regulation values (doc:14 june 1994) | P1 | P2 | P3 | P4 | Σ P1 to P4 | PT |
|--|---|-----------|--------|-----------------------|-----------------------|---------------|-------------------------------------|
| pH | 5.5-8.5 | 3.1 | 2.9 | 2.3 | 2.2 | / | 2.4 |
| DCO mg/l | 2000 | 7 840 | 18 700 | 378 000 | 597 000 | / | 365 000 |
| DBO mg/l | 800 | 4 860 | 8 800 | 300 000 | 338 000 | / | 144 000 |
| Ratio DBO/DCO | / | 0.62 | 0.47 | 0.79 | 0.57 | / | 0.39 |
| Liquid Volume (kg) | / | 82.7 | 159.3 | 117.6 | 106.4 | 466 | 377.9 |
| Liquid Volume (l) | / | 83.7 | 161.1 | 116.5 | 105.9 | 467.2 | 374.3 |
| kg DBO/day | 20 | 0.4 | 1.4 | 35.0 | 35.8 | 72.6 | 54.4 |
| kg DCO/day | 120 | 0.6 | 3.0 | 44.0 | 63.2 | 110.8 | 136.6 |
| MES in mg/l | 600 | 2.7 | 2.7 | 1567.3* | 8045.6* | / | 395.7 |
| Inhibition agents in % (test <i>daphnia magna</i>) | | / | / | / | / | | CE50-48h = 0.07 % (0.06-0.09) |
| Total Hydrocarbons | 10 mg/l | 36.4 mg/l | 7 mg/l | 0.4 %** 4 000 mg/l | 1.4 %** 1 400 mg/l | | 1.5 mg/l |

| Maritime pine | Regulation values (14 june 1994) | P1 | P2 | P3 | P4 | Σ P1 To P4 | PT |
|--|-------------------------------------|------------|----------|-----------------------|-----------------------|---------------|-------------------------------------|
| pH | 5.5-8.5 | 3.0 | 2.9 | 2.5 | 2.3 | / | 2.6 |
| DCO mg/l | 2000 | 7 430 | 9 510 | 130 000 | 261 000 | / | 72 400 |
| DBO mg/l | 800 | 4250 | 4 300 | 83 800 | 162 000 | / | 50 000 |
| Ratio DBO/DCO | / | | 0.45 | 0.64 | 0.62 | / | 0.69 |
| Liquid Volume (kg) | / | 127.3 | 314.2 | 75.6 | 84.4 | 601.5 | 562 |
| Liquid Volume (l) | / | 127.3 | 319.7 | 76 | 83.4 | 606.4 | 564.8 |
| kg DBO/jour | 20 | 0.5 | 1.4 | 6.4 | 13.5 | 21.8 | 28.2 |
| kg DCO/jour | 120 | 0.9 | 3.0 | 9.8 | 21.8 | 35.5 | 40.9 |
| MES en mg/l | 600 | 4.8 | 25.1 | 3155.7* | 298.5 | / | 34.8 |
| Inhibition agents in % (test <i>daphnia magna</i>) | | | | | | | CE50-48h = 0.33 % (0.30-0.37) |
| Total Hydrocarbons | 10 mg/l | 184.9 mg/l | 1.6 mg/l | 2.7 %** 2 700 mg/l | 0.7 %** 7 000 mg/l | | 5.1 mg/l |

2.4 – DETERMINATION OF HAP IN LIQUID AND GASEOUS WASTES AND IN TREATED WOOD

All 16 HAP compounds according to the EPA list have been searched in liquids and also in the wood. Any HAP have been detected in all by-products (<5 ppb for liquids and < 20 ng/m³ for gaseous wastes; < 30 ppb for the analysis of wood on three depths 0-1 mm; 2-3 mm; 3-5 mm)

The Volatil Organic Compounds (VOC) are detected according to the standard XP ENV 13419-1 (Wood-based products for building, march 2000) with two experimental procedures (two chambers : test chamber 51 L CLIMPAQ and test chamber 1 m³ VCE on 28 days at 23°C).

TD/GC/MS/FID analysis. The emitted VOC and levels are compared with the health criteria (indoor air) of the guidelines relative to flooring applications



Figure 3 : Description of the test equipments and procedures

1 m³ VCE Equipment (used during 28 days at 23°C)
with VOC adsorption equipment

51 L CLIMPAQ Equipment

The experimental methods are indicated in Table 3:

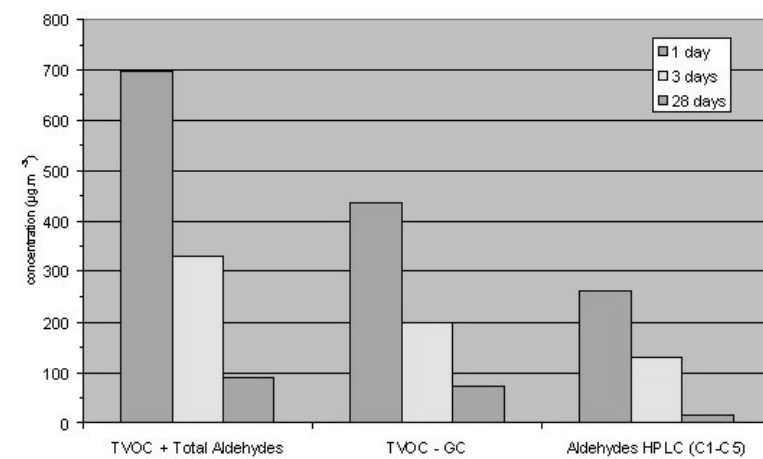
| Parameters | Methods | |
|--------------------------|---|---------------------------------------|
| | CLIMPAQ (verre) | VCE (acier inox) |
| Equipment | CLIMPAQ (verre) | VCE (acier inox) |
| Chamber volume | 0,051 m ³ | 1 m ³ |
| Temperature | 23 ± 2 °C | |
| Relative Humidity | 50 ± 5 % | |
| Sample exposed surface | 0,158 m ² | 0,418 m ² |
| Air flow | 0,20 m ³ .h ⁻¹ | 0,522 m ³ .h ⁻¹ |
| Level of air replacement | 3,9 h ⁻¹ | 0,522 h ⁻¹ |
| Load ratio | 3,1 m ² .m ⁻³ | 0,418 m ² .m ⁻³ |
| Level of ventilation | 1,25 m ³ .m ⁻² .h ⁻¹ | |
| Duration | 28 days | |

Table 3 : Experimental parameters

•VOC analysis (for the treatment of poplar) in total VOC and Aldehydes

The major components are acetic acid, acetaldehyde, propionic acid, furfural. After 28 days at 23°C, the detected concentrations in TVOC (equivalent toluene) and Aldehydes are respectively 74 µg/m³ and 16 µg/m³.

The emission amounts for poplar after 28 days : 140 µg/m³ of acetic acid, 25 µg/m³ of phenol, 16 µg/m³ of acetaldehyde, 16 µg/m³ of propionic acid and 15 µg/m³ of furfural .

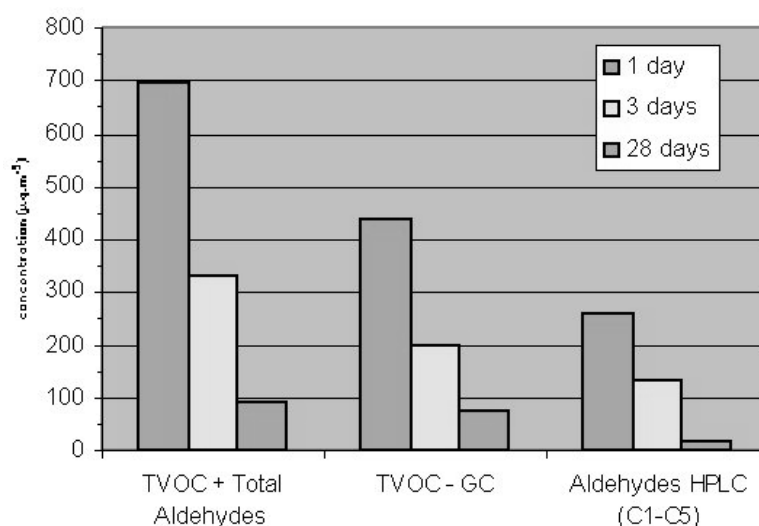


Graph 3 : VOC analysis (for the treatment of poplar)

•VOC analysis (for the treatment of maritime pine) in total VOC and Aldehydes

The major components are acetic acid, furfural, acetaldehyde, 1-hydroxy-2-propanone, propionic acid and formaldehyde. After 28 days at 23°C, the detected concentrations in TVOC (equivalent toluene) and Aldehydes are respectively 52 µg/m³ and 11 µg/m³.

The emission of treated maritime pine is after 28 days less than the treatment of poplar : 57 µg/m³ of acetic acid, 45 µg/m³ of furfural, 7 µg/m³ of acetaldehyde, 4 µg/m³ of formaldehyde, 4 µg/m³ of hydroxyl-2-propanone.



Graph 4 : VOC analysis (for the treatment of maritime pine)

•Health assessment (protocols et definition of classification)

This evaluation is based on the use of a protocol according to the health evaluation of emission come from flooring products (ECA-IAQ; « Indoor Air Quality and its Impact on

Man », 1997). The method allows to measure the concentration in chamber [VOC]_i expressed in exposure concentrations in a model chamber [VOC]_{exp}

$[VOC]_{exp} = q_c/q_e \cdot [VOC]_i$; q_c = specific ventilation rate ; q_e = specific ventilation rate according to the exposure scenario.

List of criteria to evaluate the health assessment

- Duration time = 28 days
- [benzene] < 2.5 µg/m³
- [Vinyl Chlorure monomere] < 10 µg/m³
- [2-butoxyethanol] < 600 µg/m³
- TVOC < 200=g/m³ *Classification of building construction products*

–A : all [C] are below than 0.5 xtime of limit values

–B : all [C] are below than limit values

–C : one of the [C] is up than limit values

• Summary of Results of the health assessment

| CLASS | J1 Bz | J1 VC | J1 GE 1a | J1 BG | J3 TVOC | J28 Bz | J28 VC | J1 GE 1a | J28 BG | J28 TVOC | J28 ΣCi/LCI | J28 ΣCni | CLASS |
|------------------------------|----------|----------|-------------|----------|------------|-----------|-----------|-------------|-----------|-------------|----------------|-------------|----------|
| A | < 12,5 | < 50 | ND | < 300 | < 2500 | < 1,25 | < 5 | ND | < 300 | < 100 | < 0,5 | < 10 | A |
| B | < 25 | < 100 | ND | < 600 | < 5000 | < 2,5 | < 10 | ND | < 600 | < 200 | < 1 | < 20 | B |
| C | > 25 | > 100 | ND | > 600 | > 5000 | > 2,5 | > 10 | ND | > 600 | > 200 | > 1 | > 20 | C |
| Treated Poplar | ND | ND | ND | ND | 200 | ND | ND | ND | ND | 74 | 1,3 | 6,7 | C |
| Treated Maritime pine | ND | ND | ND | ND | 90 | ND | ND | ND | ND | 52 | 0,97 | 0 | B |

ND = not detected

Bz = Benzene

VC = Vinyl Chlorure monomere

GE 1a = Glycol Ethers 1a

BG = 2-Butoxyethanol

Table 4 : Samples classification of treated Poplar and treated Maritime pine for the health assessment according -IAQ (1997) (Exposure Concentrations expressed in µg.m⁻³)

The concentrations limits (LCI) according ECA-IAQ (1997) are respectively for : Phenol
400 µg/m³

- Acetaldehyde 40 µg/m³
- Acetic acid 250 µg/m³
- Propionic acid 300 µg/m³
- Furfural 79 µg/m³

It is important to note that the targeted chemicals listed in the regulation are not detected (see table 4). Only the Ci/LCI ratio is up. The results (totalCi/LCI (TVOC; 28 days)) indicated in Table 4 according these experiments and regulation can indicate that the maritime pine can be classified in the class B and can used for flooring applications with an acceptable risk considering health safety.

The treated poplar is classified in class C. One of the exposure concentration is up than limit values.

2.6- ENERGETIC ASSESSMENT

The objective of the energetic assessment is to know the energy requirement, expressed in MJ, to produce one cubic meter of thermal treated wood.

The energetic consumption of this thermal process is respectively 1268 MJ/m³ for the treatment of maritime pine and 755 MJ/m³ for the treatment of poplar. This difference is due to the high calorific absorption factor for the maritime pine.

The specific energetic consumption is defined. The specific energetic consumption is the consumption of energy to transform the raw material in a end use product (the transport and packaging are not included). The specific energy consumption (SEC) include the energy for drying, for the sawing and for the thermal treatment. The results are summarized in the Table 5.

| Wood species | Sawing + Drying MJ/kg (source CTBA) | Retification MJ/kg | Specific Energetic Consumption (SEC) MJ/kg |
|---------------|---|-----------------------|--|
| Maritime pine | 1,5 | 2,3 | 3,8 |
| Poplar | 1,9 | 1,8 | 3,7 |

Table 5 : Specific Energetic Consumption (MJ/kg) for the production of end uses of thermal treated wood

It is interesting to compare with others SEC for the transformation of building products as :

| | |
|----------------|----------|
| Wood | 2 MJ/kg |
| Particuleboard | 4 MJ/kg |
| Plywood | 7 MJ/kg |
| MDF panels | 11 MJ/kg |
| Extruded PVC | 41 MJ/kg |
| Aluminium | 74 MJ/kg |

In comparison, the energetic consumption of this process is equivalent to produce particule board and less than plywood and MDF panels.

3- CONCLUSION

This approach to allow an innovative and positive assessment of a thermal process used to increase the general properties of wood can be summarized as follows :

- Concerning the thermal treated wood : levels on TVOC emitted are an acceptable range for flooring applications, especially for maritime pine
- Concerning the liquid and gaseous by-products generated during the process
 - o High acidity of produced liquid with a pH between 2 and 3.
 - o High content of organic compounds in the Steps 3 and 4 of the process (retification phase and cooling) with a generation of 91-97 % of the total DBO and DCO.
 - o Measured DBO and DCO levels more higher than authorised levels for sending directly liquid wastes to a purification station.
 - factor 180 for the treatment of Poplar
 - factor 35 for the treatment of Maritime pine
 - o Possible biodegradation by bacteria : ratio DBO/DCO > 0.6 for the two wood treatments

It is necessary to treat the liquid wastes before sending to the purification station. A neutralisation of the acidic liquid is be necessary. Biodegradation on site or liquid phase separation would be preferable, in order to decrease the level of DBO and DCO.

The environmental impacts of the treatment of maritime pine are less than those of the treatment of poplar. To secure the ambient air on sites, it is also necessary to treat produced diffused gas during the treatment.

Some works have been done after this study by the industrial to bring some modifications on the process and also to elaborate some possible degradation to reach these conditions, concerning the liquid wastes :

–< 45 kg/day de DCO; < 15 kg/day de DBO5, < 15 kg/J MES. $5.5 < \text{pH} < 8.5$; $T^{\circ}\text{C} < 30^{\circ}\text{C}$

Some produced chemicals could be valorised by hydrodistillation to produce added values products;

The specific energy consumption is low and less than other wood transformation processes.

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