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Reference -

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Summary :

This action, **for UIPP** and **co-financed by DRIRE Aquitaine**, consists to realize a double bibliographic study, which aims to identify the solutions able to limit the formaldehyde release of panels.

uipp DRIRE AQUITAINE

Keywords :

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Finally, we must not forget DRIRE Aquitaine and UIPP, whose co-financing has materially helped to carry out the study.

Members of the steering committee of the study

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I. CONTEXT AND GOALS OF THE STUDY

In order to anticipate an eventual European regulation[1] and following the classification of the IARC (International Agency for Research on Cancer)[2], France has reinforced the obligations related to formaldehyde : since 1st January 2007, the « works which expose to formaldehyde » are considered as carcinogen and are consequently subject to the same regulation as carcinogens, category 1 and 2.
Consequently, the regulation requires from every industrial who use a process implementing formaldehyde or whose degradation products contain formaldehyde, a set of technical requirements for protecting the exposed or potentially exposed workers.

In this regulatory context, and given the challenges of workers health and the wider public health, **it becomes a priority to find alternative technical solutions of the use of resins based on formaldehyde in industry, including the manufacture of panels**.

Following the proposition of study filed by RESCOLL in partnership with UIPP and its members, DRIRE Aquitaine has given a grant in October 2008 for the realization of this study (in which CRAM Aquitaine is involved).

Before to begin this study, it is important to note several points:

- the panels concerned by this study are particleboards, MDF and OSB, because their manufacture uses mainly aminoplastic resins (UF[3], MUF & UFm[4]), which are the resins most concerned by the problem of formaldehyde release,
- enormous efforts have been already realized on the improvement of resins and of the formaldehyde content of panels (from about 100 mg in 1975' to less than 8 mg / 100 g of dry panel today – perforator) ; these quantities are consequently already low and it will be very difficult to decrease them more,
- a large number of researches on the substitution of formaldehyde in the panels industry has been already conducted, but there is no bibliographic compilation, which implied the need to realize this bibliographical study before any study in research and development,
- apart the binders based on isocyanates, there is today no solution to replace immediately these resins.

The goal of this study is consequently to <u>realize a synthesis of all the researches already realized on the reduction of formaldehyde emissions in the manufacturing of panels</u>, before to begin "practical" steps. The difficulty is to propose a synthesis, as exhaustive as possible, given the different possibilities.
To reach this goal, a double bibliographic approach has been followed:

- **identification of works, already realized by the French industrials,**
- **identification (as exhaustive as possible) of the technologies studied in the world.**

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[1] The formaldehyde is today classified carcinogen, category 3 (cause concern for humans, owing to possible carcinogenic effects but in respect of which the available information is not adequate for making a satisfactory assessment).
[2] In June 2004, IARC has classified formaldehyde, on the basis of new studies, as definitely carcinogenic to humans (group 1), whereas it was previously considered only as probably carcinogenic to humans (group 2A).
[3] The UF resins are the most formaldehyde emitting resins.
[4] It is UF resins, doped with melamine.

II. STATE OF THE ART ON THE USE OF AMINOPLASTIC RESINS IN THE MANUFACTURING OF PANELS

The quantity of aminoplastic resins used for the French manufacturing of particleboards and MDF is, according our estimation, between 460 000 and 500 000 t/year.

Strengths	Weaknesses
• availability of raw materials • reactivity of resins • relatively low cost of resins • mechanical performances of panels (*see standards*)	• formaldehyde release of panels, bonded with these resins, because of the poor resistance to hydrolysis of these resins (the reaction of polycondensation between urea and formaldehyde is a reversible reaction) and the presence of low quantity of free unreacted formaldehyde [*1*]

Strengths and weaknesses of aminoplastic resins (used in the manufacturing of panels)

The use of aminoplastic resins in the manufacture of panels has consequently:

- the disadvantage to emit formaldehyde : **the solutions able to limit this formaldehyde release will be consequently indentified, in the most exhaustive way,**
- different strengths (*see above*) : **it will be an important criteria for the evaluation of substitution solutions.**

The criterias to study for all the identified solutions are consequently (in addition to formaldehyde release):

- **aspects of health and safety,**
- **availability of raw materials which are required to the binders production,**
- **cost of these raw materials,**
- **reactivity of resins,**
- **performances of panels (internal bond strength, modulus of elasticity, water resistance, …).**

Many solutions having been tested in laboratory, the criteria of technology transfer (from the laboratory, to the pilot and the industry) will be added to all these criteria.

Moreover, note that the production lines are adapted to the used materials (liquid aminoplastic resins). In the case of powder binders for example (proteins, …), the modification of the production line (and of the way to work) will be a parameter to take into account.

III. RESULTS

The different solutions identified during the study are listed below (***main strengths*** & *main weaknesses*).

III.1 Chemical way

III.1.1 Interesting solutions

a) Use of scavengers/catchers in aminoplastic resins

Different scavengers/catchers have been identified:

- **urea (*relatively low cost*) addition :**
  - in solution, at the end of the adhesive formulation (after synthesis) : *deterioration of the resin performance, increase of pressing time, decrease of mechanical properties and water resistance of panels [7&8]*
  - dry, on the chips, when applying the adhesive

- **melamine [7] (1-4 %, liquid basis)[5] :** ***improvement of mechanical properties and water resistance of the panels, conservation of internal bond strength and 24 h swelling[6]*** // *cost higher than UF resins, but lower than MUF resins*

- **ammonia (post treatment catcher) :** abandoned solution, mainly because of the *incompatibility with the speed of continuous press and the risk of toxicity by inhalation of ammonia*

- **Kaurit® Plus / BASF (post treatment catcher) :** ***positive long-terme effect (6 months)***

- **CHIMAR HELLAS systems (resins UF, UMF or MUF used with or without scavenger, on MDF and particleboards, at pilot or industrial scale) [1] :** ***emissions of formaldehyde lower than 0,5 mg/L (desiccators), conservation of production parameters, no loss of productivity, no significant increase of production cost, no panels properties deterioration (improvement in several cases)***

- **resin MUF + scavenger + special hardener, tested on particleboards (industrial scale) [*ACM Wood Chemicals PLC*] :** ***formaldehyde content equal to 1,9 mg / 100 g of panel (perforator), no panels properties deterioration, no increase of pressing time, no increase of the quantity of resin required for the bonding***

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[5] This corresponds to UF resins doped/fortified with melamine (UFm or mUF resins), already used in industry.
[6] These characteristics are difficult to maintain when decreasing the ratio F/U of UF resins.

- **<u>dihyhydrazides, tested on particleboards [*14*] :</u>** ***decrease of formaldehyde emissions, with a relatively low quantity of catcher (best reduction obtained with 1 % m/m)*** // *no information about panels properties*

- **<u>sodium pyrosulfite [14] :</u>** ***strong decrease of formaldehyde emissions*** // *encapsulation necessary & long-term effects of catchers*

b) <u>Modification of UF resins by expansion agents [*15*]</u>

Two expansion agents have been added to a UF resin: trichlorofluoromethane and trichlorotrifluoromethane.
Best results have been obtained with trichlorofluoromethane : ***no change in the process, improvement of mechanical characteristics & economy of raw material***.
This study has been abandoned, but deepening would be necessary (determination of the content of expansion agent to introduce, …).
It has been realized only in **<u>laboratory</u>**.

c) <u>Use of binders based on isocyanates</u>

Different uses of isocyanates can be done:

- **<u>use of only isocyanates</u>** : it is not directly MDI (methylene diphenyl diisocyanate) or TDI (toluene diisocyanate), but pMDI (prepolymer or oligomer), which do not present the same toxicity than MDI[7]; the use of pMDI must be implemented in industrial processes with a good level of containment and direct extraction : ***the substitution is total, the isocyanates are stable once cured and give to panels excellent mechanical properties and a good water resistance*** ; on the other side, *the isocyanates are more expensive than UF resins (between 3 and 4 times)[8], the available quantities are lower and they show problems of demoulding during the implementation of resins on the presses, because of their affinity to metallic parts* [18] ; **it is today the only alternative method used in the industry (particleboards, MDF and OSB), but the development of these binders, for a total use in panels, is delicate and requires a high technological level**

- **<u>separate spray of 2 binders (aminoplastic resins / isocyanates) on the chips [*18*]</u>** : *delicate implementation because of the low content of isocyanates that is economically possible to use*

- **<u>combined use of UF resins and isocyanates[9] [*8*]</u>** : in order to overcome the problem of demoulding of isocyanates, they can be used in combination with UF resins, applied on the surface : *higher cost, related to the use of isocyanates* (very undeveloped market – maybe less than 1 %)

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[7] The toxicity of polymers is generally different, often lower than those of monomers.
[8] This difference of cost is important, even if the used quantity is about 2 times lower.
[9] The manufacture of some panels can also use isocyanates (core) in combination with PF or MUPF resins (surface).

- **modification of aminoplastic resins by isocyanates [*18*]** : ***reduction of formaldehyde emissions & improvement of mechanical properties of panels*** // *difficult process for the incorporation of TDI & success linked to the choice of isocyanate (for example, 4,4'-MDI, one of the most common isocyanates, leads to non-homogeneous resins)*

III.1.2 Other solutions

Other solutions of the chemical way have been identified, but without enough available information (*) or enough strength to consider them as interesting solutions:

- **modification of UF resins with nanoparticles [*12*]** : interesting results, but *« unknown »effects of nanoparticles on health*
- **improvement of PF resins** (color of panel, polymerization time and temperature, …)*
- **use of binders based on acrylic [*DIPP Project*[10]]** : ***no formaldehyde emission*** // *high cost, compatibility with existing treatment technologies to improve*
- **use of binders based on PVAC (polyvinyl acetate) [*DIPP Project*]** : *tested only for another application (bonding of decor papers)*
- **substitution of traditional catalysts by more efficient compounds in formulation with lower ratio [*FORINTEK*[11]]** : works only accessible by industrials, who finance them
- **improvement of hardeners [*FFIF (Finnish Forest Industries Federation)*]***
- **increase of the pressing temperature [*FFIF*]***
- **addition of additives reacting during the pressing [*FFIF*]***

III.2 Vegetable way

III.2.1 Interesting solutions

a) Use of lignin

The lignin is a natural and renewable raw material. It is present in abundant quantities and has a low cost and an important substitution potential (natural polymer network cross linked and branched).
But, *it is inert: it must be activated to make it effective as an adhesive*.

Different uses of lignin have been identified:

- **use of pure lignin [*23*] :** not interesting in industry because of its *loss of reactivity (increase of polymerization time and consequently pressing time)*

[10] The European project DIPP (« Development of Innovative Particleboard Panels for a better mechanical performance and a lower environmental impact ») gathered 25 partners (research institutes, industrials & associations), between 2005 and September 2008.
[11] FORINTEK is an institute of research on wood products in Canada.

- **use of lignin in substitution of phenol (50 %) in PF resins [24]** : the lignins produced here are linear oligomers with a low degree of polymerization ; the « PFL » resin has been tested on particleboards (**laboratory** scale) : ***decrease of formaldehyde emissions*** *// panels properties deterioration (increase of swelling, decrease of the internal bond strength, …)*

- **reactivation of lignin at high temperature (more than 300 °C)** : solution followed by Finnish people (up to a full production line), but abandoned because of the *effects of the high temperature (degradation of mechanical and physical characteristics of panel & very high energy consumption)*

- **reactivation of lignin by enzymatic oxidation :** research realized by the University of Heidelberg (Germany), then stopped ; no other information obtained

- **study « Development of adhesives from renewable resources and with low impact to health and environment for the manufacture of particleboards and MDF » / AGRICE (2007-2009)** *: confidential results*

- **mechanical reactivation of lignin by heating due to important friction :** method developed by the laboratory of ENSTIB ; promising first results, but *which do not allow again to obtain high quality products*

- **chemical reactivation of lignin by use of hydrogen peroxide (H_2O_2) and implementation with a catalyst of reaction :** semi-industrial tests in Sweden and USA [*21*] ; no other information obtained

- **reactivation of lignin, adding soda at high temperature :** no relevant information recorded

- **reactivation of lignin, modifying it genetically [*21*] :** this method could be envisaged

The lignin has been also tested in combined solutions (*see § III.2.1.f) & III.3.3*), as glyoxalized lignin[12].

b) Use of tannins

Tannins are present in large quantities in nature, and are already used for the industrial manufacture of wood panels in South America.
On the other side, *their extraction is very expensive, very pollutant (water & soil) : the commercial quantity is insufficient in Europe.*
Moreover, they have a long polymerization time, and they give weak adhesive bonds (lack of intermolecular cross-linking), difficulties for applicability and handling, and a dark color of panels.
[*23 & 25*]

[12] In the last 20 years, progresses have been realized by pre-reacting lignin with formaldehyde in order to form methyloled lignin (which can be added to phenolic resins, to form adhesives, used for the production of plywood in North America). Recently, this technology has evolved: formaldehyde is replaced by glyoxal (less toxic and less volatile aldehyde).

Different solutions have been identified:

- **<u>modification of PF resins (partial or total substitution of phenol) [6] :</u>** *this technology is not very adopted in Europe,* but ***an industrial use exists in Australia, South Africa, New-Zealand and Chili (including particleboards), with excellent mechanical properties and a high water resistance***

- **<u>modification of UF resins (DIPP Project) :</u>** no relevant information recorded

- **<u>formulation of a totally natural binder based on tannins [24] :</u>** CHIMAR HELLAS has produced particleboards (**<u>laboratory</u>** scale) with condensed tannins, associated with a hardener (chemical) : ***decrease of formaldehyde emissions & conservation of panels properties***

Tannins have been also used in combined solutions (*see § III.2.1.f) & III.3.3*).

c) Modification of UF resins by liquefied wood [26]

The University of Ljubljana and the National Institute of Chemistry, Ljubljana, Slovenia, have added liquefied wood in UF resins, for the manufacture (in **<u>laboratory</u>**) of particleboards.
An improvement of the internal bond strength and the surface soundness has been observed, without high increase of swelling. But, *the substitution is limited (addition of liquefied wood limited to 10 %).*

d) Use of vegetable proteins

(1) Use of oilseeds [21]

In the years 1995-2000, AGRICE has realized a set of researches in order to produce binders for wood based on oilseeds.
The obtained resins are usable in panels, but *no test has been realized at industrial scale and the produced quantities are (very) low compared to the needs of panels industry.*

Different solutions have been identified:

- **<u>use of rape or soya proteins[13] [23] :</u>** several studies have demonstrated the collaborating role of proteins from rape or soya cake used for example in partial or total substitution of phenol in PF resins, but these proteins have a *low water resistance and the purification of the oilseeds concentrates is expensive*
  - **<u>addition of soybean in a UF resin [24] :</u>** particleboards have been manufactured at **<u>laboratory scale</u>**, with UF resins modified with 1 % of soya ; we have observed an ***improvement of the internal bond strength and the dimensional stability of panels***, but *the European production of soya stay low*

[13] Among the oilseed proteins, those of soya are the most reactive.

- o **Purebond® (soya proteins + PAE - polyamide-epichlorhydrine) [*g & 20*] :** this product, used by GOODFELLOW and COLUMBIA (plywood & particleboards), is ***water resistant*** *and* ***can give resistant bonding for the production of fire resistant panel with total substitution of formaldehyde*** ; it is an ***equivalent system to UF resins in an economic point of view (competitive cost of panels)***, but *there can a degradation of panels by insects and the production of soybean stays low in Europe*

- o **extrusion of flour and concentrates of soya to form foams used as binders for wood [*h*]**

- o **use of hydrolysates of soya proteins (concentrates or isolates) to react with PRF or UF resins [*h*]**

- **waterborne vegetable adhesive, based on proteins of sunflower meal [*23 & 27*] :** it is a collaboration between the Toulousaine de Recherche et Développement (TRD) & the Laboratoire de Chimie Agro-industrielle (LCA) of ENSIACET (École Nationale Supérieure des Ingénieurs en Arts Chimiques et Technologiques) - Toulouse ; ***the results are promising and comparable to reference binders,*** but *the binder is dark* ; works continue with a study of technico-economic feasibility of the process, the pilot productions and the reserach of industrial partners (producers and users of adhesives) ; other tests are currently carried out (viscosity variation, product presentation, …)

(2) Use of raw materials rich in proteins: wheat or corn glutens or protein concentrates from oilseeds (rape, soya...) [i]

The INRA / Montpellier has developed a resin based on agricultural raw materials for the manufacture of panels (including particleboards)[14].
The used vegetable proteins are available and are not expensive. *Moreover,* ***the process works on industrial presses for the manufacture of particleboards (direct mixture of powders of proteins and wood fibers/particles before the passage in press) and works on fibers containing 5-8 % of water (energy economy / conventional process which need the use of very pre-dried wood fibers/particles).***
On the other side, *technical and economic viability of such resins at industrial scale stays to demonstrate.*

(3) Modification of PF resins by vegetable proteins (AsWood™ resins)

DYNEA has developped AsWood™ resins (replacement of about 25 % of phenol by proteins).
We observe ***a decrease of the formaldehyde content and the 24 h swelling, and an increase of the internal bond strength after boiling***, but *a decrease of the internal bond strength.*

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[14] Following a partnership with the unit, Tate & Lyle (UK major sugar group) has developed and patented this application.

e) Use of unsaturated oils [*23*]

The FCBA and ARD (Agro industrie Recherche et Développement) have worked on the use of epoxydized linseed and rapeseed oils (plywood, MDF & particleboards).
Difficulties exist during the step of pressing/heating (low reactivity of resins at maximum allowed pressing temperatures).

f) Combined solution based on lignin, tannins and soya proteins [*23*]

The LERMAB (Laboratoire d'Etudes et de Recherche sur le Matériau Bois) has developped binders, based on glyoxalized soya flour mixed with a little proportion of tannin or glyoxalized lignin for particleboards.
The panels performances are in conformity with standards for an interior application.

III.2.2 Other solutions

Other solutions in the vegetable way have been identified, but without enough information available (*) or enough strengths to consider them as interesting solutions :

- **increase of the natural adhesion of wood fibers :** consists in doing research on the genetic modification of the characteristics of wood fiber [21] *
- **use of carbohydrates [*20*]***
- **use of conifer needles extracts [*12*]***
- **NAPAPI Project (Nouveaux agro polymères pour adhésifs aux propriétés innovantes) / « natural PU »***

III.3 Combined solutions

III.3.1 Use of glyoxalized lignin (GL), tannins and pMDI [*28*]

The substitution is total.
Moreover, the internal bond strength of particleboards manufactured with resins, based on lignin, pMDI, tannins and glyoxal is in conformity with European standards.
And the internal bond strength of panels manufactured with the resin GL/pMDI (60/40) is very good, even for short pressing times in laboratory (13 s/ thickness mm).
On the other side, *the resins are not very reactive at pressing temperatures used in laboratory (195 to 200 °C).*
The prospects of this work are the industrial transfer of the technology and the valorization of panels at the end of life.

III.3.2 Use of glyoxalized lignin (GL), associated to different materials: PF resins and pMDI, tannins and pMDI or tannins (CIMV & ENSTIB)

Three formulations have been tested on particleboards, at **laboratory** scale, then at **pilot** scale (ENSTIB):

- GL (55) / PF (25) / pMDI (20),
- GL (55) / tannin (20) / pMDI (25),
- GL (50) / mimosa tannin (48) + hexamine (2).

The 3rd formulation gives the best results (best internal bond strength, no problem of swelling, …) and has been consequently validated for the next tests.

The used lignin is « reproductible » and reactive.
The panels have a good internal bond strength and no problem of swelling. And the coloration is lower than those obtained with only tannins (the tannins degradation, which leads to color, would be "blocked" by the lignin).
The CIMV (Compagnie Industrielle de la Matière Végétale) has developed a new process able to produce, in 2011, about 35 000 t/year of lignin, all of which could be used for the production of binders, following the request. It also envisages ***producing glyoxalized lignin (with the suitable physical properties, for the referred application).***
There is an industrial validation on a German production site (a French site would also be interested).

IV. CONCLUSIONS

IV.1 Chemical way

The use of petrochemical products (PF resins, for example) has weaknesses linked for example to environment.
In addition, other possibilities, which in terms of mechanical properties may present an attractive alternative, may pose potential risks during manipulation by operators (pMDI, ...).

No economically viable alternative could have been therefore detected.
In these conditions, it seems difficult to propose additional improvement ways from petrochemical products only.

IV.2 Vegetable way

Natural materials have several disadvantages:

- their availability[15],
- their properties : the mechanical properties of panels produced with vegetable resins do not reach those of current products,
- uncertainties about health and safety (no information about the actions of theses resins on the health, …)
- their maturity : several technologies have not been tested at the industrial scale yet ; the next step will be consequently the valorization of results by transfer of these technologies at an industrial level.

About the lignin, the CIMV want to produce, from 2011, about 35 000 t of lignin and to produce glyoxalized lignin with suitable physical properties, for the referred application.
The CIMV is open to every proposition, every collaboration (laboratory, glue producers, panels manufacturers, …) in order to continue the researches on the improvement of the binders.

IV.3 General conclusion

The resins usable in the referred application must comply with a number of characteristics:

- possible use with a simple adaptation of the production line,
- supply of large quantities,
- cost compatible with the application,
- respect of products characteristics,
- lower risk to the environment, health and safety, than those of current solutions.

Consequently, as in the last studies, no « panacea » can be proposed. We can only identify ways providing a partial answer to the problem.

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[15] It is one of the main criteria identified for the evaluation of the substitution solutions (the French annual production of panels need more than 460 000 t of resins).

We have to improve again the technologic quality and the production of « natural » resins or « hybrid » resins (natural/synthetic), in order to develop products, which will be economically viable and simply usable in the industrial manufacturing of panels.

The works of the CIMV seem to be the best positioned solution to answer to the problem of reduction of formaldehyde in panels, by a reduction of the use of aminoplastic resins or by a total substitution of these resins.

APPENDICES

APPENDICE 1 : ACRONYMS

ADEME : Agence de l'environnement et de la maîtrise de l'énergie
AGRICE : Agriculture pour la Chimie et l'Energie
ARD : Agro industrie Recherche et Développement
CFP : Compagnie Française du Panneau
CIMV : Compagnie Industrielle de la Matière Végétale
IARC : International Agency for Research on Cancer
CRAMA : Caisse Régionale d'Assurance Maladie d'Aquitaine
CRITT : Centre Régional pour l'Innovation et le Transfert de Technologie
CTBA : Centre Technique du Bois et de l'Ameublement
CTP : Centre Technique du Papier
DRIRE : Direction Régionale de l'Industrie, de la Recherche et de l'Environnement
DRTEFP : Direction Régionale du Travail, de l'Emploi et de la Formation Professionnelle
EPF : European Panel Federation
ENSIACET : École Nationale Supérieure des Ingénieurs en Arts Chimiques et Technologiques
ENSTIB : Ecole Nationale Supérieure des Technologies & Industries du Bois
FCBA : Institut Technologique Forêt Cellulose Bois-construction Ameublement
FFIF : Finnish Forest Industries Federation
HDF : High Density Fiberboard
IB : internal bond strength
IRSST : Institut de recherche Robert-Sauvé en santé et en sécurité du travail (Québec)
LCA : Laboratoire de Chimie Agro-industrielle
LCPO : Laboratoire de Chimie des Polymères Organiques
LERMAB : Laboratoire d'Etudes et de Recherche sur le Matériau Bois
MDF : Medium Density Fiberboard
MDI : methylene diphenyl diisocyanate
MF : melamine-formaldehyde
MOE : modulus of elasticity
MOR : modulus of rupture
MUF : melamine-urea-formaldehyde
OSB : Oriented Strandboard
PF : phenol-formaldehyde
pMDI : polymeric methylene diphenyl diisocyanate
PRF : phenol-resorcine-formaldehyde
PU : polyurethane
SEREX : Service de recherche et d'expertise en transformation des produits forestiers
TDI (toluene di-isocyanate)
TRD : Toulousaine de Recherche et Développement
UF : urea-formaldehyde
UFC : Union des Fabricants de Contreplaqué
UIPP : Union des Industries des Panels de Process
UPPA : Université de Pau et des Pays de l'Adour
US2B : Unité Sciences du Bois et des Biopolymères

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APPENDICE 3 : LIST OF CONTACTS

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ENSTIB / Nancy	Antonio PIZZI
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UNILIN	Reynald KAWECKI Stefaan LAUWERS
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DOMUS Matériaux	-
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CRITT (Centre Régional d'Innovation et de Transfert de Technologie) Bois Epinal	Eric MASSON
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