DEPARTEMENT FORETS

COMPTE RENDU MISSION

AUTEURS :   NAPOLI ALFREDO ET CHAIX GILLES
LIEU ET DATES DE MISSION : BRESIL DU **02/07** AU **12/07/06**

AOUT 2006

Résumé

Cette mission s'inscrit dans le cadre de collaboration entre le département Forêts du Cirad et Vallourec & Mannesman Tubes do Brazil. Elle avait plusieurs objectifs :

- Présenter les résultats des expérimentations menés au Cirad sur la pyrolyse d'eucalyptus (projet V&M) ;
- Préparer la réalisation des essais industriels de pyrolyse chez Bordet (projet V&M) ;
- Définir la démarche pour le transfert de techniques liés à la SPIR ;
- Arrêter un programme de formation et de travail dans el cadre du transfert technique NIORS pour les 6 mois à venir ;
- Visiter les plantations et les infrastructures de V& M do Brazil ;
- Etudier les possibilités supplémentaires de collaboration avec l'Université Fédérale de Lavras (UFLA).

Mots clés :

Eucalyptus, charbon de bois, procédés de pyrolyse, NIRS, V&M do Brazil, Alfa GEMA, Université de Lavras

Diffusion de la note :

CIRAD Département Forêts : UPR biomasse-énergie, UPR Diversité génétique et amélioration des espèces forestières, UPR Production et valorisation des bois tropicaux, B. Mallet, C. Sales.

CIRAD Direction Régionale Brésil : P. Petithuguenin

CIRAD DREI : A. de Courville

1. Projet V&M.

Personnes rencontrées : Vamberto Ferreira de Melo, Romero Mantuano, Ronaldo, Túlio Raad, Sampaio, Paulo César da Costa Pinheiro, Jean-Claude Prouhèze, Flavio RS de Azevedo, Marco Antonio SC Castello Branco

Partie Napoli

Essais laboratoire – Après une présentation des différents participants, la réunion a portée sur la présentation des résultats expérimentaux obtenus au Cirad sur la pyrolyse d'eucalyptus envoyés par V&M, suivi d'une discussion. Les expérimentations ont été menées à trois températures 400, 500 et 600 °C et pour deux diamètres de bois 9 cm et 14 cm afin de déterminer l'influence des transferts thermique en pyrolyse. Les bilans masse ont été présentés ainsi que la qualité des charbons en termes de carbone fixe. Une attention particulière a été portée sur l'évaluation de l'homogénéité du traitement de pyrolyse. Pour cela, les mesures de carbone fixe ont été réalisées en différents points des morceaux de charbon du centre jusqu'en périphérie. Les résultats mettent en évidence qu'une température de 400°C est suffisante pour obtenir un charbon de bois respectant les exigences de V&M (75% carbone fixe) mais les questions d'homogénéité de traitement se posent. L'homogénéité du traitement thermique est sensiblement dépendante du diamètre des bois mais également du bon contrôle du taux d'humidité initial des échantillons. Ces résultats méritent des expérimentations complémentaires qui seront réalisées au Cirad avant les essais industriels.

Préparation essais industriels - Les essais industriels sont planifiés pour la période du 19 au 28 août sur le site de Leuglay en France. Les personnes en charge de mener les essais sont pour V&M : Mr Vamberto Ferreira de Melo, Mr Túlio Raad, Mr Romero Mantuano et Mr Sampaio ; pour le Cirad de Mr Napoli et Mr Da Silva Viera (thésard Alpha Gema). Un point a été fait sur l'envoi des containers de bois (300 t d'eucalyptus) et un risque de retard est possible. Mr Vamberto a en charge de suivre le bon déroulement du transport. Sur la base des résultats obtenus au Cirad à l'échelle laboratoire, les conditions opératoires de pyrolyse ont été fixées pour les essais industriels. Il est convenu de travailler en variant le temps de séjour du charbon (3 fréquences de sortie) et en baissant la température de pyrolyse du four au plus bas en fonction des contraintes du procédé (400°C idéalement). L'objectif est de produire un charbon présentant un taux de carbone fixe de 75% maximum. Les expérimentations seront réalisées en utilisant deux diamètres pour les échantillons de bois, tout comme les expérimentations laboratoires (9 cm et 14 cm).

Afin de réaliser un bilan masse et énergie du procédé, certaines questions techniques nécessitent l'avis de Mr Bordet et un questionnaire lui a été transmis par Napoli. D'autre part, les mesures ont été identifiées comme importantes à réaliser sur site comme la pesée des containers, l'analyse des gaz et la détermination des taux de carbone fixe. Alfredo Napoli est en charge de mettre en place les moyens de réalisation de ces mesures en association avec les équipes mesures de V&M en France. Ainsi les analyses de gaz seront assurées par V&M et sa filiale CEV et les analyses des taux de carbone fixe des charbons de bois seront effectuées quotidiennement dans les laboratoires V&M Nucléaire à Montbar par l'équipe. Enfin, une feuille de route des essais sera préparée par Mr Vamberto et transmise à toute l'équipe et notamment Mr Bordet pour validation.

Partie Chaix

L'objet de la mission de Gilles Chaix concerne le volet transfert technologique des techniques liées au proche infrarouge et plus particulièrement sur la définition des échantillons en fonction du matériel végétal disponible. Vallourec souhaite former un chercheur brésilien généticien (M. Nilson César Castanheira Guimarães) et a acheté un spectrophotomètre selon nos instructions (livraison prévue en septembre). Tout en assurant cette formation, il s'agit d'aborder les applications apportées par la SPIR, notamment l'analyse du taux de lignine et des extraits du bois. Cette mission a permis de :
- définir l'échantillonnage nécessaire en fonction des essais sur le terrain et des objectifs de sélection,
- de discuter du programme de formation de M. Nilson César Castanheira Guimarães en France en septembre 2006,
- de proposer un programme de travail jusqu'en Février 2006
- de discuter des programmes de recherche dans le domaine des plantations forestières à l'occasion de la venue de M. Prouhèze : programme d'amélioration génétique, sélection de clones, problèmes phytosanitaires dans les plantations forestières, études de l'influence des plantations sur le régime hydrique des sols, ...

Il était prévu que VMB développe des calibrations sur les espèces pures et plusieurs types d'hybrides. Dans un premier temps et devant les objectifs de sélection sur la formule hybride *E. urophylla* x *E. camaldulensis*, nous avons préconisé de démarrer les travaux par une première calibration portant sur des échantillons de bois provenant d'un essai descendance âgée de 7 ans issu de croisements *E. urophylla* x *E. camaldulensis*. La formation de M. Nilson César Castanheira Guimarães portera donc sur ce premier lot d'échantillons. Dans cet objectif, des visites de terrain ont été effectuées ainsi que des réunions de travail élargies au différentes problématiques : génétique, qualité bois, carbonisation. Les calibrations pour les autres espèces et hybrides se feront dans la foulée sur la bsse des connaissances acquises et avec l'appui du Cirad.

En annexes, se trouve les résultats des discussions et le détail des conclusions établies par Gilles Chaix et rendues en fin de mission à M. Nilson César Castanheira Guimarães et M. Helder Bolognani.

2. **Université Fédérale de Lavras - Projet Alfa GEMA**

Personnes rencontrées : José Tarcísio Lima (UFLA), Sebastião Carlos da Silva Rosado (UFLA), Paulo Ricardo Gheardi Hein (UFLA), Ana Carolina Maioli Campo (UFLA), Helder Bolognani (V&M), Nilson César Castanheira Guimarães (V&M),

La réunion s'est déroulée à l'université fédérale de Lavras (UFLA) au département forestier. Après une présentation des différents participants, les discussions ont porté sur le déroulement du projet Alfa-Gema en cours avec le Cirad et l'INRA et sur les possibilités de renforcement des collaborations. L'UFLA est principalement axée sur l'étude des eucalyptus du brésil avec une forte compétence en sylviculture, management forestière et en technologie du bois, particulièrement pour des applications de bois d'oeuvre. Elle travaille étroitement avec V&M depuis de nombreuses années sur ces thématiques et souhaite s'ouvrir à des collaborations internationales ciblées.

Les discussions ont porté notamment sur le travail de thèse de Renato da Silva Vieira et sur les futurs travaux des deux étudiants en Master (Paulo Ricardo Gheardi Hein et Ana Carolina Maioli Campo) que nous accueillons d'Aout 2006 à Janvier 2007.

La présentation du projet V&M-Cirad sur l'étude des relations génétique / matière première / produit final suscite de l'intérêt de la part de l'UFLA. V&MB serait favorable à un élargissement de la collaboration avec le Cirad permettant de consolider les recherches menées actuellement. Cette collaboration pourra s'étendre tant sur les aspects techniques et scientifiques que sur les aspects enseignement et encadrement d'étudiants.

<u>Programme de la mission:</u>

Lundi 3 juillet :	Arrivée le à Belo-Horizonte, nuit à Sete Lagoas
Mardi 4 juillet :	Vallourec & Mannesmann Florestal Ltda, projet CAPEF à Paraopeba. Discussions de travail et visite du site de production de charbon. Nuit à Sete Lagoas
Mercredi 5 juillet :	Vallourec & Mannesmann Florestal Ltda, projet CAPEF à Paraopeba. Discussions de travail et visite des essais dans le périmètre des plantations. Nuit à Sete Lagoas.
Jeudi 6 juillet:	Vallourec & Mannesmann Florestal Ltda, projet CAPEF à Paraopeba. Discussions de travail, présentation des résultats d'Alfredo Napoli à l'occasion de la visite de M. Marco Antonio SC Castello Branco. Nuit à Sete Lagoas.
Vendredi 7 juillet :	Bel Horizonte, visite usine Vallourec Manesmann Tubes do Brazil : hauts fourneaux, laboratoires d'analyses charbon, discussions sur les méthodes employées. Retour et nuit à Sete Lagoas.
Samedi 8 juillet :	Visite Ouro Petro et discussions avec Helder Bolognani. Nuit à Sete Lagoas
Dimanche 9 juillet :	Déplacement Sete Lagoas Lavras, nuit à Lavras.
Lundi 10 juillet :	Discussions Université de Lavras (Visite de l'université des laboratoires bois et génétiques, réunions de travail). Retour à Sete Lagoas. Réunions de restitution avec MM. Nilson César Castanheira Guimarães, Helder Bolognani, Túlio Raad, Chaix Gilles et Alfredo Napoli. Nuit à Sete Lagoas.
Mardi 11 juillet :	Départ pour Montpellier via Rio de Janeiro et Paris.
Mercredi 12 juillet :	Arrivée Montpellier.

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Annexes Recommandations et conclusions sur la partie NIRS (Chaix Gilles)

Introduction

Near-infrared spectroscopy has been recognized as a powerful analytical technique for rapid determination of various constituents in many agricultural and raw materials. The approach involves the acquisition of a reflectance spectrum after near-infrared radiation on ground or solid wood sample in a specific material plane (RT). The resulting NIR spectral information is then calibrated against standard analyses obtained using conventional analytical techniques (standard method) using linear regression. A crucial step in achieving success is ensuring that the samples have been analyzed as accurately and precisely as conventional techniques allow. Then, standard methods and NIRS method will be compared in order to evaluate the potential of NIRS for fast wood quality or charcoal quality control by prediction.

During this first step we recommended the choice of the simplest situation considering only the type of hybrid, the age and the site and the level of wood sampling are constant. For instance, the wood and charcoal properties for which we expect the prediction by NIRS are: lignin content, extract content, basic density and fixed carbon, yield rate respectively. The wood and charcoal properties links could be studied <u>especially the effect of lignin content on fixed carbon for charcoal</u>.

The calibration and prediction of wood and charcoal properties will consider the following hybrids <u>*E. urophylla* x *E. camaldulensis* or *E. camaldulensis* x *E. urophylla*</u> produced by <u>pollination controlled processing by VMB</u> or by other companies:

- trees from full sibs progenies trials *E. urophylla* x *E. camaldulensis* or *E. camaldulensis* x *E. urophylla* 7 years old <u>planted in 1999</u>
- VMB clones trees as possible <u>7 years old (3-4 clones, 6 repetitions by clone)</u> ➜ <u>If possible, 2 repetitions per region</u>
- Four trees from the two parental species/trees as possible <u>7 years old (from different provenances)</u> ➜ 4 *E. camaldulensis* and 4 *E. urophylla*
- Some tress from <u>2-3 another farms (as possible full-sibs with previous progenies)</u> could be added as possible 7 years old ➜ <u>Around 10 trees</u>

Firstly, the standards methods, achieved by VMB, will be process with the best accuracy and repeatability possible, and definitively <u>the quality of calibration and prediction depends on the error of laboratory knowledge (depends on the wet chemistry precision, what we must know well) and the knowledge of its level is crucial (we could consume a lot of time to optimize the calibration equation error and the error of laboratory could be greater)</u>. The reference method <u>(wet chemistry and thermoscale)</u> of the <u>lignin content</u> and <u>the extract contents</u> should to be validated: standard method (lignin Klason, extractives standard TAPPI) or VMB method. The basic density can be easily measured classically (water displacement method then oven drying) but the sample collection can be used to develop a calibration for this property. The same recommendations are proposed for charcoal properties. Signal-to-noise ratios, precision, selectivity and other measurement characteristics should also be given attention. These measurement characteristics should be as good as possible, so fine-tuning of instruments may be an important part of the first step. <u>As much information about these issues as possible should be present prior to calibration, to help interpretation and safe use of the techniques.</u>

Secondly, the choice of calibration samples is very crucial and theses samples should be as evenly spread as possible over the whole variation range. The sample selection will be process by two steps; first by pedigree knowledge and phenotypic (not only outstanding individuals), second by wood spectral analysis (screening of samples to calibration).

This calibration will be use for the genetic parameter estimation and the selection. This goal needs a representative sampling selection of the hybrid populations and it's very important to consider the variability of growth based on height and the diameter. The growth in height and diameter should be correlated with one part of the wood properties especially lignin content and basic density. If the high growth trees will be selected only one representative part of the population should be exclude and one part of the information of wood properties linked with the growth will be not considered as described by the figure 1.
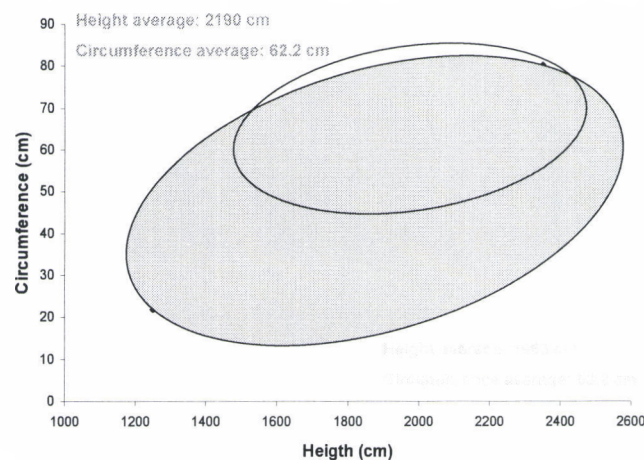

Figure 1. Sampling effect based on height and circumference

We recommended associated the variability among different trees and the variability within the trees. Figure 2 indicate the radial variability of the volumic mass for two trees (A and B) who describe the amplitude of the variability within the trees and the difference among them.
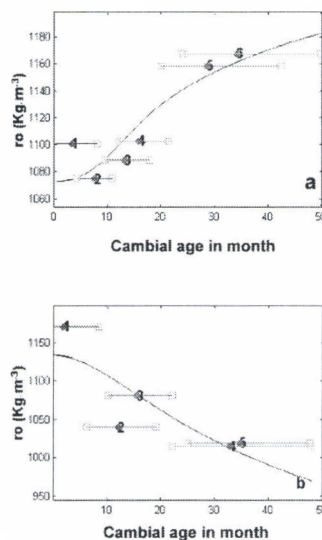

Figure 2 Variability in two trees of volume mass by cambial age. 90% of the population has the behave like **a** and 10% like **b**.

Recommendations

Spectrophotometer points

Spectrophotometer will deliver on September. Both for the spectrophotometer efficiency, sample conservation, repeatability, calibration and prediction quality, the NIRS methods need temperature and humidity conditions stabilized. The goal is to maintain the humidity of the sample at the same level during NIRS and prediction process. VMB could be transform a room and dedicated this room for the NIRS activity and the storing of wood and charcoal samples too.

Selection Methodology of calibration samples

The approach based on the powder samples from disk appears easier. But, the value of these samples were an average (different types of wood corresponding at different age were mixing) and the calibration and prediction by NIRS would be less efficient than punctual method using solid wood. In a other hand, the selection in a large scale will be process on core sample on which NIRS measurements could be easily registered.

The accuracy way proposed is a methodology step by step. This process should be optimizing the selection of sampling because the selection of a well representative sampling of variability existing in the hybrids population (full sib families) is a very crucial point.

A first set of 500 trees selected on these following levels of variability in according to the VMB mating design (PROGENY TRIALS):
- Species mother: *E. urophylla* / *E. camaldulensis*
- Species father: *E. urophylla* / *E. camaldulensis*
- Origins of *E. urophylla* and *E. camaldulensis*
20 progenies for each 10 mothers and 10 fathers (they will be the same) *E. urophylla* well distributed in according to the provenances of *E. urophylla* breeding population
20 progenies for the 5 mothers and 5 fathers (they will be the same) *E. camaldulensis* well distributed in according to the provenances of *E. camaldulensis* breeding population

CHOOSING UP TO THIS POINT! THE NEXT SELECTION IS BASED IN HEIGHT AND DIAMETER!

A second set of 250 trees among the 600 trees selected on diameter and height in according to the variability of these characters (based on index).
10 progenies for each 10 mothers and 10 fathers (they will be the same) *E. urophylla* precedent selected on provenance.
5 progenies for each 5 mothers and 5 fathers (they will be the same) *E. camaldulensis* precedent selected on provenance.

NEXT STEP IS BASED ON THE SPECTRA!

A third set of 150 trees selected among the second one based on the Principal Component Analysis of the wood spectral NIR processing during the training in September. This operation allows a gain of time before the complete installation of Bruker NIRS. All types of combinations of variables should be represented from spectral point of view. The variation in all directions should be as largest as possible, but limited to the spectra region of interest. The

Cirad previous calibration should be used, not for prediction but to classify the samples. This selection should be controlled in according to the previous selection criteria.

All these approach should be compared one by one or by different combinations. The demarche should be evaluated after the chemical analysis process.

Schedule proposals of harvesting samples

The selection process is described in Figure 3.

July 2006

- Selection of 250 trees based on the species, origin, height and circumference. The trees are marked in the field
- Trees will be cut and three 0.5 m logs by trees will collect (A: 1.8-2.3m height, B: +/- 25cm around the 50% of commercial volume height, C: +/- 25cm around the 75% of commercial volume height)
- The north direction will be marked on each sample
- One disk 50 mm of thickness by log and the diametric planks for each log (same direction for all the trees) will saw by mechanical chain and stored in drying room at ambient conditions. The diametric plank will saw by mechanical chain and stored in the following conditions: 25°C, 30% humidity.

August 2006

- The diametric plank of the log 1 will be saw and divided by 3.0 x 3.0 x 45 cm "baguette" the name of baguette correspond to the distance of the pith
- Each baguette will be split in three part: part 1: 3.0 x 3.0 x 15 cm (Lignin content), part 2: 3.0 x 3.0 x 15 cm, being 5 cm to be stored at CIRAD, 7 cm to be stored at VMB and 3 cm to be taken to CIRAD by M. Nilson César Castanheira Guimarães, and part 3: 3.0 x 3.0 x 15 cm (these dimensions depend of the minimal quantity needed for the chemical analysis, charcoal process and the quantity of charcoal needed for the NIRS.
- The part 1 will be designed for the wood properties analysis (lignin and extractive contents), this part will be grounded (Nirs measurement before and after the grounder). One part of powder should be stored for another analysis (TGA, …). One gram is needed for lignin and extractive contents (Túlio Raad com. Pers.)
- The part 2 (solid) will be designed for the NIRS measurement reference and store collection. A collection of reference samples used for calibrating and validating will be stored for controlling purposes or other to be used on different NIR devices.
- The part 3 will be designed for the charcoal process (Nirs measurement before the carbonisation) and before this parts will use for the basic density.
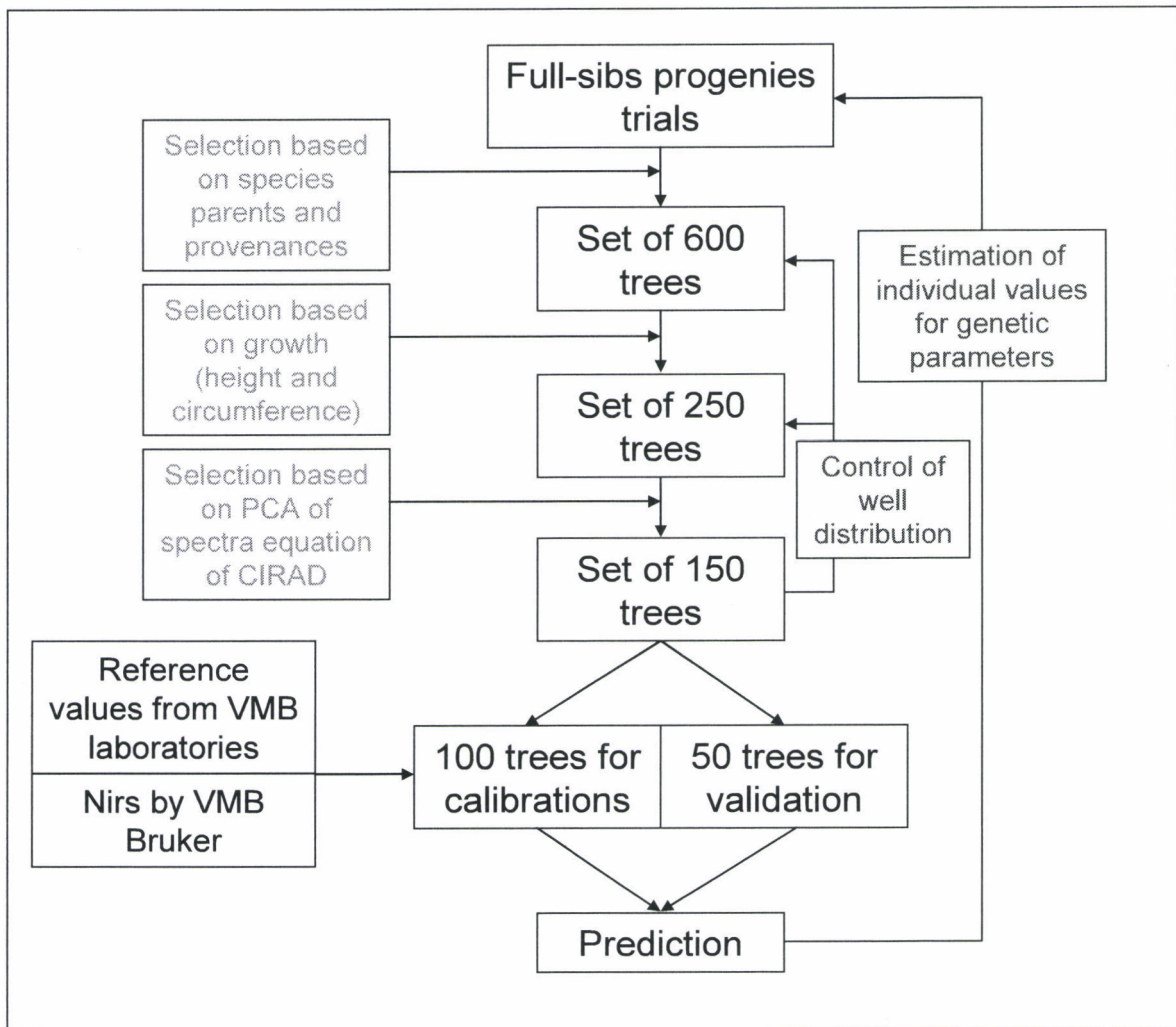All samples will be stored in NIRS room in controlled conditions.

Figure 3. Selection process for the calibration sample choose

Schedule of training program in France (from September 16 to October):

M. Nilson César Castanheira Guimarães will be training both on the wood sample of Cirad and the wood sample of VMB. The part 2 of each 250 samples for three baguettes corresponding to three positions: near the pith but not include the heart, medium part and near the extreme part as described in the figure 4. The total of sample for this step is 750.
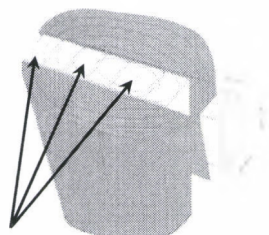

Figure 4. Description of the selection of the three samples per diametric planks

The training program is as following:

- chemometric methods training by CIRAD and ONDALYS 1 week

- OPUS (software delivered with Bruker) and Bruker Spectrophotometer training on CIRAD samples by CIRAD 1 week

- Wood spectral acquisition of VMB samples 1 week

- PCA on wood spectral and selection of the calibration and validation sets 1 week (Calibration and validation performed in Brazil).
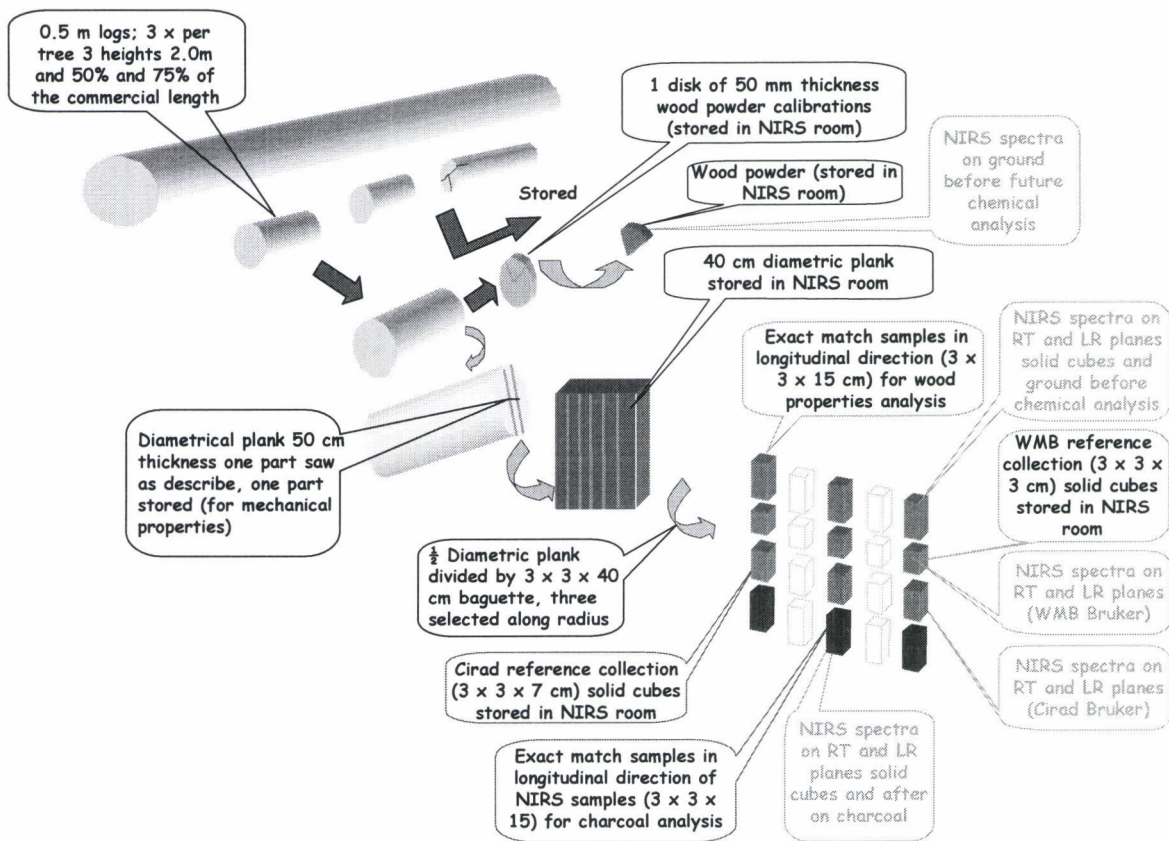

Figure 5.Wood sampling methodology

Schedule proposals after the training in France (October 2006 to January 2007):

October-November 2006

- NIRS spectra will be acquired as describe in the figure 5. The spectral data were specific in according to the spectrophotometer and the laboratory conditions. So the NIRS spectra preceded in Cirad couldn't be use in Brazil, but these could be use for a Cirad prediction by comparison.

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- As soon as possible, when M. Nilson César Castanheira Guimarães will come back in Brazil, measurements of reference values for basic density, lignin and extractive contents on selected samples (450) will be process. The lignin content is determined after the extraction step, so a spectral NIRS will be measured on the powder extracted.

December 2006

- When the reference values (lignin content, extractive content, basic density, charcoal yield, fixed carbon will be available, the step of calibration model will be begin. This step may be a time-consuming exercise if one is unlucky, i.e. if the relationships between y (properties) and x (spectra) are complex.

The first thing one should do, especially if the collinearity is substantial, is usually to run PLS or PCR and check their results. Standardizations should also be considered at this point. This checking of results should involve looking at the plots, outlier diagnostics and a test set validation or cross-validation.

If there are clear outliers in the data set, these should be taken seriously and handled. Generally, a calibration is not a one-shot exercise. It often needs some trial and error and some iteration between modeling, variable selection, interpretation, outlier detection and validation. In particular, this is true in situations with limited prior knowledge about the application under study. The more is known, the easier is of course the calibration.

The validation should always be taken seriously regardless of which of the methods is eventually chosen. In a thorough validation process, possible outliers and model quality should preferably also be considered.

The spectral data were specific in according to the spectrophotometer and the laboratory conditions. Spectral data were collected before with the VMB spectrophotometer as described by the figure 5.

January 2007

- Prediction of new sample
- The log 2 and 3 will be saw and divided by 3.0 x 3.0 x 45 cm "baguette" the name of baguette correspond to the distance of the pith
- Each baguette will be split by three 1: 3.0 x 3.0 x 15 cm, 2: 3.0 x 3.0 x 15 cm (3, 5 and 7 cm) and 3: 3.0 x 3.0 x 15 cm
- The part 2 will be designed for the NIRS measurement reference and store collection.
- The wood properties and charcoal will predict by the previous equations. Without doubt one great part of these samples will be outliers. But the equations will classify them together and some samples will select for wood and charcoal properties analysis and will include in the new calibration after. With these latest the wood and charcoal properties should be predicted and the other progenies too.

Control of the process

When a calibration is developed and one is satisfied and confident with the results, the calibration can be put into production. Before one relies too heavily on it, one should,

however, test it carefully in a practical environment. This means that frequent tests using the monitoring methods should be used. As one gains confidence in a calibration, the less frequent the tests need to be. They should, however, never be forgotten completely.

If a clear drift or change takes place during regular operation one can use specific methods for correction.

Comparison "solid sample way" and "powder sample way"

The approach based on the powder samples from disk appears easier. But, the value of these samples were an average (different types of wood corresponding at different age were mixing) and the calibration and prediction by NIRS would be less efficient.
The process (NIRS, wood and charcoal properties analysis, calibration and prediction) will be applied on powder sample issued from the 3 disks by trees. Fifty samples and thirty samples will use for calibration and validation.

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