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Bio-Norm II

Pre-normative research on solid bio-fuels for improved European standards
SPECIFIC TARGETED RESEARCH OR INNOVATION PROJECT
PRIORITY [6-1] – Sustainable energy systems

Report on protocols for sample reduction

Deliverable D.I.7

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SLU – Swedish University of Agricultural Science

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Revisions

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Abstract

Six materials were investigated: olive residues, grape residues, bark, wood chips and pellets (6 and 8 mm). Three sample reduction methods were considered: long pile, coning & quartering and riffle box. For bark only the two first methods were used due to the large particle size. The samples will be isolated from the combination among materials and methods, and then they will be analyzed. For each material, the best sample reduction technique to be used in the next phase called “sampling” will be identified and chosen after statistical analysis of the results.

Table of Contents

1 Introduction and Objectives

In this first phase of Task I.1 "sample reduction" the national teams involved are UNIVPM, CTI and SLU.
UNIVPM, SLU and CTI will execute the practical part of sample reduction and analysis, SLU the statistical part.
In two meetings in Ancona at the UNIVPM (kick-off meeting of BioNorm II WP I, 3rd of April 2007, and CEN TC 335 WG3 meeting, 4-5 June 2007), decisions concerning the execution of both the first part, sample reduction, and second part, sampling, of Task I.1 were taken.
This first part of Task I.1 should provide the best sample reduction methods to be used in the second part. The choice of the best reduction method will be done after the statistical analysis of the results. Any possible positive or negative characteristic of the materials towards different methods will be taken into consideration.
The second objective of this first phase is to compare the repeatability of each analytical method that is used with the variability of the sample reduction method, using statistical analysis on repeated determinations of sub-samples.

2 Materials and Methods

2.1 Bio-fuel materials

The following materials were under testing:
- olive residues;
- grape residues;
- bark;
- wood chips;
- wood pellets (diameter: 6mm and 8 mm).

Olive and grape residues (typical Mediterranean materials) were taken from C&T's biomass power plant located in Termoli (Southern Italy), where also the next phase of sampling will take place.
The 6 mm pellets were taken from a production plant in Northern Italy but analyses of sample reduction and sampling tests will be performed in September.
Bark, wood chips and 8 mm pellets were taken from the power plant of Skellefteå Kraft AB.
The characteristics of the materials were as follows:

Grape residues:

nominal top size:	*16 mm*
density as received:	*440 kg/m^3*
density of dry matter:	*140 kg/m^3*

Olive residues:

nominal top size:	*3,15 mm*
density as received:	*600 kg/m^3*
density of dry matter:	*550 kg/m^3*

Bark:

nominal top size:	*ca 400 mm*
density as received:	*ca 400 kg/m^3*
density of dry matter:	*ca 180 kg/m^3*

Pellets 8 mm:

nominal top size:	*15 mm*
density as received:	*650 kg/m^3*
density of dry matter:	*610 kg/m^3*

The pellets chosen were made of spruce or pine with a higher ash content as compared to usual pellets (with an ash content of about 0,3-0,4%), as decided at the CEN-meeting.
The sample reduction of 6 mm pellet and the wood chips will be performed in September because of problems in buying a device for durability determination and because the production of wood chips from stem wood is stopped during the summer.
Depending on the test methods the materials were pre-treated in an appropriate way to be able to do the analysis.

2.2 Sample reduction methods

All materials were subjected to 3 different sample reduction methods except for bark where only the long pile and the coning & quartering methods were used due to the large particle size.
In each method, the whole laboratory sample was divided into several sub-samples.

2.2.1 Long pile

The following steps were defined:
1) Realisation of a strip of material.
2) Definition of the number and size of the increments: in the case of olive residue, for example, each sub-sample was constituted by 20 increments and each increment was 25g.
3) 16 containers were arranged near the strip.
4) 25g of material (increment) were cut with a dividing plate from the end of the strip, got this increment with the shovel, being careful not to leave fine particles behind, then the increment was poured into the first container. (see Figure 1)
5) Another 25g of material were cut and with the shovel poured into the second container.
6) This operation was repeated until 20 increments were obtained in every container.

Figure 1. Photograph of the long pile method using olive residues.

2.2.2 Coning & quartering

The main steps were the following:

1) The whole laboratory sample was shovelled on a clean, hard surface, forming a conical pile: each shovelful was placed on the top of the preceding in such a way that the bio-fuel run down all sides of the cone well distributed and different particle sizes were well mixed.
2) This process was repeated three times, forming a new conical pile each time.
3) The third cone was flattened by inserting the shovel repeatedly and vertically into the peak of the cone to form a flat heap having a uniform thickness and diameter no higher than the blade of the shovel.
4) The heap was quartered along two diagonals by inserting the shovel vertically into the heap. (see Figure 2)
5) The coning and quartering process was repeated until 16 sub-samples were obtained.

Figure 2. Photograph of the coning & quartering method using olive residues.

2.2.3 Riffle box

The main steps were the following:

1) The whole laboratory sample was put into one of the containers of the riffle (Figure 3) so that it was evenly distributed throughout the container.
2) The other two containers were placed in position under the riffle.
3) The contents of the first container was poured down the centre line of the riffle and in a way sufficiently slowly that bridging didn't occur.
4) The container should not be moved from side to side (as in this case the end slots would receive less bio-fuel).

Repetition of the riffling process until 16 sub-samples were obtained.

Figure 3. Photograph of a riffle box.

3 Utilization of methods and data collection

3.1 Utilization of methods

With olive and grape residues, every reduction method started with 8 kg of material to obtain 16 sub-samples, each sub-sample being 0,5 kg, as shown in figure 4. With pellets and bark, each reduction method was started with 16 kg and 320 L, respectively, to obtain 16 sub-samples similar to the scheme below. The pellet sub-samples were divided once more to sample A and B, which were analysed for ash content and mechanical durability, respectively.

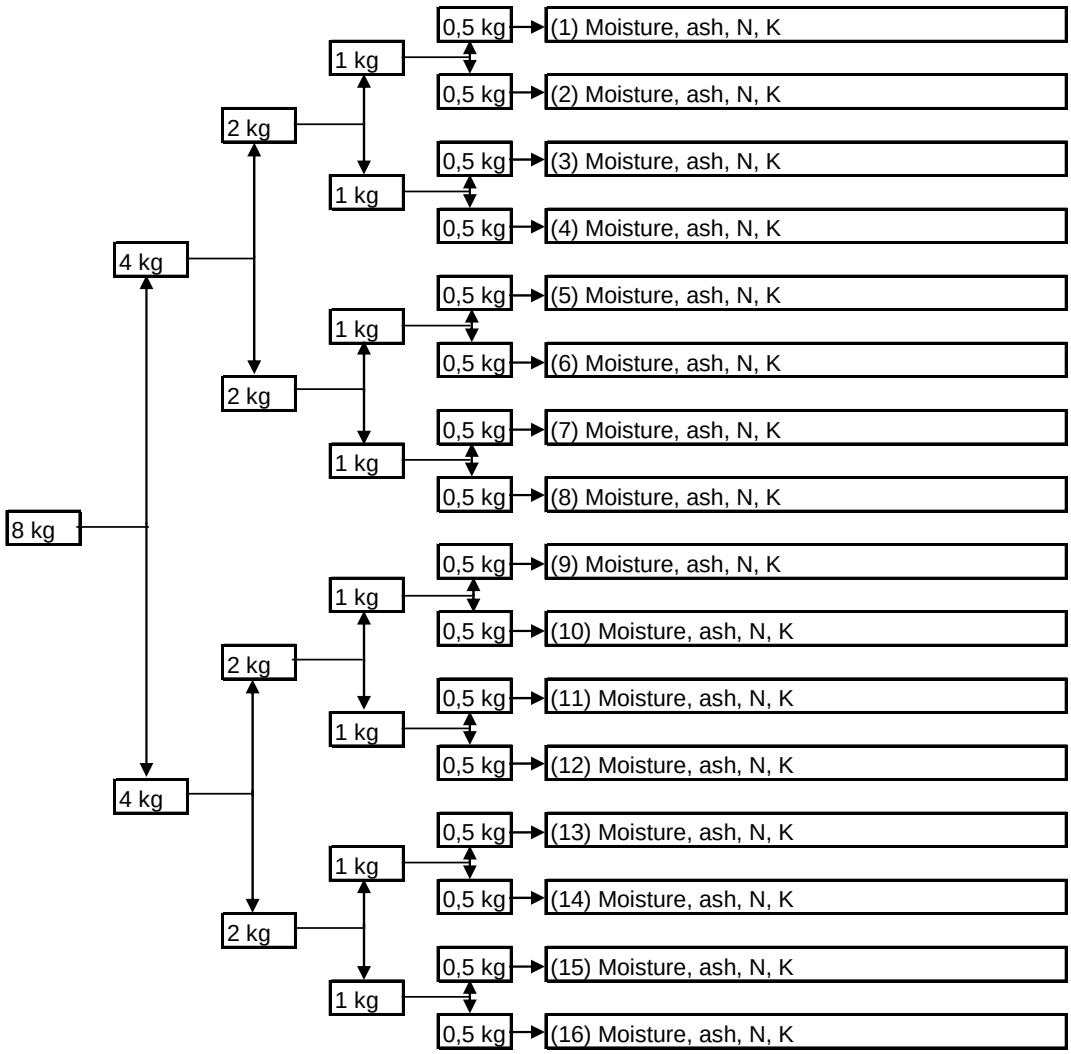

Figure 4. Scheme for the sample reduction when using coning & quartering and riffle box as sample reduction methods.

3.2 Repetitions

In order to evaluate the variability of the test methods it was been decided to either make single analysis of the sub-samples except for one sub-sample (randomly chosen) for which 5 determinations were made or make duplicate analyses of all subsamples.

3.3 Analysis

The analytical parameters chosen in this study were decided in discussion with the CEN TC 335 WG3. For more details, see the deliverable D I.6. The parameters analysed for olive and grape residues were:

- *moisture content;*
- *ash content;*
- *N;*
- *K.*

The parameters analysed for pellets were:

- *ash content;*
- *durability.*

The parameters analysed for bark were:

- *moisture content;*
- *ash content;*
- *calorific value.*

The parameters analysed for wood chips were:

- *moisture content;*
- *ash content;*
- *particle size distribution.*

NB1: concerning olive and grape residues analysis, the choice between N or K and other additional parameters determination will be decided after the statistical analysis, then the best set of them will be taken into consideration in the sampling phase.

NB2: the analysis of the ash content and durability of the 6 mm pellets will be performed in September 2007, when UNIVPM will receive the new device for durability determination.

4 Comments related to the methods used

Some comments about this first phase of sample reduction can be done, taking into consideration material characteristics and method executions.

Long pile: In general this method requires long execution times (5-6 hours for each material). In particular, using grape residues that are very moist, a high loss of moisture occurred and this represents a real practical problem.

Coning & quartering: no particular problems.

Riffle box: using olive residues, fine fraction (powder) losses occurred.

5 Programme of the activities

The activities started in March 2007 and the work with materials in June. The UNIVPM also purchased new instruments (tester for durability of pellet; new Nitrogen analyser, etc.) that will to received hopefully in October, so to better fit the BioNorm II requirements.
In Figure 5 the different activities performed until July 2007 are shown.

	2007							
Activities	Jan.	Feb.	Mar.	Apr.	May.	Jun.	Jul	Aug.
Info collection								
Meetings								
Material collection								
Analysis execution								
Purchase								

Figure 5. Different activities performed until July 2007.