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Report on ash melting behaviour

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Abstract

The development of European Standards (EN) is a precondition to expand the market for solid biofuels and a very important step to fulfil the political and environmental goals of the European Union.

The aim of the BioNorm II project is to carry out pre-normative research in the field of solid biofuels in close collaboration with the work of CEN TC 335 "Solid Biofuels". This includes the development of an overall quality assurance system for solid biofuels, their characterization and corpus of legislation.

In WP II Task II the work focuses on the development of chemical test methods. This includes ash melting of solid biofuel ashes. Results and conclusions are presented in this deliverable.

Investigations focussed on the existing CEN/TS 15370-1 aiming at reproducibility and repeatability data to validate the method CEN/TS 15370-1 and upgrade it to an European standard.

Additionally, the applicability of a new and simple method used in coal fired power plants was investigated and evaluated for biofuels. The method is based on a visual comparison and classification of ash samples heated in one-way ceramic crucibles in an oven at specified temperatures.

The overall conclusion of this task was that results obtained by CEN/TS 15370-1 are in principle useful when the characteristic temperatures occur similar to e.g. coal samples. Currently the method is difficult to use with some biofuel ashes leading to deviating or wrong results with respect to observations in combustion furnaces. Quick methods show potential but are currently not developed good enough to be suitable for an European standard. With respect to a reliable determination of ash melting as an indication for slagging in private or industrial furnaces still research is needed.

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1 Introduction

In BIONORM I, relatively high attention was paid to the investigation of methods characterizing ash melting behavior. The purpose was to identify methods that are suitable for standardization and/or as reference methods. Some of the methods investigated (MAF, TGA/SDTA, SEM-EDS and XRF) are all laboratory methods that require very small amounts of ash sample. Except from MAF, they are well-known techniques but they had to be adopted for biofuel ashes.

- MAF (Melt Area Fraction) is based on computer analysis of images from a microscope, acquired during heating of the sample. In principle, shape - or phase - changes of individual particles are observed.
- DIN, a method adopted from coal analysis, needs a sample quantity that is about ten times bigger.
- In bench scale, two fluidized bed tests were studied – the continuous feed method and the controlled agglomeration (CFBA) method. Both use samples of the original fuel, about 1 to 10 kg. They were designed to simulate the combustion environments in a fluidized bed boiler and could be suitable as reference methods for the sintering/agglomeration behavior of biomass ash.

The essential questions about the tests are:

- What information is really needed to forecast problems related to ash melting?
- What analysis cost is acceptable?

It is generally accepted that it is very important to identify the temperature of initial melt formation. And also the rate of melt forming as the temperature increases is a valuable information. The cost should be as low as possible – this eliminates the fluidized bed methods and methods that produce „hard to evaluate“ results that have to be evaluated by specialists, as TGA/SDTA. The coal ash melting standards DIN 51730 and ISO 540 provide melt information in a relatively simple, lowcost manner. The “Improved DIN” method developed in BIONOM I is an attempt to get around with the subjective element in the standards.

However, there certainly are some problems about the identification of the characteristic temperatures for biomass ashes. Furthermore it is known, that the preparation of the laboratory ash influences the melting behavior, the influences have to be investigated.

Alternative methods like the recently developed MAF method was suggested to be used to better understand the typical characteristic temperatures received by the “Improved DIN” method especially the sintering temperature. The idea behind it was to gain additional information from the analysis how the ash may behave in a combustion unit. However, during this BioNorm II project, neither the scientific partners nor industrial partners in general were interested in fundamental research which is indeed not in the scope of this project dedicated to support the development of standards for investigation of state of the art fuel properties.

It has to be recognized that ash melting is a complex physical/chemical process, where material properties (e.g. density, particle size distribution, melt viscosity, heterogeneity) play a significant role. Therefore, it is unrealistic to expect perfect repeatability from any ash melting analysis, however the investigation of reproducibility and repeatability of the method was one important goal of the project.

A demand for an even simpler method compared with the “Improved DIN” method is expected to be helpful for industry. Coal power plants but also test institutes have developed more or less simple “inhouse“ methods e.g. by putting ash samples in one-way ceramic crucibles in an oven at specified temperatures. The sintered/melted products are kept and are compared with new fuels to predict their suitability for a plant. This BioNorm II task aimed at a comparison of this methods with the standardized method.

2 Description of work

Critical parameters in the CEN/TS 15370-1 “Solid Biofuels - Methods for the determination of ash melting behavior” were investigated in comparative measurements in different laboratories with a variety of biofuel ashes. The results are presented in this report and conclusions for a better definition of the appearance of the characteristic temperatures are drawn.

As explained in the objectives, it is unrealistic to expect perfect repeatability from any ash melting analysis, however aiming at reproducibility and repeatability data to validate the method CEN/TS 15370-1 and upgrade to an European standard was one goal.

The applicability of a new and simple method used in coal fired power plants will be investigated and will be evaluated for biofuels. The method is based on a visual comparison and classification of ash samples heated in one-way ceramic crucibles in an oven at specified temperatures.

2.1 Methods overview

Ash melting is a complex process where sintering, shrinkage and swelling can occur in addition to softening and melting of the ash. Most test methods are empirical. The ash used for the tests should be a homogeneous material, prepared from the fuel in a laboratory under defined conditions e.g. by EN 14775. In contrast, under full-scale conditions, the complex processes of combustion and fusion involve heterogeneous mixtures of particles, variable heating rates and gas compositions.

For the laboratory determination of ash melting behavior is performed at a controlled rate of heating in a controlled atmosphere. Determined characteristic temperatures in the tests can be used for comparison of the tendency of the ashes from different types and qualities of solid biofuels to form fused deposits or to cause bed agglomeration on heating.

Methods tested and investigated within this task:

- CEN/TS 15370-1
- *ofi*-quick method
- Ciemat sieving method

Samples used:

- Orujillo (prepared Ciemat, lab ash, 550°C)
- Cynara (prepared Ciemat, lab ash, 550°C)
- Coconut (prepared ECN, lab ash, 550°C)
- Straw (prepared VTT, lab ash 550°C)
- Straw (prepared by dong with 40% KCl)
- Energy grain (approx. 80% triticale, 20% spruce; prepared by ofi, industrial ash)

2.1.1 CEN/TS 15370-1

The test method described in the Technical Specification CEN/TS 15370-1 provides information about fusion and melting behavior of the composite inorganic constituents of the fuel ash at high temperatures. The method is based on the methods described in ISO 540:1995 and DIN 51730:1998. The terms ash fusibility and ash softening are synonyms to ash melting.



Figure 1: example of an apparatus according CEN/TS 15370-1 (source CIEMAT)

Characteristic temperatures

- **Shrinkage starting temperature** (abbreviation: SST): The temperature at which shrinking of the test piece occurs. This temperature is defined as when the area of the test piece falls below 95 % of the original test piece area at 550°C. Shrinking may be due to liberation of carbon dioxide, volatile alkali compounds, and/or sintering.
- **Deformation temperature** (abbreviation: DT): The temperature at which the first signs of rounding of the edges of the test piece occurs due to melting. For computerised evaluation a shape factor change of 15 % marks the deformation temperature. According to CEN/TS 15370-1 the shape factor (F) of the test piece is determined when a computer is used for image analysis of the test piece during the heating. In order to determine the shape factor, the circumference of a perfect semicircle (b) with the same area (A') as the test piece's shadow (A) is calculated. This ideal circumference is then put in relation to the actual measured circumference of the test piece (a). This relation gives the shape factor.

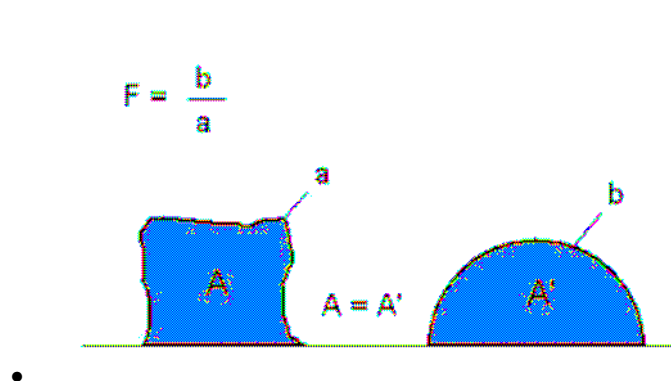


Figure 2: calculation of the shape factor according to CEN/TS 15370-1

- **Hemisphere temperature** (abbreviation: HT): The temperature at which the test piece forms approximately a hemisphere i.e. when the height becomes equal to half the base diameter.
- **Flow temperature** (abbreviation: FT): The temperature at which the ash is spread out over the supporting tile in a layer, the height of which is half of the height of the test piece at the hemisphere temperature. Half of the height of the test piece has been defined due to frequently occurring bubbling effects. This is especially important for automatic image evaluation. This definition is different to other standards, e.g. ISO 540:1995 and DIN 51730.

Principle

A test piece made from an ash prepared according to EN 14775 is heated up with constant rate and continuously observed. The temperatures at which characteristic changes of the shape occur are recorded.

The test piece used, shall have sharp edges to facilitate observation. The mass of the test piece shall be such as to ensure equalization of the temperature within the test body. Hence, dimensions that are too large shall be avoided. An upright cylinder of height 3 mm to 5 mm and a diameter equal to the height should be used:

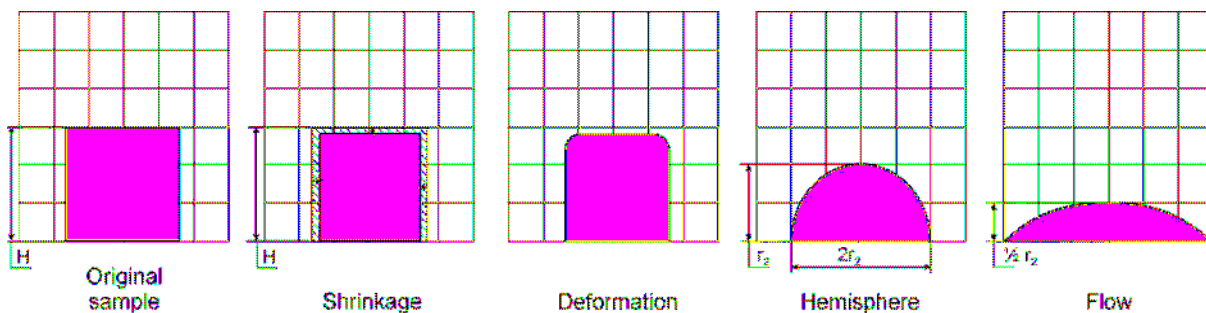


Figure 3: Phases in the ash melting process (original shape = shape and size at 550 °C)

Procedure

An electrically heated furnace is used, having following properties:

- reaching the maximum temperature at which the properties of the ash are to be determined (a temperature of 1500 °C or more may be required);
- provide an adequate zone of uniform temperature in which to heat the test piece(s);
- provide means of heating the test piece(s) at a uniform rate from 550 °C upwards;
- be capable of maintaining the required test atmosphere (see 7.1) around the test piece(s);
- provide means of observing the change of shape of the test piece(s) during heating.

A sufficient amount of ash is prepared according to the method specified in CEN/TS 14775. In deviation of CEN/TS 14775, larger plate type dishes or crucibles for the preparation of the ash can be used. The use of additives to ensure complete combustion is not allowed e.g. addition of water solutions to the ash may leach salts from the sample. Continuous ashing by refilling of the sample on the previous ash in the crucible is not allowed. The ash is grinded in an agate mortar or adequate grinding instrument until the maximum particle size is less than 0,075 mm. Nothing of the ash must be withdrawn during grinding.

A sufficient quantity of the prepared ash is moistened with demineralised water, dextrin or ethanol, make into a paste and is pressed into the mould with a defined pressure. The test piece is prepared by a hand press with spring pressure compression, the required spring pressure is about 1,5 N/mm². After drying test piece it is mounted on the support as vertically as possible. The test piece must neither be distorted, not react with nor absorbs the ash during the determination. Support of sintered alumina or fine textured mullite are generally satisfactory, but difficulties may arise with individual ashes, in which case zirconium supports or a non-absorbent interface such as platinum foil may be used between the original support and the test piece.

The test piece is transferred on its support to the furnace and adjust the composition and flow rate of the either oxidizing or reducing atmosphere at minimum linear rate of flow past the test piece between 100 – 250 mm/min, calculated at ambient temperature:

- An oxidizing atmosphere is obtained with air or carbon dioxide.
- The reducing atmosphere is obtained by introducing a mixture of
 - 55 % to 65 % carbon monoxide with 35 % to 45 % carbon dioxide or
 - 45 % to 55 % hydrogen with 45 % to 55 % carbon dioxide into the furnace at a

Due to safety reasons, reducing atmosphere is rarely used in labs.

The temperature is raised to 550 °C or a point which is minimum 150 °C below the expected shrinkage starting temperature, SST. Then rising temperature is proceeded at an uniform rate within the range of 3 °C/min to 10 °C/ min. The temperatures at which the characteristic changes in shape occur are recorded. During the whole. According to CEN/TS 15370-1 the determination of SST is only voluntary.

Due to experiences with biomass, in CEN/TS 15370-1 a paragraph can be found, indicating difficulties in determination of the characteristic points: With some ashes, difficulties may be encountered owing to such effects as blistering, distortion, shrinkage, swelling, non-wetting of the support (caused by high surface tension) and bursting of internal gas bubbles, and in such cases it is desirable to record these phenomena and possibly repeat the experiment using a different type of support.

2.1.2 Simple oven method - *ofi*-Method

Principle

A test piece made from an ash prepared according to EN 14775 is inserted into an oven at a preset temperature for a fixed time. This procedure is repeated with new samples at different temperatures selected as required. After removing and cooling a sample to room temperature, the sample is visually inspected and if necessary, the degree of sintering is determined by simple finger test.

Procedure

From the prepared ash (550°C ash according to pr EN 14775) test pieces are prepared. Since this method is currently not standardized, a special press tool is not a precondition. It was found that any rectangular tool is suitable, see Figure 4. At *ofi* a press tool is used providing a test specimen with 30 mm x 10 mm.

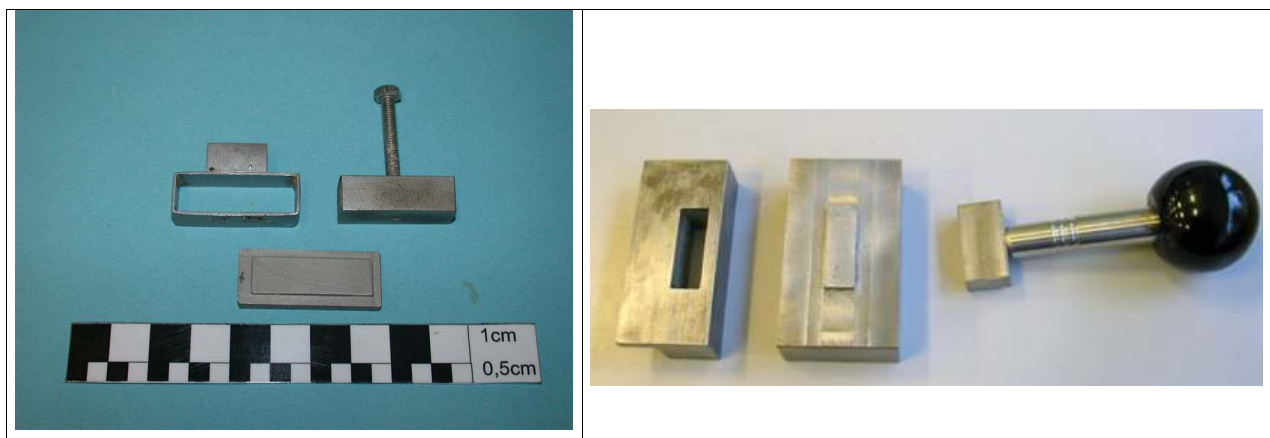


Figure 4: example for press tool, left - *ofi*, right - *dong*

The test piece used, shall have sharp edges to facilitate observation. It is put into heat resistant crucibles e.g. used for other thermal analysis as shown in Figure 5. The test piece must neither be distorted, not react with nor absorbs the ash during the determination. If it is not possible to produce a test piece from an ash sample 1-2 drops of ultra pure water may be added to the sample.



Figure 5: crucible for ring furnace, used by ofi

ofi performs tests from the high temperatures down to low temperatures, so starting temperature was 1400°C due to thermal limitation of the existing oven. The furnace is heated to 1400°C, in case of using a ring furnace tube shall be closed with special, fireproofed stopper. The temperature has to be checked in the tube with a thermo couple before analysis starts. With blank samples it was found, that the crucible reaches the final temperature after 3 minutes in the closed tube furnace. It was decided to leave samples for 2 minutes at the end temperature in the tube. The furnace heated to the desired temperature is opened, the sample is inserted into the tube, closed with the stopper as soon as possible and kept for 5 minutes. After 5 minutes the sample is removed from the oven, and cooled down to room temperature.

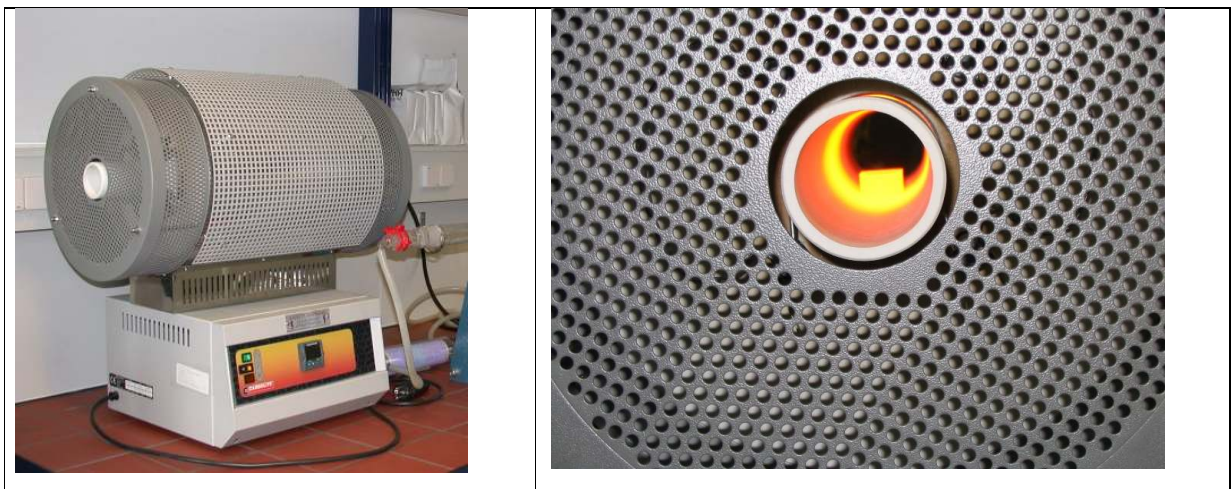


Figure 6: ring furnace used by ofi



Figure 7: Eltra CS 500-analyzer used by dong



Figure 8: Compression machine to determine the pellet compression strength used by CIEMAT

2.1.3 CIEMAT rapid tests for ash melting behaviour

The initial deformation temperature (IDT) in an oxidising atmosphere has been used as a reference to characterise the biomass ash fusion behaviour in laboratory, as well as to compare the temperature of biomass ash sintering obtained in laboratory with the experimental agglomeration and sintering results in combustors. However, sintering and ash agglomeration in power plants could occur at a temperature below the IDT. Moreover, the IDT possesses a high uncertainty. Consequently, a new temperature of fusibility is obligatory to predict the starting of sintering, e.g. shrinkage starting temperature (SST).

If the SST or other temperatures are not suitable in some biofuels, we will need new methods of sintering to predict the beginning of the sintering and have the possibility of comparing data between at least two methods. New methods can be divided into:

- 1) New sintering tests. They can be based on the measurement of physical properties such as ash thermal conductivity, ash electric resistance, ash viscosity (high temperature viscometer) and image and micro-elemental analyses (SEM-EDX).
- 2) Small pilot plant placed in laboratory (<100 kWth).
- 3) Theoretic methods. They can be based on element and compound concentrations, phase diagrams, software with thermo-dynamical data base.

However, most methods are complex and/or expensive methods. Besides, the prediction in commercial plants could also generates mistakes because this prediction depend on multiple factors such as location of ash within the plant or gradient of temperature in the ash deposited on a heat exchanger. Therefore, it would be convenient to remember simple and cheap methods again, e. g. CIEMAT sieving method, which is explained next.

Procedure

Two simple and cheap methods were used by CIEMAT. One of them is a traditional method called the disintegration method, and the second one is an extension of the first one to quantify their results. The disintegration method is a suitable method, which was validated by comparing their data with results obtained from combustion tests performed in a bubbling fluidised bed pilot plant (1 MWth).

Disintegration of pre-heated ash. This test was carried out heating in a laboratory furnace the biomass ashes previously produced at 550° C, at different temperatures: 800° C, 900° C, 1000° C and 1100° C. In this test loose ash was added to five porcelain capsules, 0.5 g ash/capsule. Then, the capsules were heated, at a heating rate of, approximately, 3° C per minute, maintaining 10 minutes the capsule into the furnace at each temperature. The atmosphere was air. Finally, the alterations produced in the resultant ash were observed such as the visual aspect and the easiness of manual disintegration. In regard to this last characteristic, four levels of difficulty were established: “very easy”, “easy”, “difficult” and “very difficult”. This method will be referred as the “disintegration” method, and it was always performed by the same laboratory operator.

The second one is called “ash durability” or the “sieving method” and it is based on the measure of the resistance of ash towards friction and/or disintegration, similarly to the mechanical durability of pellets and briquettes. At the end of this heating process, the heated ash is placed in a little sieve (6 cm diameter) with an aperture of 0.25 mm. The sieving lasted 5 minutes and the level of vibration was fixed at a 5 power level by using a CISA device, model RP 15 with digital regulation from 5 to 15 power level, seeFigure 9. Finally, it was calculated the following index:

Tendency to slag formation = mass of sample remaining on the sieve/initial mass *100



Figure 9: CISA device, model RP 15, used in the sieving method used by CIEMAT

2.2 Results

2.2.1 CEN/TS 15370-1

Results were provided from:

- CIEMAT
- SP
- *ofi* (TU-Wien)
- VTT

Table 1 Characteristic temperatures (CEN-TS 15170) obtained by CIEMAT.

	SST	IDT	ST	HT	FT
	°C	°C	°C	°C	°C
bark	1250	> 1400	> 1400	> 1400	> 1400
wood	1060	1360	nd	nd	> 1400
wood+bark	1200	1370	nd	nd	> 1400
rape straw	660	830	nd	nd	> 1400
straw	680	810	930	1080	1270
cynara	650	680	nd	nd	> 1400
olive residues	1210	1310	1340	1350	1350
coconut shells	870	910	940	950	970
almond kernels	740	720	nd	1310	1330
palm bit kernels	1210	1220	1240	1260	1280
seaweed	1150	1200	nd	1230	1260

(SST): shrinkage starting temperature, (IDT): initial deformation temperature, (ST): sphere t., (HT): hemisphere t., (FT): fluid t., (nd): not detected. SST based on 5% area contraction. IDT based on 1.5% shape factor variation and 10% corner angle variation.

Table 2 Characteristic temperatures (CEN-TS 15170) obtained by SP.

	DT °C	HT °C	FT °C
Cynara ash	1239	1277	1346
Cynara ash	1203	1251	1307
Cynara ash	1267	1295	1382
Cynara ash	1250	1294	1378
Industrial ash	1113	1169	1343
Industrial ash	1115	1181	1351
Industrial ash	1106	1167	1383
Industrial ash	1106	1162	1396
Staw ash	773	922	1113
Staw ash	-	1057	1108
Staw ash	817	914	-
Staw ash	816	926	1109
Dong ash	598	632	-
Dong ash	598	635	774
Dong ash	600	635	1189
Dong ash	595	764	1189
Orujillo ash	1179	-	1265
Orujillo ash	1193	1219	1241
Orujillo ash	1185	1219	1227
Orujillo ash	1156	-	-

Table 3 Characteristic temperatures (CEN-TS 15170) obtained by *ofi* (TU-Wien).

	SST	IDT	ST	HAT	FT
Orujillo	900	1047	1164	1180	1200
Coconut	600	1257		1272	1281
Cynara		1274		1309	1335
Dong (straw)	540	589	600	612	661
Industrial (grain/wood)		1020		1194	1329
Straw	640	790	855	935	1062

Table 4 Characteristic temperatures (CEN-TS 15170) obtained by VTT.

Sample		Shrinkage starting temperature, SST	Deformation temperature, DT	Hemisphere temperature, HI	Flow temperature, FT
Coconut Ash	A	675	1177	1210	1255
	B	665	1192	1250	1255
Cynara Ash	A	1151	1240	1302	1370
	B	1101	1240	1296	1360
Dong Ash	A	565	570	640	1145
	B	563	570	640	1145
Industrial Ash	A	979	1080	1179	1358
	B	984	1100	1175	1349
Orujillo Ash	A	1029	1145	1207	1200
	B	1004	1145	1200	1239
Straw Ash	A	680	810	1042	1140
	B	657	810	1053	1126

2.2.2 Simple oven method - *ofi*-Method

Results were provided from:

- Dong
- SP
- *ofi* (TU-Wien)
- CIEMAT

The following figures show selected examples of samples heated according to the simple oven method.



Figure 10: Orujillo, *ofi*



Figure 11: Orujillo, SP



Figure 12: cynara, DONG Energy



Figure 13: Cynara, SP

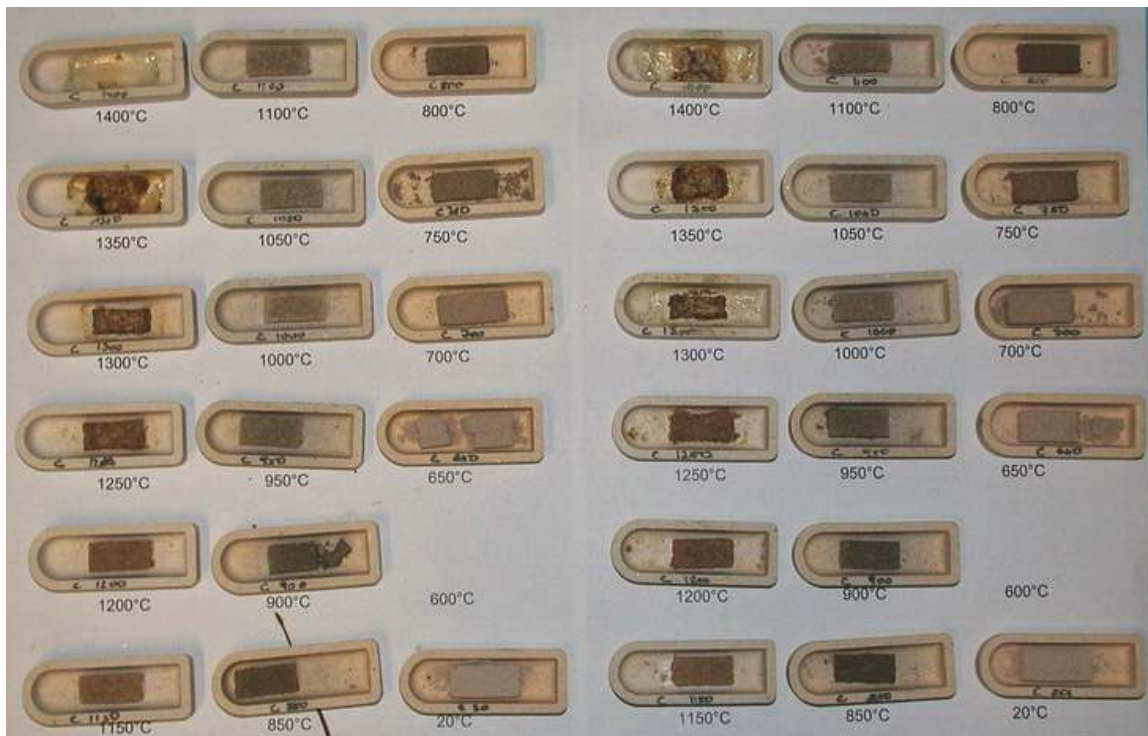


Figure 14: Cynara, ofi

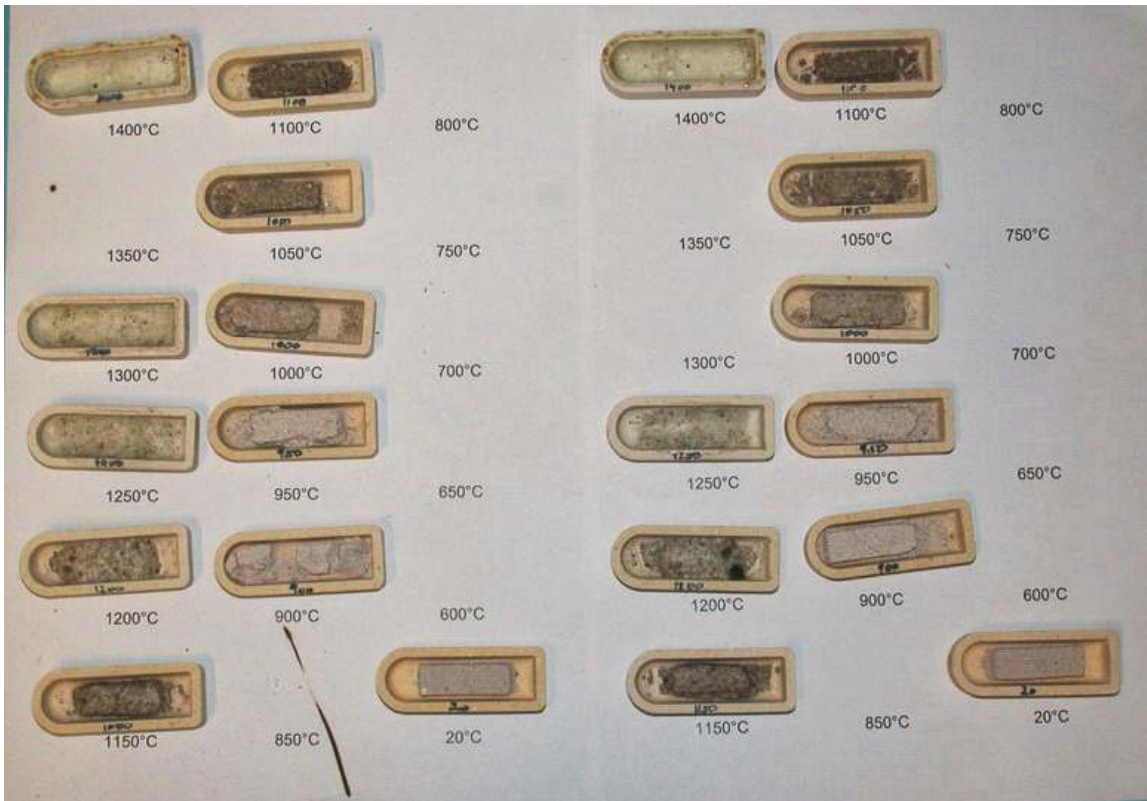


Figure 15: Industrial ash, ofi



Figure 16: Biomass ash prepared VTT, measured by ofi

Table 5 Example of determination of characteristic temperatures by simple oven method (ofi).

	SST	IDT	ST	HAT	FT
Orujillo	900	950		1200	1250
Coconut	700	800		1050	1100
Cynara	650	-		-	1350
Dong (straw)	-	600		675	700
Industrial (grain/wood)	900	1050		1200	1250
Straw	650	700		950	1000

2.2.3 CIEMAT rapid tests for ash melting behaviour

With the aim of quantifying the durability method, their results are compared to the disintegration results, see Figure 17. According to this figure, the following levels can be established:

- | | |
|-------------------------------------------------------------|--------|
| (1) dust-very easy disintegration: | < 45% |
| (2) weak sinter-easy disintegration: | 45-80% |
| (3) hard sinter-difficult disintegration: | 80-95% |
| (4) very hard sinter or slag-very difficult disintegration: | > 95% |

Similar ash durability results corresponding to the four levels have also been obtained when other samples were tested in laboratory. However the ash durability method is difficult for normalization due to the influence of several factors such as thermal effect of the furnace, distribution of ash particle sizes, phase changes and chemical reactions involved in the ash placed in the capsule. Another important variable is the vibration level of the sieving equipment, which generates the friction to disintegrate partially the heated ash. Wood results shown in Table 6-2 does not seem to detect the beginning of sintering at temperatures lower than 1100° C and its SST is 1060° C.

According to Table 6 Comparison between durability method and disintegration method., bark seems to indicate the beginning of the sintering at 1100°C, coconut at 900° C and seaweed at 1000° C. SSTs indicate 1250°C, 870°C and 1150° C for bark, coconut and seaweed, respectively. Conversely to wood, the beginning of sintering in bark, coconut and seaweed is detected at temperatures near or lower than SST. Therefore SST does not seem very reliable in wood sample and the comparison with other method could be necessary.

Table 6 Comparison between durability method and disintegration method.

	800° C		900° C		1000° C		1100° C	
	Durability	Disintegration	Durability	Disintegration	Durability	Disintegration	Durability	Disintegration
bark	3	VE	2	VE	5	VE	68	E
wood	14	VE	28	VE	27	VE	30	VE
wood+bark	7	VE	4	VE	8	VE	25	VE
rape straw	95	VD	98	VD	99	VD	98	VD
straw	44	E	100	VD	100	VD	100	VD
cynara	96	VD	100	VD	99	VD	99	VD
olive residues	31	VE	36	VE	42	VE	58	E
coconut shells	8	VE	89	D	100	VD	100	VD
almond kemels	94	D	98	VD	97	VD	97	VD
palm bit kemels	4	VE	9	VE	12	VE	21	VE
seaweed	32	VE	47	E	94	D	99	VD

DISINTEGRATION.- (VE): very easy disintegration, (E): easy, (D): difficult, (VD): very difficult.

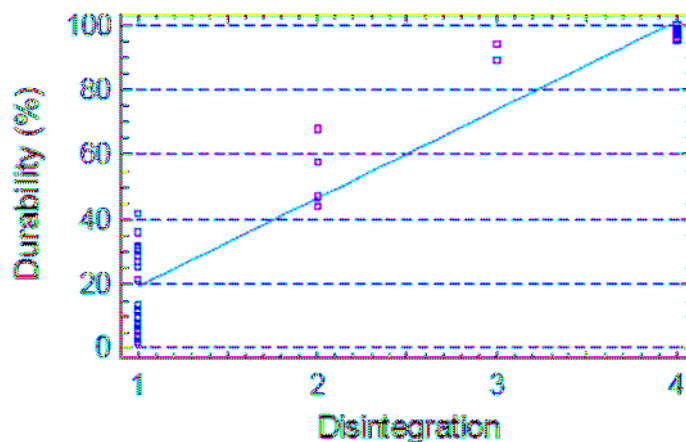


Figure 17: Comparison between durability method and disintegration method, Ciemat

2.3 Method comparison

		SST	IDT	ST	HAT	FT
Orujillo						
VTT (averg.)	CEN/TS 15370	1016	1145		1203	1220
TU-W	CEN/TS 15370	900	1047	1164	1180	1200
ofi	quick method	900	950		1200	1250
dong	quick method	1200	1250		1300	1350
Ciemat	quick method		< 1100			< 1400
Coconut						
VTT (averg.)	CEN/TS 15370	670	1185		1230	1255
TU-W	CEN/TS 15370	600	1257		1272	1281
ofi	quick method	700	800		1050	1100
Ciemat	quick method		< 800			
Cynara						
VTT (averg.)	CEN/TS 15370	1126	1240		1296	1360
TU-W	CEN/TS 15370		1274		1309	1335
ofi	quick method	650				1350
dong	quick method	1100	1250		1350	1400
Ciemat (old sample)	CEN/TS 15370	650	680			> 1400
Ciemat	quick method		< 800			< 1400
Dong (straw)						
VTT (averg.)	CEN/TS 15370	564	570		640	1145
TU-W	CEN/TS 15370	540	589	600	612	661
ofi	quick method	alle	600		675	700
dong	quick method		< 650			< 750
Ciemat	quick method		< 800			
Industrial (grain/wood)						
VTT (averg.)	CEN/TS 15370	982	1090		1177	1353
TU-W	CEN/TS 15370		1020		1194	1329
ofi	quick method	900	1050		1200	1250
dong	quick method		1050		1200	1250
Ciemat	quick method		< 900 / 1150			< 1300
Straw						
VTT (averg.)	CEN/TS 15370	658	810		1048	1133
TU-W	CEN/TS 15370	640	790	855	935	1062
ofi	quick method	650	700		950	1000
dong	quick method					
Ciemat	quick method		< 800			< 1100