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CHARACTERIZATION OF RESIDUAL BIOMASS FROM AGRICULTURAL AND AGROINDUATRIAL ACTIVITIES

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Abstract. Currently, fossil fuels such as oil, coal and natural gas represent the main energy sources in the world. However, renewable energy sources, including biomass, have to be used to reduce environmental damage. Biomass wastes generated in the agricultural and agroindustrial activities are left in the field or discarded, usually without any value. In this paper a characterization as solid fuels of the main residual biomasses generated in Uruguay is approached. Six analyses were performed to characterize the biomass samples and their ashes: moisture, proximate and ultimate analysis, heating value, thermogravimetric analysis and ash melting temperature determination. It was found that olive and grape pomace have the best properties as potential fuels (even several properties better than some traditional biomass fuels), followed by vine and olive prunings, then grape stems and switchgrass. On the other hand, cereal crop straws and wild cane did not show so favorable characteristics, specially on the heating values and ash contents.

Keywords: Biomass, Solid fuel characterization, Agriculture residues

1. INTRODUCTION

The rational use of fossil fuels and renewable natural resources, among other actions such as the reduction of green-house gas emissions and conservation of soil and water, are crucial for a sustainable development. Energy production is one of the most intensive uses of natural resources in human activities. Moreover, it is also the biggest source of pollutants emission (Cortez *et al.*, 2008). Biomass is a renewable energy source because its availability is not limited in time, we can always grow trees and crops, and waste will always exist (Saidur *et al.*, 2011).

The characteristics and quality of the biomass influence directly in the thermoconversion technologies. Moreover, as the less homogeneous the fuel is and with a lower quality, there has to be a more sophisticated system for its combustion or gasification (Basu, 2010; van Loo and Koppejan., 2008).

In this context, to evaluate a solid fuel, the first, and most important step is to perform a detailed characterization of it. In this way, their properties and biomass quality as fuel, as well as their potential applications and the environmental problems that may be associated, are determined. The characterization is based on the knowledge of the chemical, physical, geometric and mineral characteristics of the fuel (Saidur *et al.*, 2011)

In Uruguay, in 2015, renewable energy accounted for 57 % of the energy matrix, from which 14% corresponded to hydropower and 40% of biomass, mainly from black liquor, but also from wood and charcoal (MIEM, 2015). Agriculture plays an important role in Uruguay, where a great amount of biomass waste is generated and can be used as an energy source. Soybean is the major crop which has more than 1.300.000 ha cultivated, followed by wheat (400.000 ha). Sorghum, corn and barley are crops with also a relevant presence in Uruguay. Others agroindustrial activites, like wine and olive oil producction, also generate residual biomass, such as pomace and prunings (MGAP, 2015). All these biomass are generaly left in the field or discarded without any value. In this paper the characterization of these wastes as solid fuels is approached.

2. EXPERIMENTAL PROCEDURE

2.1 Sampling

The analyzed samples were solid residues generated in agricultural and agroindustrial activities from Uruguay: straw of soybeans, barley, corn, wheat and sorghum, vine and olive prunings, grape stems, olive and grape pomace, as well as wild cane (arundo donax) and switchgrass (both crops with high growth rate and high lignin content) (see Table 1).

Table 1. Biomass samples list.

Biomass	Type	Nomenclature
Olive Pomace	Frantoio	O.Pom.
Olive Prunings	Arbequina	O.Prun.
Grape Pomace	Cabernet Frenc	G.P.
Grape Stems	Tannat	G.S.
Vine Prunings	Tannat	V.P.
Corn Straw	SPS 2866	C.S.
Sorghum Straw	Topper (TRC)	Sg.S.
Wheat Straw	DM fuste	W.S.
Barley Straw	CLE 233	B.S.
Soybeans Straw	DM 6.2i	Sb.S.
Switchgrass	Shawnee	Swch.
Cane	Arundo donax	Cane

The samples preparation was made with the ASTM - E1757 standard. First, the samples were dried in an oven at $60°C$ until constant weight to determinate *in natura* moisture content (ISO 18134-1 and 2). Then, the samples were ground in a knife mill to homogenize them. Quartering was not necessary to select the samples because the ground material was enough homogeneous.

2.2 Analysis

Six analyses were performed to characterize the biomass samples and their ashes: moisture *in nature*, proximate and ultimate analysis, heating value, thermogravimetric analysis and ash temperature fusion determination. Self-ignition temperature as well as relevant points in the thermal degradation were determined from the thermogravimetric plots, in inert and oxidant atmosphere.

The mass percentages of moisture, volatile matter, fixed carbon and ash content were determined in the proximate analysis. It was performed under standards ISO 18134-3, ISO 18122 and ISO 18123 employing a muffle and an analytic balance (Radwag AS 310, resolution of ± 1 mg) to determinate the mass. Eight repetition were made with $1,1g$ each one in ceramic crucibles.

The ultimate composition (C, H, O, N, S) was provided directly by the dry sample employing an Elemental CHNO analyzer. The high heating value was obtained by the calorimetry pump methodology under the standard UNE-EN 14918 with a PARR 6100 calorimetry. The thermal analysis were provided by an analyzer Shimadzu TA 50 doing thermogravimetry (TGA). The heating rate was $10°C/min$ and the samples masses $10\pm 0,5$ mg. Both nitrogen and synthetic air were employed as atmosphere gas, with a flux of 50,0 ml/min, in the temperature range from ambient to 900°C.

The ash melting temperature were determined by a LECO AF 700 under oxidizing atmosphere. The temperature was increased from $600°C$ up to $1490°C$ emploing a heating rate of $10°C/min$. The ashes for this analysis were prepared at $550°C$ and sieved (Mesh 200). This analysis was made under ASTM D1857-04.

3. RESULTS AND DISCUSSION

3.1 Moisture *in natura*

The main results for the fuel characterization are presented in Table 2. It can be seen that the highest moisture content are from those industrial wastes where the main process is juice extraction (olive oil and wine). Grape stems have higher humidity content with 75%, followed by olive pomace and grape pomace with 66% and 53% respectively. Otherwise, the water content of vine and olive prunings, decreases considerably to 21% and 31% respectively.

On the other hand, regarding cereal crops, it is possible to classify them in two groups. On one side, soybean, barley and wheat with exteremely low humidity content (between 8% and 9%) and on the other, corn and sorghum with humidity content between 27% and 29%.

Table 2. Biomass moisture (w.b.), proximate composition (d.b.), ultimate composition (d.b.a) and high heating value (d.b.)

Biomass	W	Poximate Composition (% d.b.)			Ultimate Composition (% d.b.a.)				HHV
	(% w.b.)	FC	VM	Ash	C	H	O	N	(MJ/kg)
O.Pom.	66	13,6	84,4	2,0	64,9	8,8	24,3	2,0	25,3
O.Prun.	31	14,9	80,9	4,2	54,8	7,0	36,2	2,0	21,4
G.P.	53	19,2	74,5	6,3	56,8	6,8	34,1	2,3	21,6
G.S.	75	23,9	71,3	4,8	52,6	5,4	40,7	1,3	18,8
V.P.	21	20,4	74,6	5,0	54,7	5,6	38,8	1,0	19,4
C.S.	27	16,5	78,7	4,7	50,1	5,2	43,8	0,9	18,2
Sg.S.	29	15,0	78,0	7,0	50,6	5,9	42,8	0,7	17,9
W.S.	8,1	15,7	75,1	9,2	49,8	5,9	43,2	0,2	17,7
B.S.	8,8	15,5	77,1	7,4	48,8	6,1	44,8	0,3	18,2
Sb.S.	9,8	15,4	80,8	3,8	51,4	6,2	41,7	0,7	19,8
Swch.	9,7	15,2	79,1	5,8	51,1	5,8	42,6	0,6	18,4
Cane	27	13,5	82,9	3,5	48,4	5,8	45,3	0,6	17,8

These differences are associated with intrinsic conditions of each crop at harvest, but it is likely that their natural drying velocity is high and that the samples were not collected all at the same time after harvest. Likewise, bibliografy references data presents values for wheat straw humidity very low (Basu, 2010). Finally, switchgrass, with a humidity lower than 10%, was also one of the biomass with lower water content.

A high value of moisture in the biomass, with energetic purpose, has associated a series of problems, related to low capacity of ignition and instability of the combustion, reduction of the flame temperature, decreasing of gross heating value, loss of equipment efficiency, higher costs of transportation, problems in handling, among others.

Natural drying of biomass strongly depends of weather conditions, like temperature, solar radiation and rainfall, and also of the storage conditions, if it is scattered in the field or stacked, its size and geometry, as well as the compaction (Ortíz *et al.*, 2003). Ortíz *et al.* (2003) mention that in Spain it is difficult to obtain humidities below 20% for wood with natural drying. In case of need, forced drying technologies allow to obtain smaller humidities.

Considering the biomasses studied in the present work, it is expected that for grape and olive pomace, which are arranged in piles and with a high initial humidity, the natural drying will be difficult, therefore a forced drying would be necessary. On the other hand, in residual biomass of cereal crops, which are scattered in the field, a fast reduction of moisture is expected, but dependent on precipitations.

Biomasses frequently used for fuel, such as eucalyptus wood and bark, as well as sugarcane bagasse, have initial humidities above 50% (Pena-Vergara *et al.*, 2014; Brito *et al.*, 1979). In the case of eucalyptus, or other wood, it is generally expected that its moisture will be reduced before being used.

3.2 Ultimate composition

Olive pomace has the highest carbon and hydrogen contents, showing atypicals values for biomass (64,9 and 8,8% respectively). The high values observed can be related to the remaining oil presence.

Compared with some biomasses frequently employed as fuel, it is highlighted that most of the waste biomass studied in this work, have more content of carbon and less of oxygen. This is a positive point to these alternative biomasses because it has as a consequence a higher calorific value. On the other hand, biomass from vines and olive crops, show high percentages of nitrogen, which is a negative point due to the soil nutrient extraction. Straws, as well as switchgrass and cane, have a content of nitrogen lower than the traditional biomasses.

The relationship between the content of carbon, hydrogen and oxygen of a solid fuel can be ploted as shown in Figure 1. It can be seen that all samples are located in the biomass region, except olive pomace and there is also a slight deviation in corn straw. Olive prunings and grape pomace are the samples with atomic O/C ratio between 0,4 and 0,5. Increasing the atomic O/C ratio, it follows the olive prunings and grape stems. Finally and more grouped, it can be found straws, switchgrass and cane.

3.3 Proximate composition

Biomass is generaly characterized by a high volatile matter content, this is not the exception in the analyzed samples results, all being above 70 % in dry basis, more specifically between 74 and 81 % (except the olive pomace with 84.4%).

The ash contents of the samples analyzed are in the range of 2% and 9% approximately, which is a reasonable value for some traditional biomasses, lower than rice husk and similar to sugarcane bagasse or eucalyptus barck (Bizzo *et al.*,
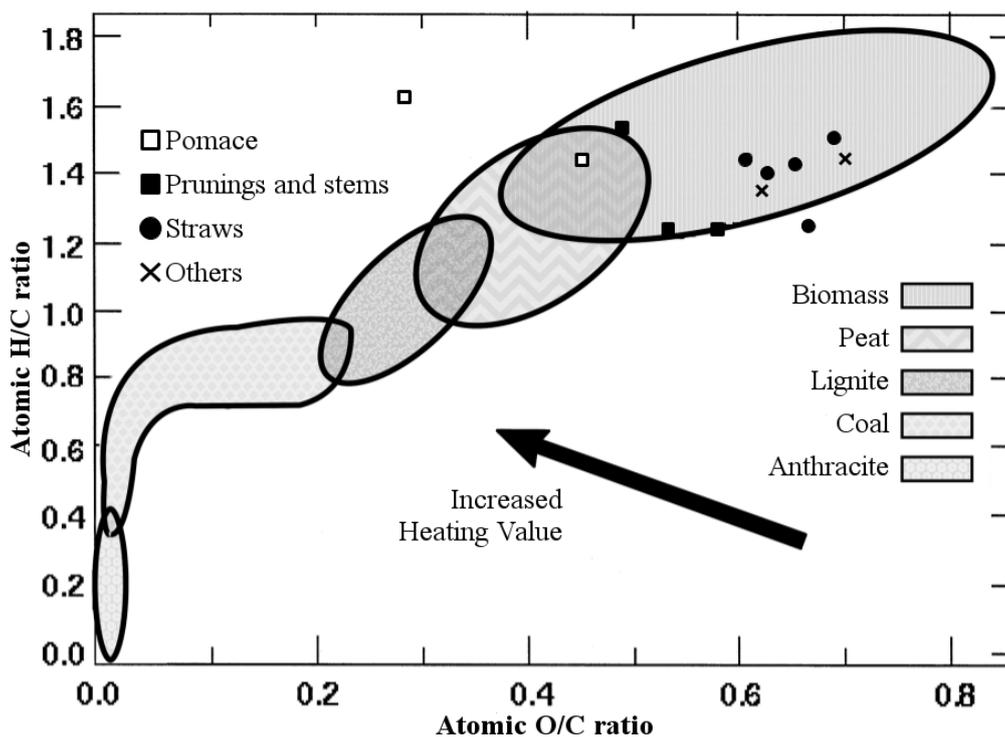
Figure 1. Biomass classification through H/C and O/C atomic ratios (Jenkins *et al.*, 1998).

2014; Almeida *et al.*, 2010; Pena-Vergara *et al.*, 2014; Brito *et al.*, 1979; Basu, 2010; Cortez *et al.*, 2008).

The waste biomass from wine production (grape pomace, stems and prunings) are the samples with the highest fixed carbon content, between 19 and 24%. The rest of the samples have an oscillating fixed carbon content, between 13 and 16%.

3.4 Heating value

Olive pomace has the highest carbon and hydrogen contents, as well as the lowest ash content. In terms of energy, olive pomace also has the highest heating value (HHV), being even higher than some traditional biomass, such as eucalyptus or pine wood (HHV around 20 MJ/kg) (Cortez *et al.*, 2008; Basu, 2010). Grape pomace and stems, as well as vine prunings, have high atomic carbon and fixed carbon contents. These biomass show HHV between 18,8 and 21,6 MJ/kg like olive prunings (21,4 MJ/kg). Regarding the straws, the soybeans straw has the major HHV (19,8 MJ/kg) while the wheat, barley and sorghum samples show values between 17,7 and 18,2 MJ/kg. Among all the samples studied in the present work, the straws and wild cane present the lowest HHV.

3.5 Thermal analysis

Figure 2 and 3 show the evolution of the thermal degradation of olive prunings and wheat straw (as particular cases), obtained from thermogravimetric analysis (TGA and DTG), in both oxidant (air) and inert (nitrogen) atmospheres. The thermal degradation of the biomasses have some behaviours in common between all of them and some particularities depending on each sample. Allways, a first mass loss is observed due to the drying. This occurs at temperatures close to 100°C and it is manifested as the first peak in DTG curves. The evaporation phenomenon is independent of the atmosphere type, therefore the curves remain together in the evaporation process. From that point, the thermal degradation differs according to the atmosphere.

For air atmosphere, two groups of peaks can be identified (excluding the evapotarion peak): those corresponding to volatile matter combustion, between 180 and 350°C, and those corresponding to fixed carbon combustion, arround 400°C. The first peak is also called as the first reaction zone and the second peak the second reaction zone (Ergiidenler and Ghaly, 1994). Olive pomace and pruning are the samples with the greatest difference between start and finish temperature in the first peak, this means that pyrolysis and combustion of volatiles show a great thermal amplitude, followed by the grape pomace and stems, and corn stubble. Finally, biomasses like grape prunings, straws of barley, sorghum, soybeans, switchgrass and cane are the samples that show shorter thermal amplitude in that first reaction zone. Temperatures of peaks oscillate between 270 y 305°C. The second reaction zone (fixed carbon combustion), which has a similar thermal amplitude compared to the first one, starts at 445°C and finishes at 515°C having a maximum at arround 390-430°C.
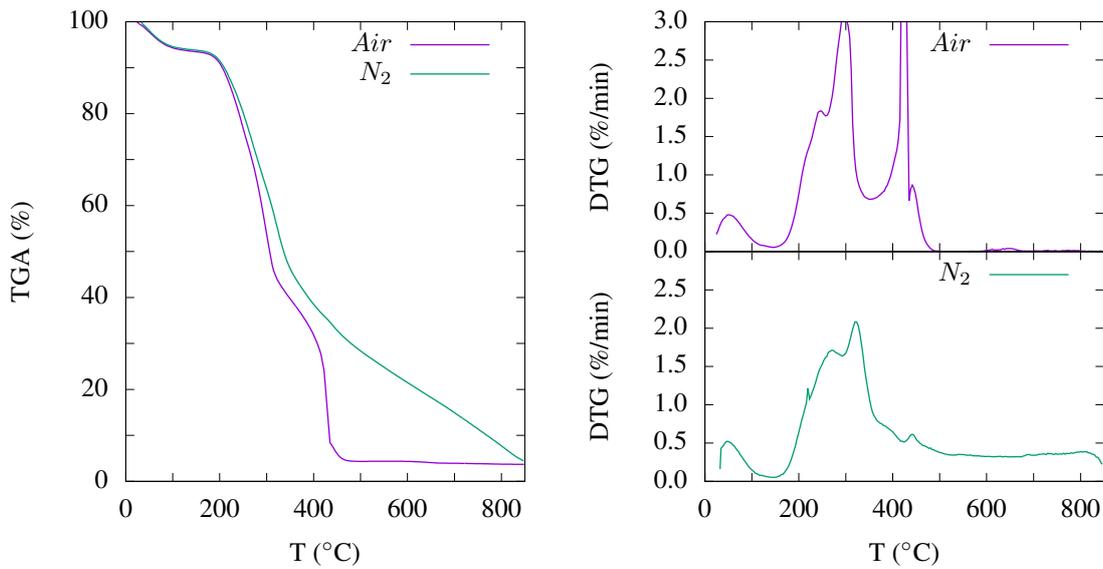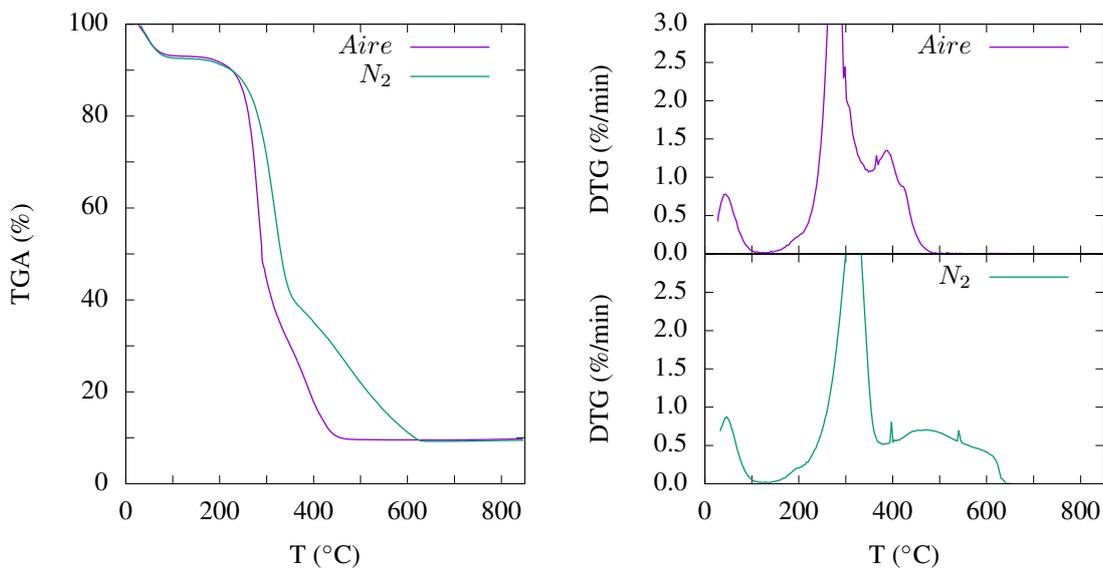
Figure 2. Olive Prunings Thermogravietry.

Figure 3. Wheat Straw Thermogravimetry.

For nitrogen atmosphere, it is seen that pyrolysis occurs mainly between 180°C and 400°C. A first peak value is observed at approximately 220-315°C due to the decomposition of hemicellulose and a second one is noticed at approximately 315-400°C as a consequence of the decomposition of cellulose. At this last temperature range the highest rate of mass loss is observed. The mass loss continues at constant rate until it achives the same mass percentage that there is in the oxidating atmosphere.

Table 3 summarizes, for all samples, the most relevant temperature ranges and mass loss percentajes in both atmosphere types, as well as the temperatures that correspond to the peaks.

The self ignition temperature can be estimated by differents ways (Grotkaer *et al.*, 2003; Mortari *et al.*, 2011; Huang *et al.*, 2008). In this work it was determinated by the intersection of both tangents before and after the pyrolisis starts. Results are shown in Tabel 3, where is observed that most of the samples have the expected values for biomass, between 230°C and 260°C, like pine or eucalyptus wood (Basu, 2010; Jones *et al.*, 2015). Only the olive pomace and prunnings show lower self-ignition temperatures from the expected ones, being 215°C and 195°C respectively.

3.6 Ash melting temperature

The characteristic temperatures of ash were determined for each biomass sample by test cone method. These temperatures are: deformation temperature (DT), softening temperature (ST), hemispheric temperature (HT) and flow temperature

Table 3. Thermogravimetry

Biomass	T_{s-i} (°C)	Oxidant atm. - 1st Peak			Oxidant atm. - 2nd Peak			Inert atmosphere		
		Range (°C)	Mass Loss (%)	Peak (°C)	Range (°C)	Mass Loss (%)	Peak (°C)	Range (°C)	Mass Loss (%)	Peak (°C)
O.Pom.	215	160-355	65,4	305	355-485	30,9	410	160-480	75,4	370
O.Prun.	195	170-350	58,2	295	350-475	37,1	420	170-517	71,3	320
G.P.	240	190-365	48,1	280	365-515	42,7	440	190-500	62,7	275
G.S.	230	175-357	53,4	275	357-510	39,4	424	175-380	46,7	335
V.P.	260	200-317	59,4	300	317-500	34,3	425	200-380	53,8	335
C.S.	250	180-330	66,4	275	330-490	27,3	400	180-390	63,1	300
Sg.S.	245	200-330	80,5	290	330-510	13,1	390	200-380	62,0	330
W.S.	255	175-350	67,3	280	350-495	22,3	390	175-380	59,6	315
B.S.	250	200-310	79,0	290	310-490	12,2	410	200-390	64,5	310
Sb.S.	250	190-330	84,3	295	330-495	10,4	430	190-385	65,3	340
Swch.	240	170-315	74,9	295	315-490	19,3	405	170-400	65,4	335
Cane	240	175-320	65,2	300	320-505	26,8	400	175-375	61,6	330

(FT). The results obtained are shown in Table 4 as well as some comparative values from bibliography. Vassilev *et al.* (2013) and Garcia-Maraver *et al.* (2017) mention that some biomasses like straw from cereal crops have low ash fusion temperatures. Otherwise, wood and bark trees, as well as the grain husks are characterized by higher ash fusion temperatures.

In the present work, both pruning olive and vine were the samples that show the higher initial deformation temperatures, 1230°C and 1325°C respectively. On the other hand, grape and olive pomace, as well as stems, had low ash melting temperatures, starting the deformation at temperatures below 900°C. Straws show also low ash melting temperatures, DT between 810 and 1050°C, ST from 980°C up to 1130°C, HT from 1090 up to 1210°C and FT from 1140°C up to 1350°C. The soybeans straw results are striking, based on the bibliografy data, it were expected values under 1000°C for DT. Same samples did not achive the fluidization temperature under 1500°C. Figure 4 shows the ash cone evolution as the temperature increases, identifying the four characteristic temperatures.

Table 4. Ash fusion temperature (°C).

Biomass	DT	ST	HT	FT
O.Pom.	845	-	-	-
O.Prun.	1325	>1500	>1500	>1500
G.P.	902	1455	1476	1490
G.S.	857	-	-	-
V.P.	1233	>1500	>1500	>1500
C.S.	852	1036	1098	1141
Sg.S.	1048	1132	1152	1196
W.S.	974	1078	1213	1346
B.S.	811	982	1090	1250
Sb.S.	1328	1473	>1500	>1500
Swch.	794	1110	1270	1367
Cane	1025	1329	1405	1454

One way to clasificate the biomass, by the ash melting temperature, is by considering HT in five differents categories. That five categories are: very low (<1000°C), low (1000-1200°C), moderate (1200-1400°C), high (>1600°C) and very high (1400-1600°C) (Vassilev *et al.*, 2013). Under this clasification and the results shown in Table 4, both prunings biomass con be considered as high melting temperature, as well as the ash from grape pomace, soybeans straw and cane. In the moderate range, it can be found the switchgrass and wheat straw ashes. Finally, the other ashes fit in the low melting temperature.
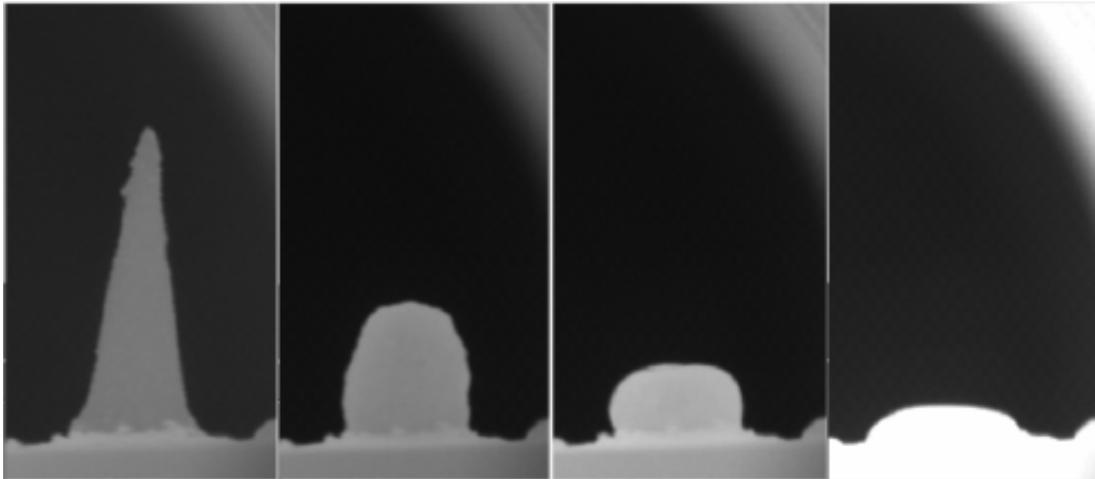
Figure 4. Pictures of wheat straw ash evolution by increasing temperature.

4. CONCLUSIONS

The main conclusion of the present work is that all biomass wastes studied are adequate as solid fuels. Samples can be divided in two grups. On one hand, biomass that comes from olives and vines and on the other hand straws, switchgrass and cane *Arundo Donax*. Rusults for the first group can be summarized as:

- Present moisture contents considerably high. Grape stems and olive pomace shows values higher than 60%, so they should be dried before being used as fuel. The others exhibit values nearby 30%.

- From the point of view of heating values, olive and grape pomace, and olive prunings show the best results, higher than some traditional biomass like *eucalyptus*. These samples also showed high carbon contents and the olive wastes have also low self-ignition temperature (arround 200°C).

- Ash content between 2,0 y 6,3%, where the lowest value correspond to olive pomace. Olive prunings shows ash melting at high temperature, while for the others, the melting point was achived at lower temperatures.

The main results for straws, switchgrass and cane *Arundo Donax* are:

- The cereal straw and switchgrass moisture is low at harvest time or a few days later. For soybeans, wheat and barley straw the humidity values are approximately 10%, whereas for straw coming from sorghum and corn achieve, as well as for the cane, values between 25% and 30%.

- High ash content (between 3,8% y 9,4%) compared to traditional woody biomass, but similar to other biomass like bark and sugar cane bagasse. Cane and soybean straw were the samples with lower ash content and wheat straw the one with worst results. These ash had low or moderate melting temperature, where the soybeans straw was the sample that achived the best results. The fact that in general straws, switchgrass and cane presents high ash content with low melting temperatures, is an important limitation for its use as a fuel.

- The heating value for biomass from cereal crops, switchgrass and cane, is comparable with wood biomass reaching values between 17 and 19MJ/kg. Again the soybean straw shows the best results.

- Regarding the thermal degradation, these biomass have an important mass loss at low temperature (first zone) and self ignition temperature between 240 and 255°C.

5. ACKNOWLEDGEMENTS

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