Analysis of some civil and industrial wastes ashes by using the heating microscope

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Introduction

The waste-to-energy process consists of the combustion of some types of refuses in order to have an energetic gain. To get the most out of some types of civil or industrial refuses as alternative fuels, it is necessary to know well each characteristic of them.
The aim of this work is to study the thermal behaviour of some ashes, which derive from the combustion of such refuses.
It is necessary to find a technique which not only determines the melting and softening points of the ashes (parameters which influence strongly the choice of a fuel), but also is able to give information on the behaviour of the samples during the whole thermal cycle.
The experimental method used in this study is the analysis with the heating microscope, which is a very helpful instrument to follow the behaviour of materials subjected to a heating cycle [1].

Experimental method

The heating microscope used to perform the analyses is the model MISURA HSM (see fig. 1), manufactured by Expert System Solutions.

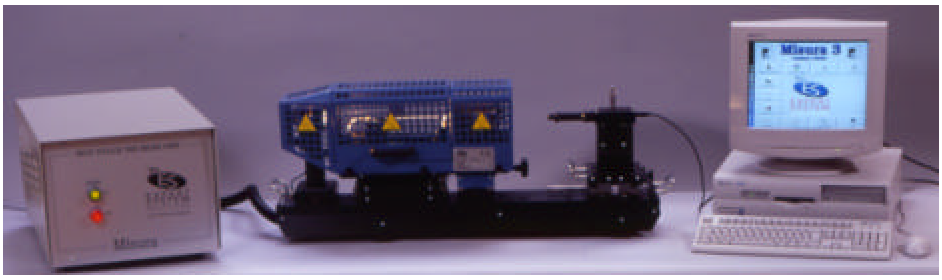
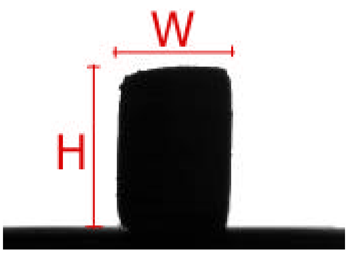


fig. 1: Heating microscope MISURA HSM of Expert System Solutions

This instrument is able to acquire and store the images of a specimen subjected to a heating cycle, at predetermined time or temperature intervals.
All the dimensional parameters (height, length…) are measured automatically during the test, in order to identify some characteristic points of the material.
The heating microscope MISURA HSM consists of a kiln equipped with an automatic programmer, which can reach a heating rate of 80°C per minute and a maximum temperature of 1750°C.

Referring to the norm ASTM D1857 and fig. 2, it is possible to define the following characteristic temperatures:



- "initial deformation temperature": the specimen height, H, assumes a value of 75% with respect to its value at the beginning of the test;
- "softening temperature": W=H;
- "emispherical temperature": W=2H;
- "fluid temperature": H assumes a value of 8,38% with respect to its value at the beginning of the test.

fig. 2. Dimensions of a specimen for heating microscope.

From the memorized data, the instrument provides also the curve of the dimensional variations of the sample as a function of the temperature, which is called "flattening curve".

The samples studied in this work derive from:

1) two types of industrial wastes, in particular biomasses (olive husk and chipped wood);
2) three types of "RDF" (refuse derived fuel);
3) a conventional fossil fuel.

fig. 3

fig. 4

fig. 5

The ashes of the biomasses analysed are shown in fig 3 (on the left: olive husk ashes, on the right: chipped wood ashes), the ones of RDF in fig. 4 and, finally, the ash of fossil coal in fig 5.

All the samples were kindly provided by the "Stazione Sperimentale per i Combustibili" (SSC) of San Donato Milanese (MI).
The various samples are subjected to the following heating cycle, as required by the norm ASTM D1857):
- rapid heating (50°C/min) up 400°C;
- heating rate of 8°C/min up to the fluid temperature of the material.

A difference from the norm was adopted in the specimens form: instead of the traditional pyramidal mould, a cylindrical one has been preferred. In this way, the preparation of the specimens results much simpler and quicker.
With the addition of some water drops for increasing the plasticity, the ground ashes were pressed with a manual press, in order to obtain a little cylinder 3mm high, with a diameter of 2mm.

The support for the samples consists of an alumina substrate; the data acquisition was carried out every 5°C.

Results

1) Ashes derived from RDF

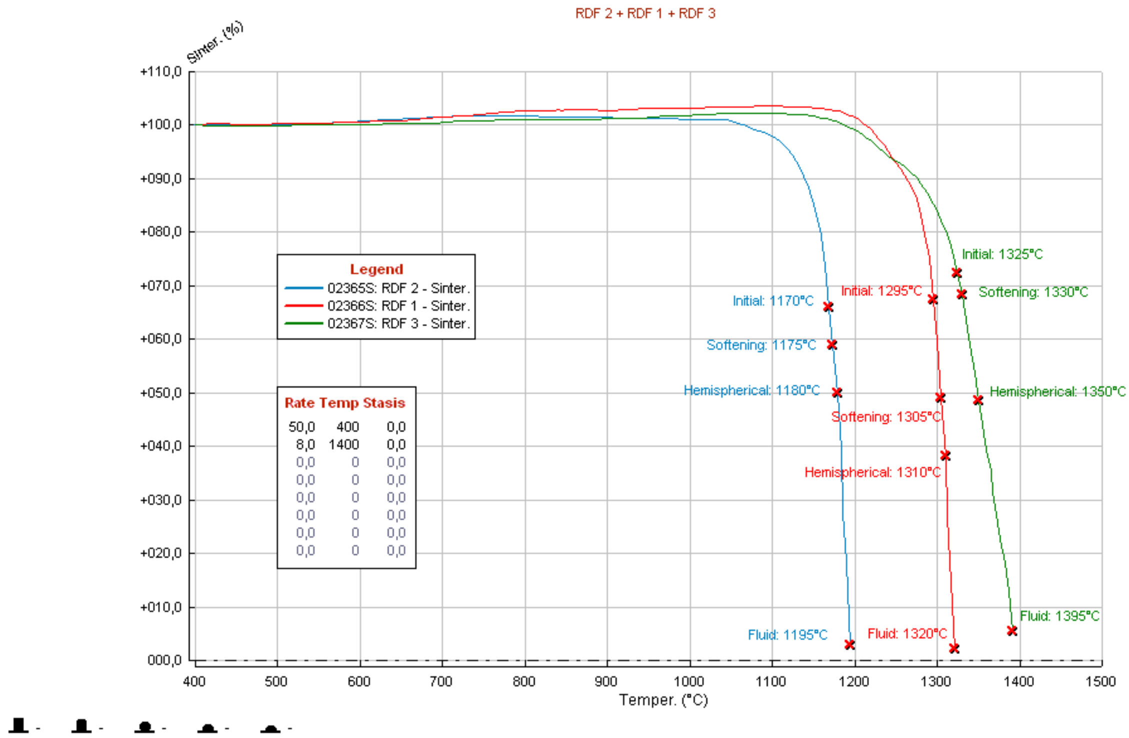


fig. 6: Flattening curves of RDF ashes

Fig. 6 shows the three curves obtained, in which the expansion and the contraction are expressed in percentage with respect to the height of the specimen at the beginning of the test.

It is possible to see that the RDF ashes start to soften at a temperature interval between 1175 and 1330°C. The fluid temperature, instead, is found between 1195 and 1395°C.

This big difference can be explained considering that the ashes have a different chemical composition, which depends on the types of waste they derive from.

The difference of colours of the samples (see fig. 4) indicates the presence of different chemical elements inside them.

In fig. 7, the summarizing table of the behaviour of the sample "RDF 1" is shown. The forms assumed by the specimens in correspondence of the characteristic temperatures are reported.

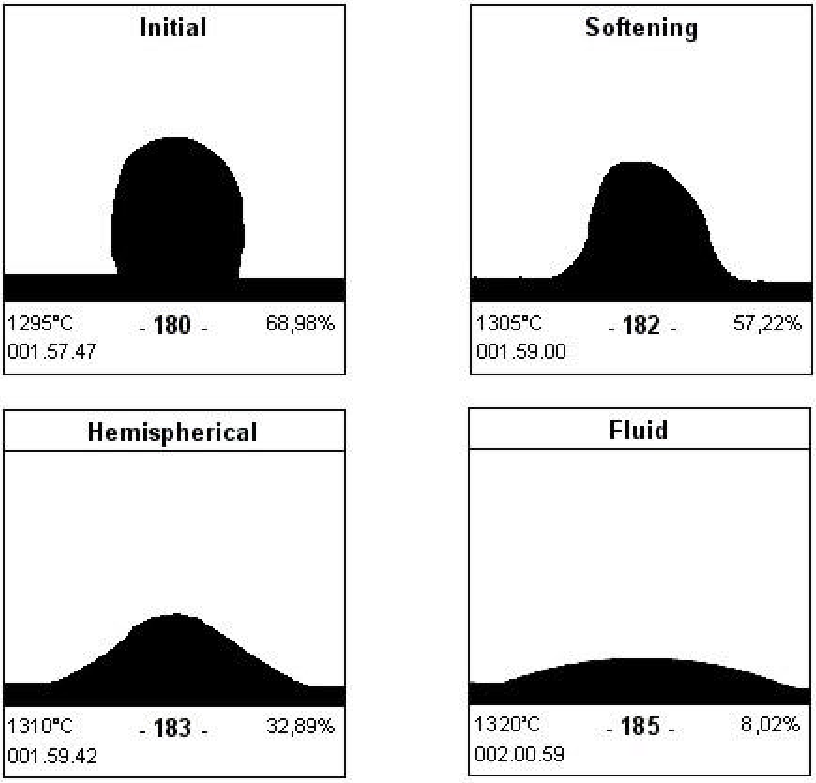


fig.7: Images of the sample “CDR 1”

2) Ashes derived from industrial wastes (biomasses)

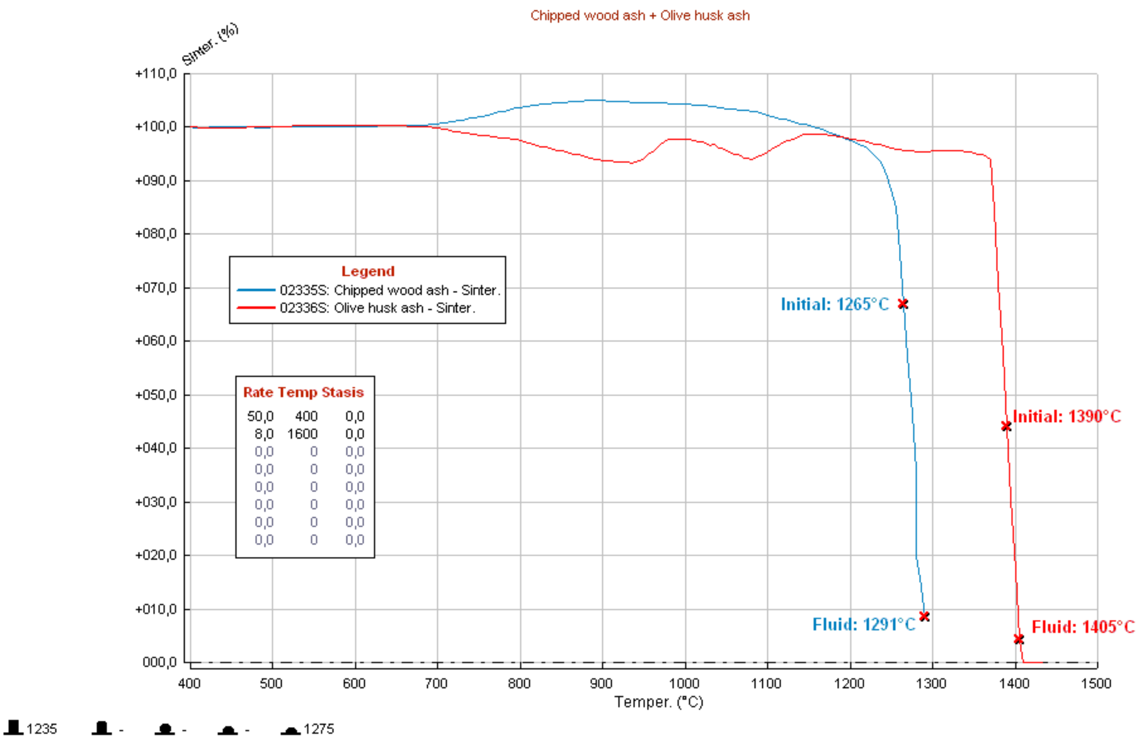


fig. 8: Flattening curves of the ashes of the two biomasses

In both cases, the instrument has identified only the initial and fluid temperatures, because the specimens assume irregular and unusual forms.

3) Ash derived from a conventional fuel (fossil coal)

fig. 9 The specimen before the test

fig. 10: The specimen after the test

The specimen obtained by pressing the ash of fig. 5 is shown in fig. 9. Observing the colour of the sample, it is possible to say that the ash contains iron.
The same specimen at the end of the heating cycle is shown in fig. 10.
In the next graph (fig. 11), the flattening curve is reported.

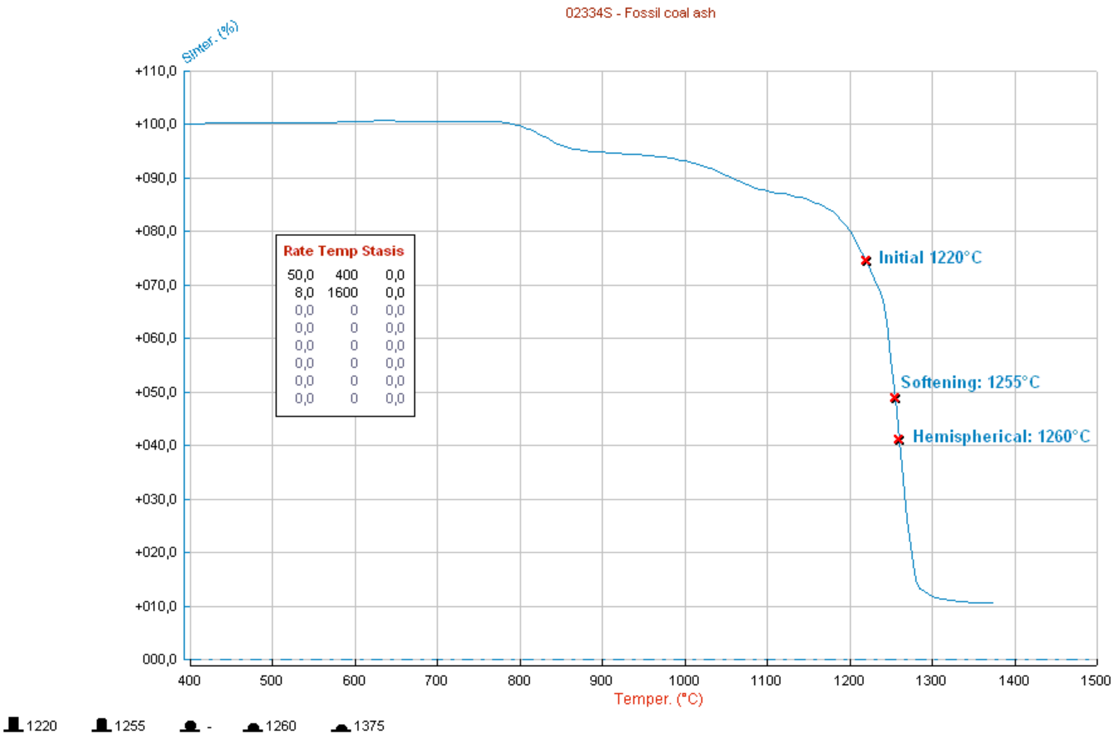


fig. 11: Flattening curve of the fossil coal ash

The sample analysed reaches a complete melting (it is liquid), but it does not respect the definition of the norm ASTM 1857, because its dimensions are stable on a value of 10% with respect to its initial height (instead of 8 % specified by the norm).

Discussion

A limit found using the ASTM norms is that the thermal behaviour of the materials is studied by subjecting them to heating rates too low, far from the real industrial conditions of utilization. For this reason, in some industrial fields, for example in the ceramic sector, these norms were abandoned because not suitable to reproduce the thermal stresses that actually occur in industrial kilns [1].

With the old manual heating microscopes it is not possible to reach heating rates very high. This heating microscope of new generation, instead, allows to achieve heating rates of 80°C/min for the model which can reach a maximum temperature of 1600°C and 50°C/min for the model which can reach a maximum temperature of 1750°C.

The heating microscope MISURA HSM is also able to work applying an instantaneous heating. The kiln is always kept hot at the desired temperature and then it slides over the sample in few seconds thanks to a motorized slide.

In order to study the effect of a very high heating rate, we carried out a comparison between the behaviours of the three samples of ash deriving from RDF subjected to three different heating cycles.

The results are reported in the following graphs (figures 12, 13 e 14). The curve obtained previously applying the ASTM cycle is represented in blue, the one obtained with a heating rate of 80°C/min in red and the curve obtained with an instantaneous heating in green.

1) RDF 1

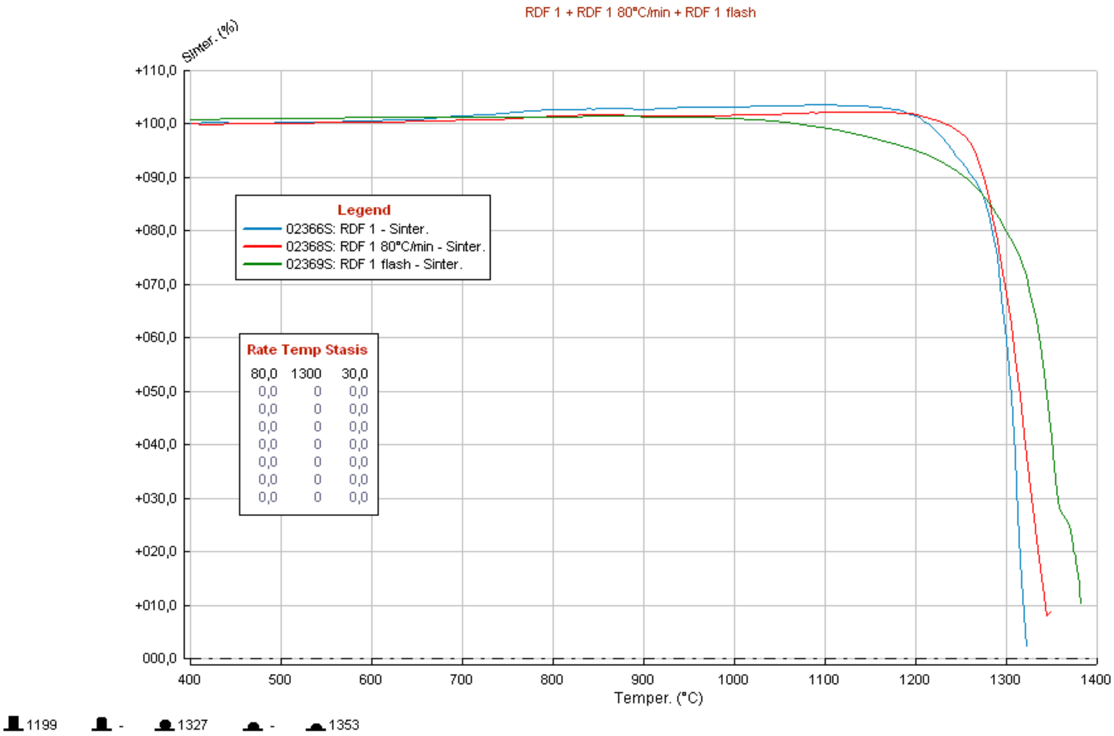


fig. 12: Flattening curves of RDF 1 ash, subjected to three different heating cycles

2) RDF 2

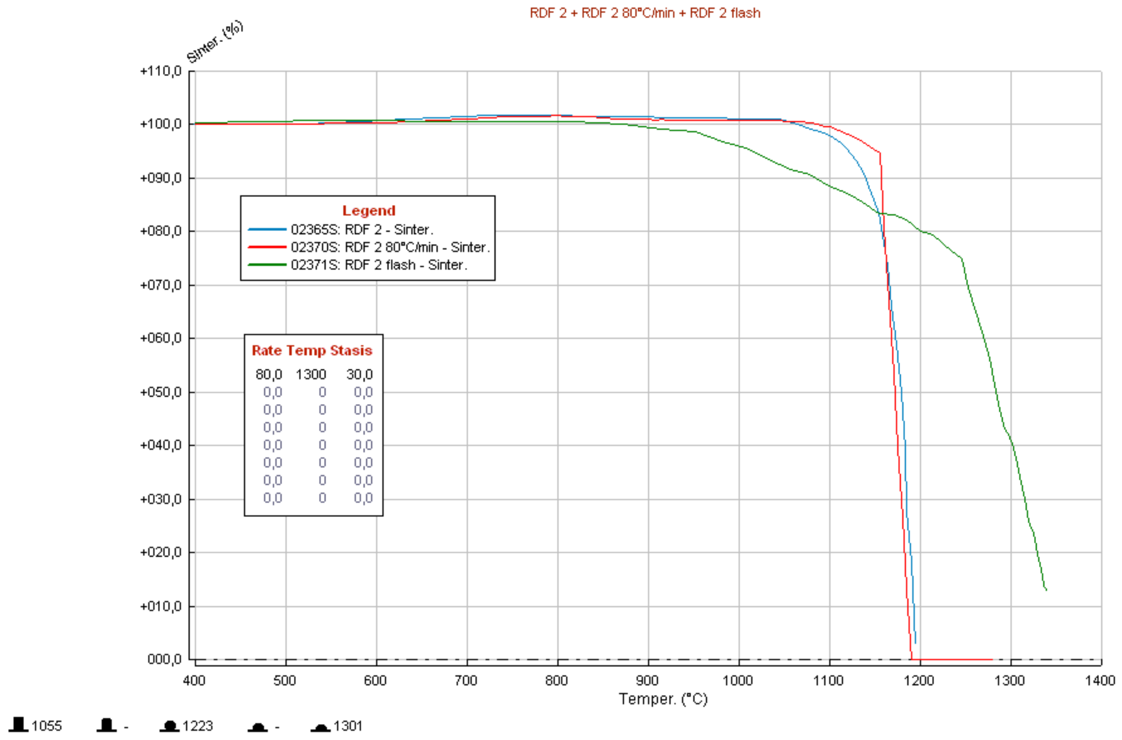


fig. 13: Flattening curves of RDF 2 ash, subjected to three different heating cycles

3) RDF 3

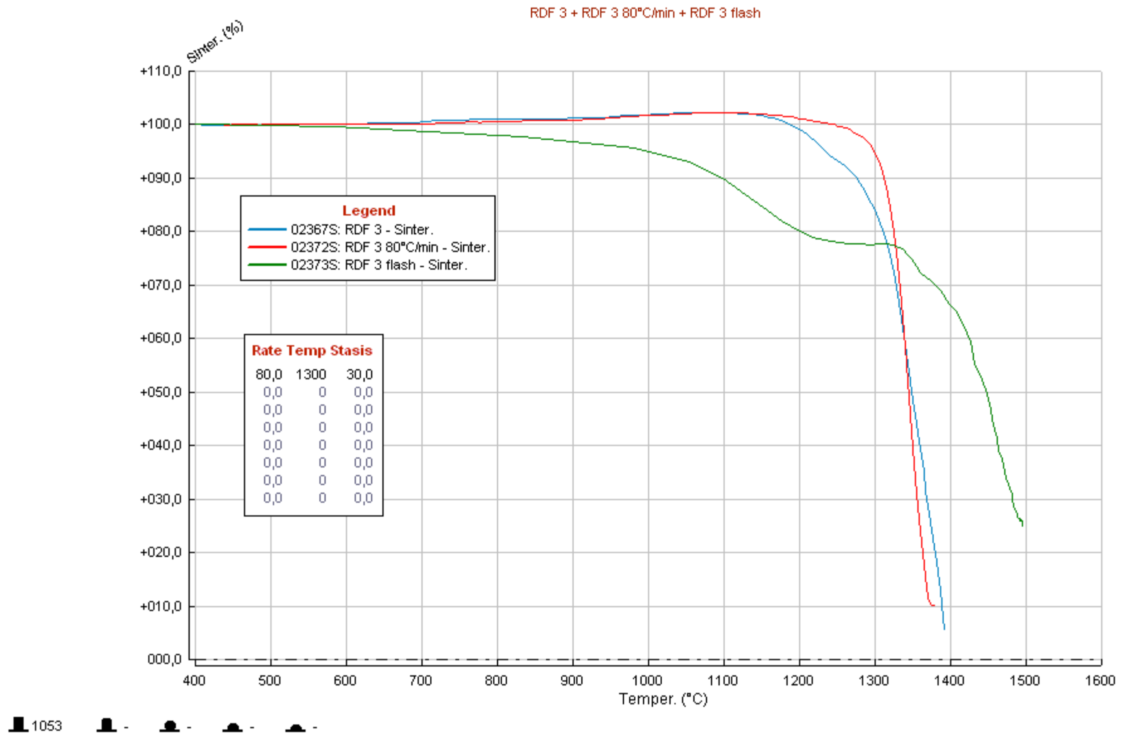


fig. 14: Flattening curves of RDF 3 ash, subjected to three different heating cycles

As it is possible to see, the application of the two heating cycles with heating speeds of 8 and 80°C/min does not cause big differences in the thermal behaviour of the samples.
An advantage found in the case of the quicker heating is obviously the reduction of the time of test execution (only 20 minutes). Using the cycle of the ASTM norms, the execution takes 130 minutes.
If we consider the instantaneous heating, the differences are more evident. In this case, all the RDF ashes start to shrink earlier, but reach higher fluid temperatures.
In the cases of heating rate of 80°C/min and instantaneous heating, the behaviour of the ashes will be more similar to the their real behaviour during a combustion process.
In the following graph (fig. 15), the three different heating cycles applied are represented: the curves are expressed as a function of the time.

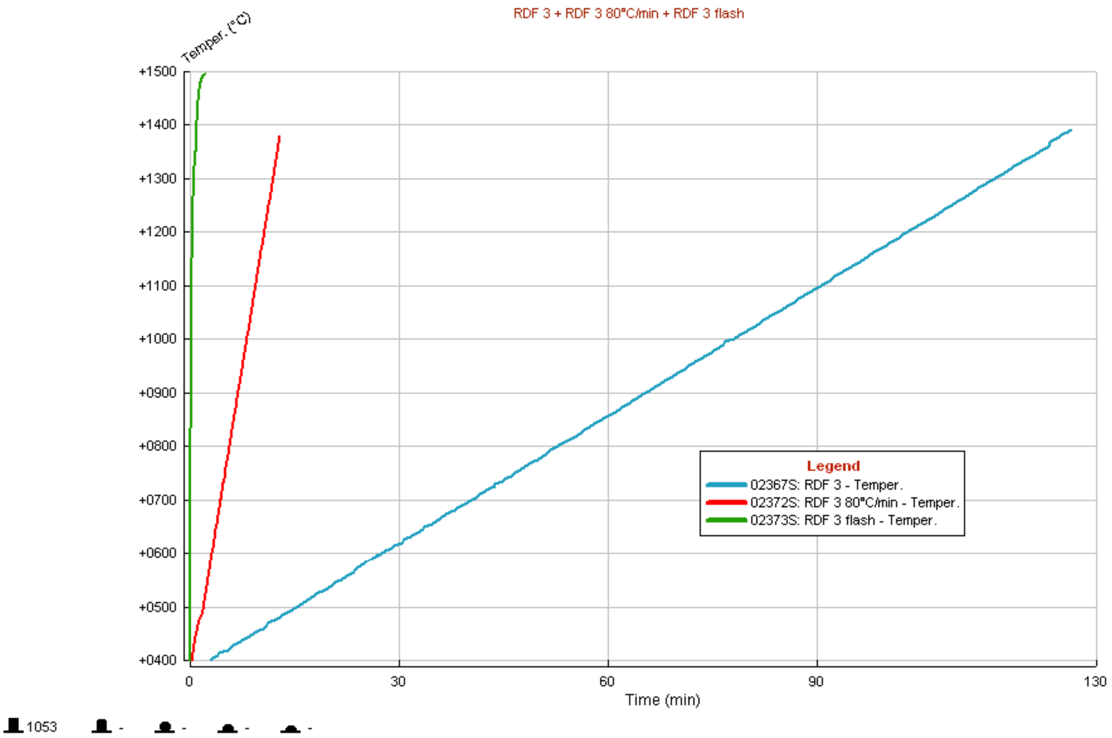


fig. 15: Curves of the three heating cycles applied to the samples

The heating microscope MISURA HSM permits also to print all the images stored during a test (in our case, one image each 5°C), or to observe them in rapid sequence to give an animation effect (it can create an AVI file).

Conclusions

The automatic analysis of the images provided by the heating microscope allows to obtain accurate information on materials subjected to a heating cycle [1].
The characteristic temperatures, in fact, are very important parameters for characterizing fuel ashes. Their behaviour influences the choice a fuel.

In particular, it is known that the working temperature of a boiler must be kept lower than the melting temperature of the ashes, in order to avoid the formation of glassy residues and deposits on the walls and on the top end.
The use of fuels which originate low-melting ashes imposes lower temperatures of work and thus a lower efficiency of the boiler.
A more complete information is provided observing the general behaviour of the flattening curves, which represent the dimensional variations of the material as a function of the temperature.
The possibility of new generation microscopes to reach high heating speeds permits to reproduce the actual thermal stresses industrially present.
Finally, it is important to underline that the heating microscope MISURA HSM was the first able to reach heating rates so high and it is still the only one which can apply the instantaneous heating.

Acknowledgements

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Bibliography

[1] Studying frits with the heating microscope (studio delle fritte col microscopio riscaldante) M. Paganelli - CWR (Ceramic World Review) n° 24 1997 Pag 48.