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Material screening - MCC testing report and analysis

Projektadresse

Kunde: Burnblock

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Udarbejdet af: Konrad Wilkens

Kontrolleret af: Dan Lauridsen

DBI – Dansk Brand- og sikringsteknisk Institut

Jernholmen 12, 2650 Hvidovre · Tlf.: 36 34 90 00 · Fax: 36 34 90 01 · E-mail: dbi@dbi-net.dk · www.dbi-net.dk

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1. Background information

The purpose of this report is to provide a summary of the test outcomes and analysis results performed on the material sample(s) supplied by the client.

The aim of this work is to assist the client by providing information on the material samples combustion behaviour and potentially aid in accelerating their product development. This was undertaken by performing materials tests using the Micro Combustion Calorimeter (MCC). The outcome of these tests were then numerically analysed in order to obtain a range of metrics by which the materials can be compared.

1.1. The test apparatus

The Micro Combustion Calorimeter - Pyrolysis-Combustion Flow Calorimetry (PCFC)

Experiments performed in this report were undertaken using the Micro-scale combustion calorimeter (MCC), developed by the US Federal Aviation Administration (FAA)[1]. The MCC was principally developed as a screening tool for testing newly synthesised materials in the early stages of a material/product development phase.

The MCC employs PCFC, which principally works by separating out the two main processes in flaming combustion; solid-phase pyrolysis of the specimen and the gas-phase combustion of the pyrolysis gases. This is accomplished by using a two-stage rector, where a specimen is pyrolysed in one chamber under controlled atmospheric and heating conditions, and the evolved pyrolysis gases then travel into a second furnace chamber, which mix with oxygen and are then combusted at high temperatures, as shown in Figure 1.

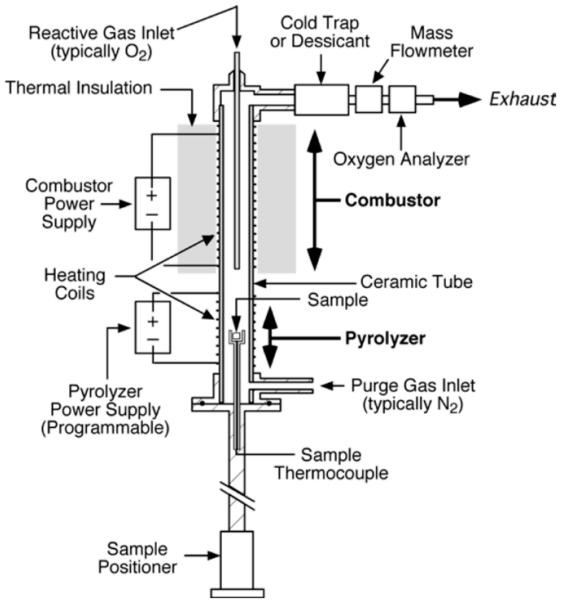

Figure 1 - Schematic of the MCC

The MCC measures the rate of oxygen consumed by the conversion of pyrolysis gases to heat through the combustion process. This can then be transformed into a heat release rate (HRR) through the principles of oxygen consumption calorimetry[2]. Fuel samples tested in the MCC are typically in the order of milligrams.

1.2. Materials

The materials for this project were supplied by the client: Burnblock.

Table 1 - materials summary

Sample ID	Sample name
NFR	Non-fire retarded wood sample
FR	Fire retarded wood sample

Test inputs

Three different inputs variables may affect the outcomes of the MCC testing:

1) Sample preparation

   Even though the sample sizes are very small (2-4mg), and the assumptions in the test neglect the effects of heat transfer though the sample. How the sample is prepared for testing has been shown to affect the outcomes of the test[3]. The nature of the samples provided by the client was inherently inhomogeneous and this can increase the level of uncertainty in the outcomes.

   A sample mass of approximately 3±1mg was chosen for this testing, accuracy on the weight of sample is not a critical factor however as all test outputs are normalised by the sample mass.

2) Heating rate

   The heating rate can affect the outputs from the MCC, to high and previous research has shown there are effects of thermal lag within the sample. Too low, and the noise to signal ratio may affect the results. A heating rate of 1°C/s was chosen to minimise the above effects based on guidance in previous literature and research by the author [3–5].

3) Pyrolysis chamber atmosphere

   Tests are performed in both nitrogen and oxygenated atmospheres. The reasoning for the two different atmospheres is two-fold: firstly, it is a common assumption that during flaming combustion, underneath the flame where the material is being heated and pyrolysing there is no oxygen available for solid-phase oxidative reactions to occur. Thus, in order to best replicate this condition, samples are generally tested in an inert, unreactive environment to prevent these additional reactions occurring. However in real fire cases this may not always be the case, hence secondly, it is prudent to also investigate the sample material within the oxidative case scenario. As in an oxygenated atmosphere, additional reactions in which the char or residue leftover from the first thermal decomposition phases under goes an oxidative reaction process, which adds to the total energy released by the material sample. In an oxygenated environment, the MCC is considered comparable to a Bomb calorimetry test, and gives the maximum potential energy that could be released by the sample material. This is commonly known as the heat of combustion or the calorific value of the material, and is measured in J/g or kJ/g.

1.3. Material fire behaviour metrics

It has been shown that the thermal combustion properties obtained from MCC are independent of test conditions, accurate, and highly reproducible. In contrast, fire and flame tests (flammability) are highly dependent on test conditions; so standardised procedures are required to obtain reproducible and repeatable results. Consequently, the results of fire and flame tests, which may be pass/fail outcomes or fire response parameters, are not material properties. Fire and flame test results are subject to extrinsic factors, such as thickness and edge effects, as well as physical and dimensional changes during burning, like swelling, melting, and dripping. Moreover, fire and flame test results are influenced by condensed/gas phase processes, such as barrier formation/flame inhibition that may be intrinsic to the material or caused by flame retardant additives under conditions of incomplete burning/combustion. The strong dependence of fire and flame test results on test conditions suggests, at best, a qualitative relationship to MCC thermal combustion properties[6].

That said, the MCC and the resultant analysis has shown favourable outcomes when using it as a screening or comparative tool, where samples can be compared to each other and ranked in terms of fire performance.

There are numerous metrics that may be obtained from the MCC testing[4], and all of them give some information about the materials thermal combustion behaviour. The main metrics used in this project are briefly summarised below:

- ***Peak heat release rate*** **[Q_p]**
  (units: Watts per gram, heat released normalised by the original sample mass)

  The peak heat release rate (PHRR) is simply the value(s) at which the heat release rate (in the units W/g), as measured in the MCC, reaches its peak(s) during the test period. This parameter is important as it gives a measure of the maximum heat that may be released by the sample.

- ***Temperature at*** **Q_p [T_p]**
  (units: degrees in Celsius)

  Based on the selected heating rate (°C/s), and the measurement of the heat release rate, the temperature at which the PHRR occurs can be determined. This gives information on when reactions within the material take place with regards to temperature within the sample.

- ***Specific heat release of the sample*** **[Δh_c]**
  The net heat of complete combustion of the volatiles liberated during controlled thermal decomposition per unit initial specimen mass.

- ***Net calorific value of the sample*** **[$\Delta h_{c,0}$]**
  The net heat of complete combustion of the specimen measured during controlled thermal oxidative decomposition per unit initial specimen mass.

- ***Specific heat of combustion of specimen gases*** **[$\Delta h_{c,gas}$]**
  Net calorific value of gases, calculated via the specific heat of combustion of the sample/ (1-*Y*)

- ***Char Yield*** **[Y]**
  Measured by weighing the sample cup after the test has been performed. It is the unburned residue remaining after the test. Higher char yields in oxygenated atmospheres indicate preferable behaviour in the event of a fire.

2. Results and Analysis

2.1. MCC results

MCC tests were performed based on guidance provided in ASTM D7309 - Standard Test Method for Determining Flammability Characteristics of Plastics and Other Solid Materials Using Microscale Combustion Calorimetry[4].

Tests were performed and the raw data was then analysed to investigate the thermal combustion behaviour of the set of materials provided by the client. The tests were performed for development purposes, not for certification.

2.1.1.Normalised HRR vs. Temperature

After the tests were performed, the raw data was collected and then treated in order to examine the transient heat release rate (normalised by the samples mass = *W/g*) against the heating rate profile of the sample. This was completed so that the exothermic reactions that occur within the material as it is heated may be investigated with regards to the temperature of the sample. The decision of choosing to view the results vs. temperature and not time was made as it facilitates further understanding of how the material reacts under thermal attack rather than an arbitrary time period. The figures below illustrate the outcomes of this analysis.

The figures below are separated into two different groups; Nitrogen atmosphere tests and Oxygenated (or air) atmosphere tests. The explanation as to why tests are performed in both nitrogen and oxidative atmospheres was discussed previously in Section 1.3.

2.1.1.1. Nitrogen atmosphere tests

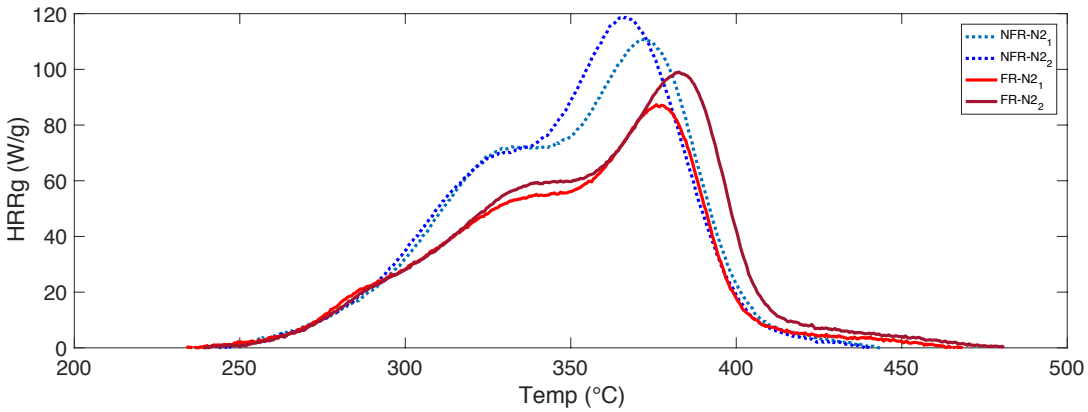

Figure 2 – All Nitrogen atmosphere tests for materials

2.1.1.2. Oxygenated atmosphere tests

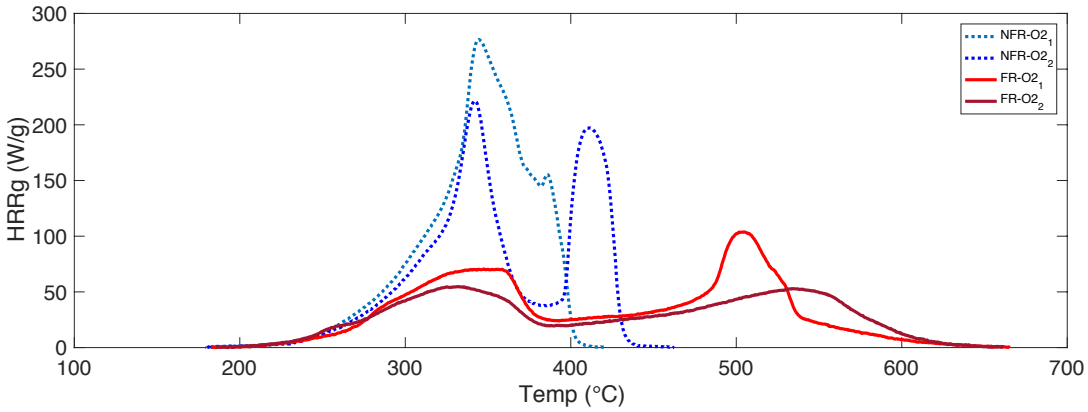

Figure 3 – All Oxygen atmosphere tests for materials

2.1.2. Additional material metrics

Table 2 – quantitative materials metrics summary

Sample ID	Q_p (W/g)		T_p (°C)		Δh_c (kJ/g)	Δh_{c0} (kJ/g)	$\Delta h_{c,gas}$ (kJ/g)	%Y (N2)	%Y (O2)
N2	Peak1	Peak2	Peak1	Peak2					
NFR-N2	-	114.8	-	369	8.25	-	9.73	15.23	-
FR-N2	-	93.185	-	379.3	7.01	-	9.07	22.71	-
O2									
NFR-O2	249	176.4	343.05	398.7	-	15.77	-	-	0
FR-O2	62.715	78.36	339.65	519.35	-	13.86	-	-	0

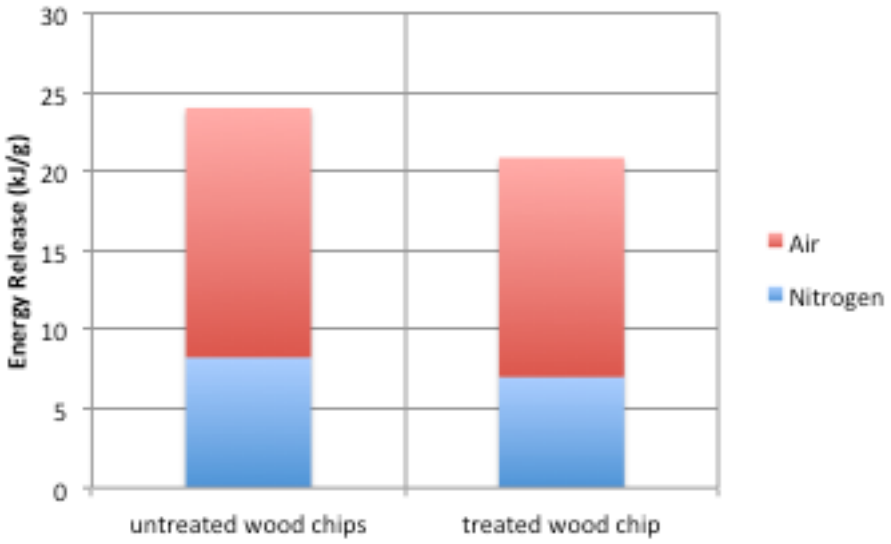

Figure 4 – visualisation of total energy release by samples in both N2 and O2 atmospheres

3. Comparisons and Summary

The aim of this work was to compare treated wood waste against non-treated wood waste, to examine how the combustion of these products compares in idealised combustion conditions. A brief list of the findings is provided below, to summarise the results.

- Overall the treated wood waste has a lower energy content when compared to the untreated samples, the reduction in energy content is likely due to the addition of incombustible mass to the treated samples. However the reduction is not significant as to nullify the aims of this project.
- The treated wood waste released its energy at higher temperatures when compared to the untreated samples, which is a point to consider with regards to the potential application of this product for wood pellet combustion, however the temperatures required are still within those expected in this use case.
- The release of energy from the treated wood waste extended when compared to the untreated samples. This suggests that the treated wood pellets would indeed have a longer burn time when compared to the untreated wood pellets (which was the idea behind the initial project from burnblock). However it should be noted that this release while longer, is lower in energy, thus what could be expected is a slower lower heat output from such treated pellets when compared to untreated pellets.
- Tests performed in this report were performed in ideal combustion conditions, where combustion of volatiles is forced through to completion. In real-world scenarios this is rarely the case, and the efficiency of the combustion is likely to be reduced which may also affect the results of the pellet burning behaviour. Hence it is the suggestion of this report that additional tests be performed at a larger scale and in more 'real-world' conditions to get a full picture of the behaviour of the treated wood as a pellet, burned in less efficient conditions (suggestion of cone calorimeter tests of prototype pellets is suggested as the next step in this process).

4. References

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