Fuel 251 (2019) 779–788

Contents lists available at ScienceDirect

Fuel

journal homepage: www.elsevier.com/locate/fuel

Full Length Article

The impact of ash pellet characteristics and pellet processing parameters on ash fusion behaviour

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ARTICLE INFO

Keywords:
Coal
Biomass
Advanced ash fusion test
Ash
Pellet preparation
Initial deformation temperature

ABSTRACT

The Ash Fusion Test (AFT) is considered to be the most popular method of characterising the melt characteristics of solid fuel ash. This study shows how pellet preparation can make significant improvements to repeatability. Pelleting pressure, pellet particle size, pellet shape, and furnace ramp rate were investigated to establish the most repeatable representation of ash melting relevant to pulverised fuel combustion in a furnace in an oxidizing atmosphere up to 1600 °C. A 5 mm machine pressed pellet was found to produce the best results as it identified the earliest initial deformation temperature (IDT), gave the least error, and displayed the greatest visible change in pellet height to enable easy identification. Reducing maximum ash particle size to < 72 µm and increasing the pressure of the pelleting process was also shown to produce a 120 °C reduction in the IDT when compared with other methods. Reducing the ashing temperature and retaining volatiles lost during high temperature ashing were shown to have a negligible impact on IDT. The characteristic AFT curve was also used to quantify the extent of shrinkage and swelling during the test.

1. Introduction

Slagging and fouling are key issues for the power industry and cause significant problems with continuous, long term boiler operation [1,2]. Whilst there are various methods for the prediction of slagging and fouling such as dilatometry/shrinkage tests [3–5], sinter strength tests [6,7], viscosity measurements [8,9], and a range of empirical indices [8,10–13], the AFT is one of the most popular methods [7,9,11,14,15]. The AFT was developed in the early days of the power industry to predict clinker (large lumps of ash) forming characteristics in stoker furnaces [16]. It continues to be used, with minimal changes to the methodology since its inception in the early 1900s [16]. The current method of preparing samples for the AFT relies on hand-made cones or cylinders of ash, combined with a binder, and sometimes with the use of a hand press. The use of hand pressed cones and can lead to poor repeatability [4,16,17], as well as uncertainty and subjectivity to the results. Attempts to make the test more analytical have been achieved by means of Thermo-mechanical Analysis [4,18–21]. Further to the empirical tests, multiple predictive methods are documented [9,13,14,22–25]. A more recent review correlates sticking probability to ash fusion temperatures alongside other modelling techniques [26].

During AFT, four discrete temperatures are noted; shrinkage temperature (biomass only), initial deformation temperature (IDT), softening temperature (coal only), hemisphere temperature, and flow temperature (FT) based on shapes given by the regional standards [27–30]. These temperatures provide a prediction of slagging and fouling onset. The key temperatures utilised in power stations are IDT and FT. These temperatures are used to predict melting phenomena of a fuel ash in the boiler. In today's more sophisticated boilers, the test is used to inform fuel selection, as a method to identify the potential for unexpected slagging phenomena. Clearly this knowledge can be used by generators who use mixed fuels from a range of locations to create a blend with controlled slagging/fouling characteristics [31]. The repeatability of the test has been questioned with differences of up to 400 °C in IDT having been documented [4]. This uncertainty in IDT results makes it hard to make informed decisions based on melting temperature. Poorly identified melting characteristics of fuel ash could lead to premature outage or sourcing of unsuitable fuels. Thus, generators require a more reliable process with a clear illustration of behaviour.

Multiple efforts have been made to improve the ash fusion test [3,4,21,32,33], none of which have been adopted by industry. Questions surround the stability and consistency of the cone structure, and whether the toppling of the top of the cone is truly the first deformation of the pellet. If any of these criteria are not satisfied by the current test method, then the cone pellet may not be the optimal shape to represent

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https://doi.org/10.1016/j.fuel.2019.03.142
Received 5 January 2019; Received in revised form 20 March 2019; Accepted 27 March 2019
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Nomenclature

AAFT	Advanced ash fusion test
AFT	Ash fusion test
CHP	Carbolite hand press
CHPD	Carbolite hand press with dextrin
FT	Flow temperature
IDT	Initial deformation temperature
MPPs	Machine pressed pellets
OAAFT	Optimised advanced ash fusion test
PAnG	Picture Analysis and Graphing
TGA	Thermogravimetric analysis
ΔH	Change in height
(Av(SD (H/H_i)))	Average standard deviation of height/initial height
(ΣΔH)	Sum of change in height

trigger points for deformation.

Ashing temperature (where the fuel is combusted to provide the ash for the AFT) is defined by standardised procedures [34,35], it has since been shown that ashing temperature plays a significant role in the AFT [36–38]. Image analysis techniques have proven useful in aiding flame characterisation [39–41] and coal petrography [42–44]. Pang *et al.* [17] adopted the basis of the AFT but used an automated image analysis method to plot a curve of relative height of the pellet against temperature. This study focused on providing a characteristic curve for biomass ash, however, the workers did not examine the effect of different pelleting methods on the AFT results.

The current coal standard cone theorises that the IDT is identified when the top point of the cone (considered to be a single point) melts [17]. The biomass standard uses cylinder which has 2 corners which enables 2 points to be averaged. However, the significance of this point of initial deformation is contentious in literature, as melting phases can be identified at earlier points in the test without a change in sample geometry [26,45]. Thus, gaining more insight into the melting processes, as well as developing a more repeatable and reliable process are of paramount importance.

In this study, a range of pelleting processes have been investigated to identify the most repeatable and representative method of identifying the IDT. A progressive investigation was conducted to optimise each aspect of the process, with the aim of producing a robust and highly repeatable ash fusion test. Additionally, a modified version of the PAnG AAFT was used to provide novel analysis of the ashes and develop AFT characterisation curves which might provide an insight into the ash fusion behaviour of solid fuels. This study aims to address the issues with repeatability by presenting a fully mechanised pellet production process and automated AFT process using a novel image analysis technique. Furthermore, for the first time, a wide range of pelleting methods will be directly compared, and an optimal preparation methodology has been identified.

2. Materials and methods

Kellingley is a typical UK coal and was selected as the 'standard' for the optimisation of the test as it is well known throughout the industry and has been widely used in UK coal fired power stations. A further 4 fuels were selected to verify the findings of the optimised method; two international coals including a Colombian (La Loma) coal, and a South African (Zondag) coal; two white wood biomass, denoted B117 and EC78.

2.1. Milling and ashing

The coal was milled to below 212 μm in accordance with the ASTM standard [46], and the biomass was milled to below 1 mm to represent the size specification for biomass fired boilers [47]. The coals were then milled down using a Retsch Jaw crusher BB 100 mill from 10 cm down to 2–10 mm. The samples were then milled sequentially using 3 screens of the Retsch SM2000 Knife mill to obtain a particle size of 70% below 325 μm (4 mm screen: 5–10 mm to 70% below 2 mm, 1.5 mm screen: to 70% below 750 μm, and 750 μm screen: to 70% below 325 μm). A TEMA machinery disc mill was then used to achieve a particle size of 100% below 212 μm. The biomass was milled below 1 mm using the Retsch SM2000 knife mill with 4 mm and 1.5 mm screens in series. This generated a particle size of 70% below 750 μm.

Table 1
Pelleting shape descriptions.

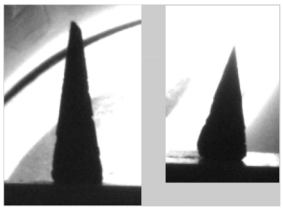
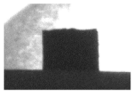
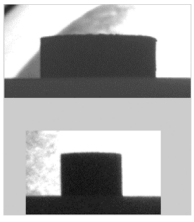
Shape/standard	Description	Diagram
COAL: CONE MOULD WITH DEXTRIN	The two cones shall be 19 mm [3/4″] and 13 mm [1/2″] in height and 6.4 mm [1/4″] in width	
BIOMASS: CHP	This method involves making a, roughly, 5 mm high × 5 mm diameter pellet, with a handheld press	
MPPs	10 mm and 5 mm dies are used to make pellets under 5000 psi (34.47 Mpa) of pressure (2707 N and 677 N, respectively) and held for 1 min. Pressing profiles shown in Fig. 1	

Table 2
Sample preparation of the high pressure milled and low pressure un-milled scenarios.

Sample	Ashing Temperature	Milling	Pelleting Pressure
High pressure milled	575	Milled < 75 μm	7500 psi
Low pressure un-milled	575	Un-milled	2500 psi
High pressure milled	815	Milled < 75 μm	7500 psi
Low pressure un-milled	815	Un-milled	2500 psi

Coal samples were ashed at 815 °C ± 15 [34] and biomass at 575 °C ± 15 as required by the standards [48]. Large alumina rectangular crucibles (SS200S: L204 mm X W105 mm X H25 mm) from Almath Crucible LTD were used to maximise ash product. The ashing took place under a continuous air flow condition in a Carbolite GSM Ashing Furnace. Coal was raised to 500 °C and held for 1 h, then raised to 815 °C and maintained for 3 h. Biomass was raised to 575 °C and held there for 3 h. After ashing, the ash was sieved to pass 75 μm sieve [29,30], and a TEMA disc mill was used to ensure that any leftover particles passed through the sieve.

For the voidage study and the milled and un-milled study, 72 μm and 250 μm sieve screens were used to represent the milled ash based on the standards and non-milled ash, respectively. The ash was stored in sealed glass vials (until use) to minimise ash moisture uptake.

2.2. Pelleting method optimisation study

Six pelleting methodologies were used in the Pelleting Method Optimisation study; the 13 mm and 19 mm cone presses (dimensions given in Table 1) were made according to the standards [30] using dextrin (Acros Organics) and cone moulds supplied by Carbolite Gero with the Carbolite Gero Ash Fusion furnace; 5 mm Carbolite Gero hand press with dextrin (denoted CHPD) and without dextrin (denoted CHP), and a custom cylindrical hand press made by Carbolite Gero for the purposes of the ash fusion test. Finally, 5 mm and 10 mm machine pressed pellets (denoted MPPs) were made using an Instron Universal testing system 3360 Series using varying pressures as detailed in Table 1.

During the voidage investigation the Instron Universal testing system 3360 is used to study additional pressures of 2500 psi and 7500 psi for pelleting the 5 mm pellet. Once pressed, the pellets were stored in a glass vial for protection to minimise moisture uptake.

2.2.1. Cumulative Pelleting, preparation and ashing study

To validate the investigations of the pelleting and preparation studies, a high pressure milled sample and low pressure un-milled matrix was created to test a range of fuels, detailed in Table 2. Included in the study was the addition of two different ashing temperatures to assess the impact. All milled and un-milled samples were pelleted at 2500 and 7500 psi to establish the scenarios.

2.3. Advanced ash fusion test (AAFT)

The AAFT profiles were obtained using the Carbolite Gero Ash Fusibility Test Furnace – CAF G5 (Fig. 2). Samples were placed on Carbolite Gero 25 mm × 25 mm recrystallised alumina ceramic tiles and loaded in the furnace. The furnace temperature was increased from 25 °C to 1600 °C at a rate of 7 °C/min under oxidising condition (air flowrate of 4 l/min at 27.5 kPa (4 psi)). Images were taken every 1 °C on an integrated HD 1.3 Mb camera with an image size of 1280 by 1024 pixels. MATLAB (version R2017b) was used to analyse each image in a process adapted from work by Pang and co-workers [17]. The images are cropped automatically based on the starting location of the sample. The pellet was then automatically tracked during the experiment using a combination of edge detection and thresholding techniques. 5 thresholding techniques were used in total including Canny [49], Otsu [50], Prewitt [51], Bradley Adaptive Thresholding [52], and Log methods [49]. These techniques were chosen based on the different image histograms obtained at different points in the test. Canny and Otsu were both suited to the early test back lit range of images (0–500 °C). Prewitt was used in the transition where the back light turns off and the radiative light begins (500–800 °C). Finally, the Bradley and log methods were better suited to the end test images (800–1600 °C). These techniques were used to create a pellet outline.

This outline was then used to capture the height and centroid parameter of the pellet. The centroid was used to track the location of

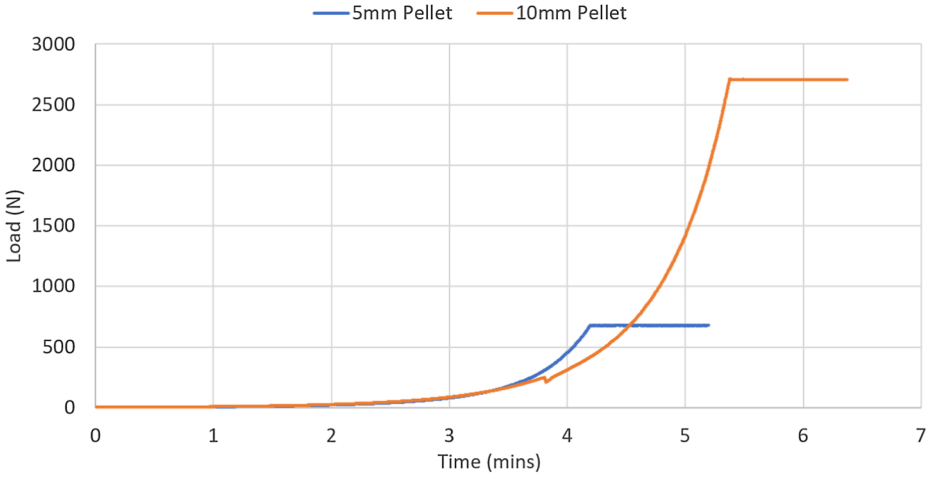


Fig. 1. Profiles for the MPPs tested during the pelleting method optimisation study.

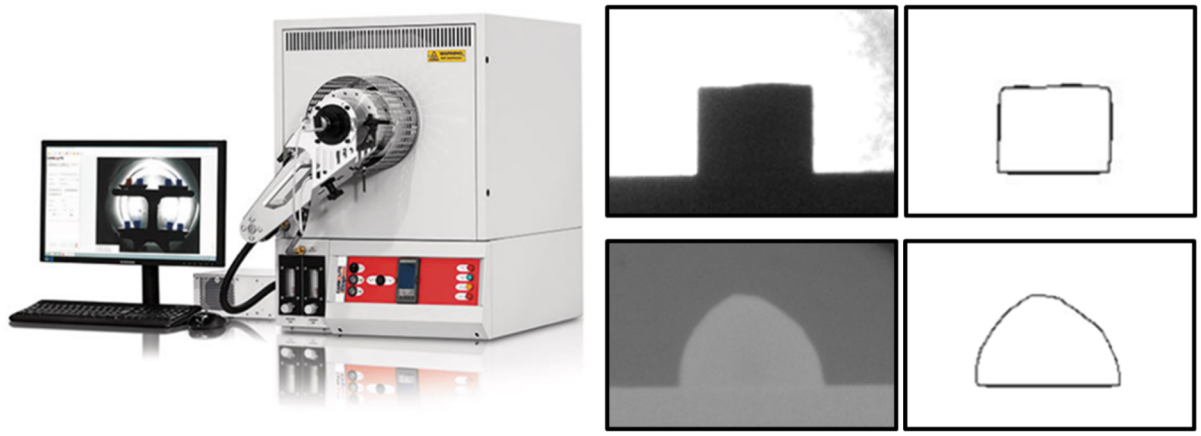


Fig. 2. CAF furnace, images generated, and processed images.

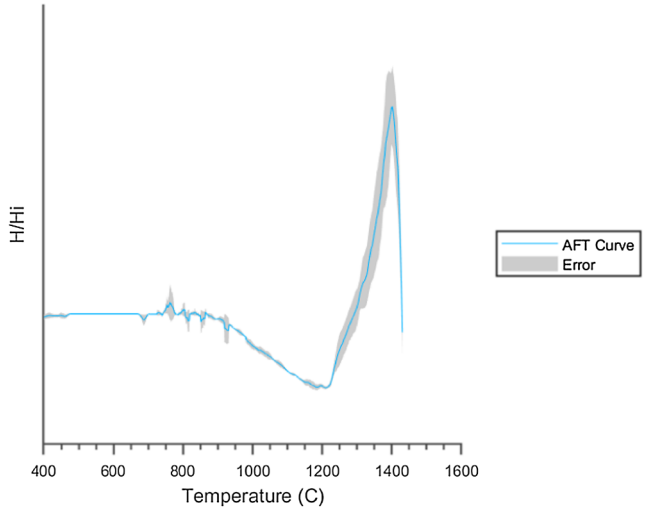


Fig. 3. Typical AAFT profile (250 μm sample from the voidage investigation).

the sample and the height was compared to the initial height of the pellet to plot relative height. Each sample was run in triplicate, with the average displayed on the AAFT plot. The standard deviation of the three runs is displayed on the AAFT profile as a shaded-out area and illustrated in Fig. 3.

2.4. Thermogravimetric analysis (TGA)

Proximate analysis was carried out using TGA. Thermal profiles were produced using TA Instruments Q500 TGA. Tests used 10–15 mg of sample with a particle size range of 75–300 μm. The sample was heated in a furnace at 5 °C/min in 100 ml/min of nitrogen from atmospheric temperature to 900 °C, after which the gas was switched to air at 100 ml/min. The composition of the samples is given by moisture, dry ash free volatile, fixed carbon, and dry ash contents. In addition, the percentage of volatiles at 575 °C, 815 °C and 900 °C was obtained from the profiles [53].

2.5. Pelleting mould type selection criteria

Four criteria were selected to evaluate the optimal pellet mould type for the AAFT. Repeatability, error, morphology and IDT 'trigger' (defined in Table 3) were selected and each were given a weighting based on discussions with industrial end users and the key parameters taken from the AFTT. This is summarised in Table 3; a higher value represents higher importance. The performance of each pellet was ranked from 1 to 6, with 6 being the best.

3. Results and discussions

3.1. Impact of pellet type on the AAFT

In this section Kellingley coal ash has been pelleted using a range of different methods and tested in the Carbolite Gero CAF G5 furnace. Image analysis was used to produce a plot of relative height of the pellet during melting and manual analysis was done to highlight the IDT trigger, which is defined as the first deformation after shrinkage. Due to the evidence that melt phases occur earlier than that identified by the traditional AFT method [26,45], it has been assumed that the earlier the first significant visible melting event occurs, the more accurate the measure of the actual IDT trigger will be.

3.1.1. Repeatability

The first stage of the study explored the repeatability of the different mould processing methods. Fig. 4 displays the AAFT curves for each of the moulds tested. As noted in Table 3, the error is represented by the shaded area between 1100 and 1300 °C, which is the key area of interest. Morphology is represented by the total change in height (ΔH) over the range of 1100–1300 °C. Finally, the IDT trigger point is

Table 3
Selection criteria for evaluating pelleting process.

Criteria	Description	Evaluation	Importance value
Repeatability	How closely the key temperatures of the same sample match	Based on how close IDT readings are for repeat samples	5
Error	How close each plot matches the plot of repeats at the IDT	Average standard deviation (Av(SD (H/H_i))) of the repeat samples between 1100 and 1300 °C	3
Morphology	How much the gradient of the plot changes at the IDT	Based on the sum of change in gradient (ΣΔH) between 1100 and 1300 °C	3
IDT Trigger	How early the plot displays IDT	Based on the average IDT for repeat samples	5

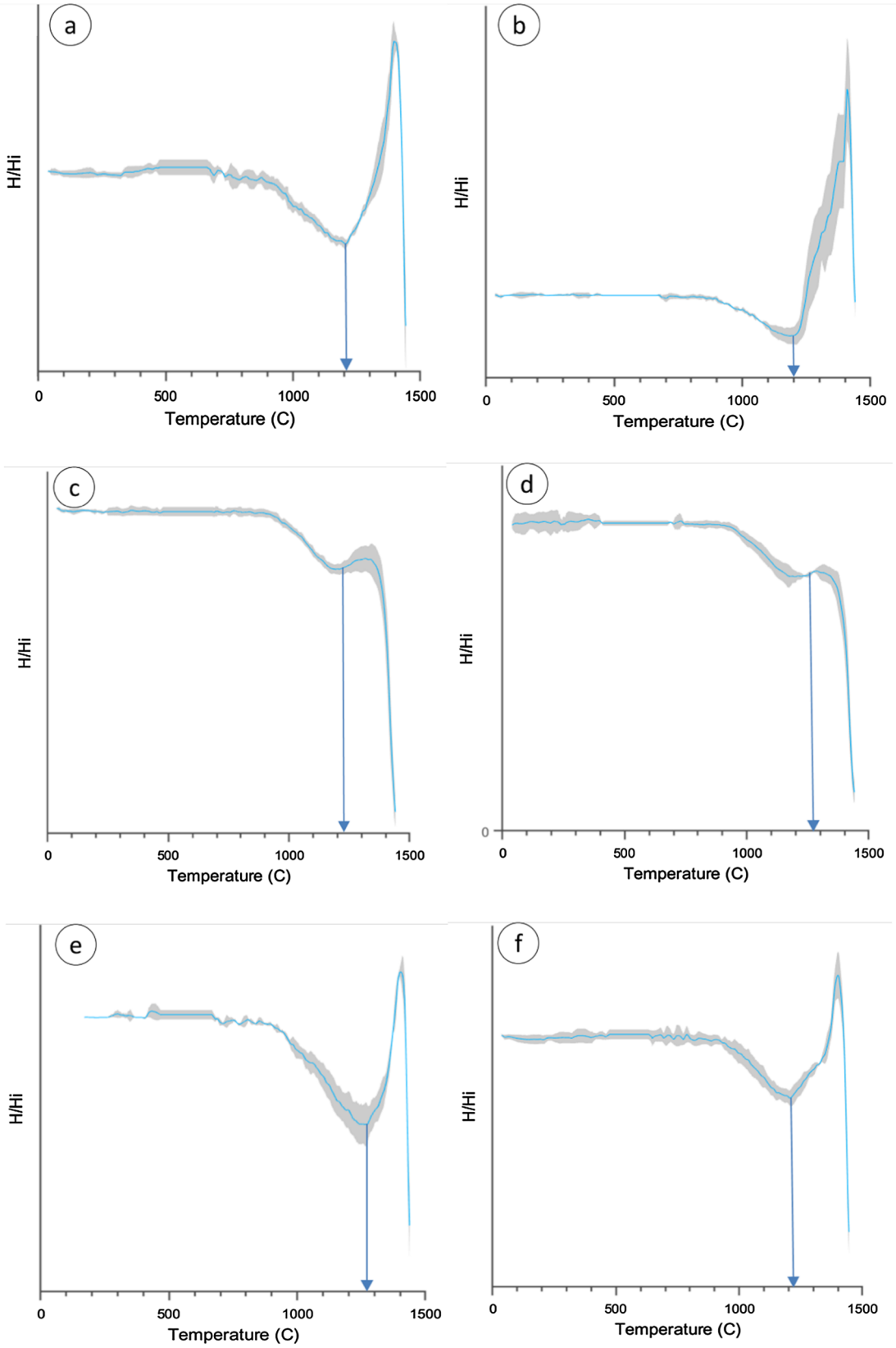


Fig. 4. Plots demonstrating the error, morphology, and IDT trigger of each pelleting method; a) 5 mm MPP, b) 10 mm MPP, c) 13 mm cone, d) 19 mm cone, e) CHP, f) CHPD (n = 3).

Table 4
Selectivity matrix ranks for the pelleting methods in the categories on repeatability, error, morphology, and IDT trigger.

Sample	Repeatability (°C)	Error Av(SD (H/H_i))	Morphology Σ(ΔH)	Trigger IDT (°C)
MPP (5 mm)	< 10	0.0133	0.118	1204
MPP (10 mm)	< 20	0.0803	0.363	1200
Cone (13 mm)	100	0.0179	0.059	1320
Cone (19 mm)	80	0.0135	0.033	1256
CHP (5 mm)	20	0.0368	0.093	1270
CHPD (5 mm)	40	0.0210	0.086	1220

Table 5
Maximum acceptable differences between results [28]

Fusibility of ash	All atmospheres	
	Repeatability (°C)	Reproducibility (°C)
Deformation temperature, DT[a]	30	80

[a] If the sphere temperature is not reached, the precision on deformation temperature might not be achievable.

denoted by the arrow on each curve.

One of the fundamental aspects of the test is how repeatable it is. Table 4 shows the variability of IDT for different mould samples for Kellingley ash. The 5 mm MPP was the most repeatable (< 10 °C), while the 13 mm cone performed the poorest with a repeatability of 100 °C. The cones had significantly more variation in IDT than cylinders, with the MPPs and hand presses showing a repeatability 4 times lower (± 20 °C) than that of cones (± 90 °C). The CHP (20 °C) and CHPD (40 °C) display comparable repeatability to the MPPs with a simpler pelleting method. When compared to the British standards for coal fusibility repeatability guidelines for deformation temperature in Table 5 (30 °C), only the 5 mm and 10 mm MPP and the CHP meet the standards. This investigation would need to be repeated to evaluate reproducibility.

3.1.2. Error

There are no units for error as it is a relative value of height/initial height. The averaged results from the error study for cones (13 and 19 mm) compared to cylinders (CHP and CHPD) would suggest that the cones performed best (0.0157) when compared to the cylinders (0.0379). The 2.5× reduction in error can be attributed to the lack of change in morphology displayed in Fig. 4, which hinders the indication of IDT. Furthermore, Fig. 4 shows that the low error may be a result of coincidental crossing of the plot as opposed to common curve shape. The 5 mm MPP gives the most repeatable curve with an average error of 0.0133. This is attributed to the smaller size of the pellet as well as the consistency of the pelleting process. The 10 mm MPP appears to be an outlier, as it is made using the same process as the 5 mm MPP but produces the highest error of 0.0803. In contrast to the 5 mm pellet, the 10 mm pellets greater size allows more scope for error. The relative height shows a 3× increase whereas the 5 mm pellet show a 2× increase as shown in Fig. 4b. This suggests that minimising the size of the pellet in the analysis will give less error and create a more consistent morphological change.

3.1.3. Morphology

When comparing the morphology, the IDT is best represented by the cylindrical pellets (0.093, 0.086, 0.118, 0.363), as the cones (0.059, 0.033) do not drastically change shape at the key trigger temperature. This is illustrated visibly by the plots in Fig. 4, and by the low ΣΔH in Table 4, which is, on average, 4x greater deformation displayed by the cylinders. A similar change is seen when handmade pellets (0.059, 0.033, 0.093, 0.086) are compared with the MPPs (0.118, 0.363). This suggests that the increase in morphology is linked to the MPPs as opposed to the change in shape. This means that making the pellets at a higher pressure will result in greater change in morphology and easier indication of the IDT. The 10 mm MPP showed the greatest change (0.363), however this was at the expense of increased error as it also displayed the greatest error (0.0803).

3.1.4. IDT trigger

Alongside the repeatability, the IDT trigger is of equal importance. If the IDT trigger can be reduced, earlier melting will be indicated, and thus the allowable limits for IDT may need to be shifted. Table 4 shows that the range in IDTs identified by all the tests was 116 °C. This illustrates the extent of impact that pellet type has on IDT. The lowest IDT achieved was 1200 °C by the 10 mm MPP, closely followed by the 5 mm MPP (1204 °C). Although the CHP (1270 °C) was more repeatable than the CHPD (1220 °C) the dextrin appeared to improve the IDT trigger of the CHP. The clear results in Table 6 originate from the 5 mm MPP. The 5 mm pellet was therefore carried forward to the next stage of testing.

3.2. Impact of material preparation and furnace heating rate for the 5 mm MPPs

Factors such as mineralogy, particle size, water content and applied pressure affect thermal conductivity of a material [54,55]. The size of the ash particles in the pellets, and the pressure with which they are made will determine the physical void fractions and thus the degree of differential temperature and stress within the pellet. The results from the previous study highlighted the 5 mm MPP as the best option for pelleting. This pelleting method will be used to investigate the impact of particle size, pelleting pressure and heating rate on the IDT trigger. Kellingley ash was again used to study 2 particle sizes, 4 pressures and 3 heating rates.

Table 6
Selectivity matrix summary for the mould type analysis.

Criteria (weighting)	5 mm MPP	10 mm MPP	13 mm Cone	19 mm Cone	Hand press	Hand press w/d
Repeatability (5)	6	5	1	3	4	2
Error (3)	6	1	4	5	2	3
Morphology (3)	5	6	1	2	3	4
IDT Trigger (5)	5	6	1	3	2	4
Totals out of 96	93	71	25	51	45	51

Table 7
IDTs of the particle size, pressure and heating rate investigations on Kellingley coal.

Sample	Particle size		Pressure		Heating rate		
	72-μm	212-μm	2500 psi	7500 psi	4 °C/min	7 °C/min	10 °C/min
IDT (°C)	1200	1220	1228	1200	1196	1198	1210

Table 8
Comparison of the change in thermal composition with increasing ashing temperature.

Sample	Moisture Content (%)	Dry Volatiles @575 °C (%)	Dry Volatiles @815 °C (%)	Dry Volatiles @900 °C (%)	Fixed Carbon (%)	Dry Ash (%)
Kellingley	2.7	15.65	27.64	28.2	47.7	24.1
La Loma	3.6	19.7	30.3	30.2	51.3	19.2
Zondag	4.9	14.3	27.0	27.6	63.2	9.1
B117	6.5	80.3	83.9	84.7	13.8	1.4
EC78	6.4	79.9	82.9	83.6	14.3	2.1

3.2.1. Particle size: Milled (< 72 μm) and Un-milled (< 212 μm)

To examine the impact of particle size, two extremes were tested. A particle size of < 72 μm, (according to the ash fusibility standards [29,30]), and < 212 μm (the standard for coal milling prior to pf combustion [46]) were used. This has been designed to investigate how an additional ash milling stage impacts on the results. Table 7 shows that the < 72 μm (No. 400) particle size sample gave an IDT of 1200 °C which matches the outcome of the 5 mm MPP from the previous mould study. The < 212 μm (No. 60) resulted in an IDT of 1220 °C, a difference of 20 °C. This difference in IDT is attributed to the increase in particle size and thus increase in voidage in the pellet which reduces the heat transfer through the pellet.

3.2.2. Pelleting Pressure: Low pressure 2500 psi and high pressure 7500 psi

The next investigation studied the impact of increased pressure during the pelleting process displayed in Table 7. As before, two extremes were used, 2500 psi which was at the low end of pressure and produced an IDT of 1228 °C, and 7500 psi and gave 1200 °C. Increasing the pressure during the pelleting process decreases the IDT of a sample by 28 °C, this is comparable to the difference from the MPPs and the standard hand press with dextrin. The difference is that the low pressure MPP still had excellent repeatability, but with a delayed response to IDT. The delay in IDT can be attributed to increased voidage in the pellet. Whereas non-uniform particle size will increase voidage, the increase in pressure applied to the pellet will reduce the voidage in the pellet and increase particle to particle contact improving heat transfer.

3.2.3. Heating rate: Fast (10 °C/min), medium (7 °C/min), and slow (4 °C/min)

The heating rate was varied to investigate the sensitivity of the key melting phenomena and the characteristic profile. Three heating rates were used, 4 °C/min, 7 °C/min, and 10 °C/min as displayed in Table 7, the resultant IDTs for each were 1196 °C, 1198 °C, and 1210 °C, respectively. The slower the heating rate, the lower the measured IDT. This marginal decrease in IDT can be attributed to slower heating rate allowing the pellet more time to heat up and reach the specified temperature.

3.3. Cumulative influence of pellet preparation and ashing on five different fuels

This study has already shown that the temperature of the first melting phenomenon can be reduced by using machine made pellets and ensuring that the ash being used in the pellets has been pre-milled. The maximum IDT range were 28 °C and 20 °C based on pelleting pressure and particle size, respectively. The following section investigates whether these effects are cumulative. Furthermore, it is still speculated as to whether ashing temperature has a significant impact on the AFT [36,38,56,57]. Four additional fuels were selected to examine the impact of pelleting method, preparation and ashing temperature.

Initially proximate analysis was carried out on the five fuels to identify differences in volatile content at different points in the ashing process. This was broken down into moisture in the coal, dry ash %, fixed carbon %, and the amount of volatiles released at the low ashing temperature, 575 °C, the high ashing temperature, 815 °C, and the total volatiles released at the end of the test, 900 °C. The fixed carbon, dry ash, and volatiles at 900 °C sum to 100%.

Table 8 shows that as temperature increases, the coal samples observed the greatest change in thermal composition, which released double the volatile contents at 815 °C compared to 575 °C. Thus at 575 °C, half the volatiles in the base coal are still present in the ash. In contrast the biomasses displayed a 5% increase in volatile mass released between the ash produced at 575 °C and ash at 815 °C. Thus, for the biomass samples, almost all the volatiles in the biomass have been released by 575 °C. The biomass contains on average 3 times the amount of volatiles (78%) compared to the coal (26%). Furthermore, the coals have generally at least 10x the ash content of biomass e.g. Kellingley (24.1% ash) and B117 (1.4% ash).

Section 3.1 highlights the optimum pelleting methodology to be used. Any further variation in this test is due to the preparation of the ash or pressure during pelleting. Impact of pellet type on The key temperatures for comparison were Initial Deformation Temperature (IDT), and the Flow Temperature (FT).

3.3.1. Impact of pellet preparation on IDT trigger

Table 9 shows that the IDT is delayed from the high pressure milled to low pressure un-milled pellets and this effect is consistent across all samples. The average increase in IDT is 32 °C, and in some cases, there is as great a difference of up to 58 °C (EC78). This large change suggests there is a cumulative impact (for biomass) on the IDT from both increased pelleting pressure and no pre-milling.

Table 9 also shows that a similar phenomenon was observed when the same samples were ashed at 815 °C. The average increase in IDT in this case was 47 °C, excluding the B117 sample. The average in this instance is skewed by the EC78 sample, but even so there is a general increase in IDT of 24 °C, which is comparable to the results of the 575 °C ash. This suggests that the difference between the high pressure milled and low pressure un-milled methods is responsible for an average delay in IDT trigger of 28 °C. The results illustrate there can be a cumulative impact of pressure and particle size, but there is a delay in IDT due to poor preparation methods. These results were obtained with excellent repeatability by using the most repeatable pelleting method, MPP, from

Table 9
Comparing pellet preparation methods on IDT trigger.

Sample	High pressure milled 575 (°C)	Low pressure un-milled 575 (°C)	Difference (°C)	High pressure milled 815 (°C)	Low pressure un-milled 815 (°C)	Difference (°C)
Kellingley	1168	1220	52	1194	1238	44
B117	1208	1211	3	1314*	1238	−76*
EC78	917	975	58	920	1040	120
La Loma	1169	1194	25	1181	1192	11
Zondag	1272	1298	26	1275	1292	17

* *Note*: The B117 ashed at 815 and pelleted at 7500 psi gave inconsistent outputs even with multiple repeats.

Table 10
Comparing effects of ashing temperature on IDT trigger.

Sample	High pressure milled 575 (°C)	High pressure milled 815 (°C)	Difference (°C)	Low pressure un-milled 575 (°C)	Low pressure un-milled 815 (°C)	Difference (°C)
Kellingley	1168	1194	29	1220	1238	18
B117	1208	1314*	107*	1211	1238	28
EC78	917	920	3	975	1040	65
La Loma	1169	1181	12	1194	1192	−3
Zondag	1272	1275	4	1298	1292	−6

Section 4.1. The high pressure milled, and low pressure un-milled pelleting conditions support the results of the voidage study with the high pressure milled method consistently reducing the IDT. In the case of EC78, the improvement was as high as 120 °C. There is an indication from the results that the relative change in IDT for biomass is bigger than for coal. It is possible that some fuels are more impacted than others. Further work is required to explore this for a greater range of biomass and emerging fuels such as algae and refuse derived fuels.

3.3.2. Impact of ashing temp on IDT trigger

Table 10 illustrates that there was a small impact on IDT as a result of ashing temperature. For the high pressure milled scenario, the average reduction in IDT was 11 °C, and for the low pressure un-milled, no general trend was observed. Increasing the ashing temperature delayed the IDT for both biomass samples. The impact was not consistent for the coal samples in the low pressure un-milled scenario, with the majority seeing negligible impact.

There are no trends to link to the TGA volatile release data from Table 8, which showed that 50% of the volatilised species were lost after 575 °C in the coal samples. This suggests that any effects of ashing on the IDT or FT may be linked to the release of residual volatiles. It can be concluded that the release of volatiles between 575 °C and 815 °C does not play a role in AAFT. Whilst the biomass samples did not show a greater volatiles release between 575 °C and 815 °C, there was a delay in the IDT in the worst-case samples. This further supports that volatile release and ashing temperature does not play a role in ash fusibility. This supports the conclusions from some literature [37], whilst contradicting the findings of other researchers [36,38]. The latter clearly states that an increased ashing temperature increases IDT. The method of pelleting for these studies were the standard cone press, which has been shown to have poor repeatability and delayed IDT trigger. A larger investigation including a wider range of fuels, using the optimised method would provide a strong conclusion to the impact of ashing temperature on AAFT behaviours.

3.3.3. Flow temperature

The impact of pelleting, preparation and ashing on FT was negligible, and proved to be less affected by varying the particle size, pelleting pressure and ashing conditions. On average, the difference in FT for coal high pressure milled and low pressure un-milled cases was 3 °C, and for the ashing comparison was 14 °C. The biomass results showed a reduction in FT of 12 °C for the high pressure milled and low pressure un-milled comparison. The ashing study revealed a 28 °C general increase in FT for the 815 °C ash compared with the 575 °C ash, but this was the only instance that showed comparable change to the IDT study.

3.4. The optimised advanced ash fusion test (OAAFT)

The optimised version of the AAFT satisfies the need to create a standardised test that is clearly repeatable whilst giving the best representation of the first point of melting. The equipment used to create the MPPs, IUTS 3360, is common in mechanical engineering test laboratories throughout the world, is low cost and requires low skill to operate. The potential impact on standard fusibility tests for coals [29,30] may require increased detail in preparation of ash and adopting of 5 mm MPPs. Furthermore, the repeatable nature of the OAAFT test curve means that there is a potential to utilise this characteristic curve to understand more about ash behaviour in relation to onset of slagging and fouling. This curve is unique to the fuel being tested and a database of fuels might then be used to help predict blending properties. The OAAFT process includes:

- The selected fuel milled to below 212 μm
- Ashed at 575/815 °C (biomass/coal)
- Ash milled to below 76 μm (if any particles do not pass include them in the sample)
- 0.100 g of ash (± 0.05 g) compressed into 5 mm pellet at 7500 psi
- Ash fusion test run at 7 °C/min from room temp (or 300 °C for consecutive tests) to 1600 °C/completion of the test (or capacity of the furnace)

This test used a novel pelleting methodology to provide greater repeatability, change in morphology and earlier indication of IDT. It has been verified by 5 different fuels including 3 coals and 2 biomass and reinforces the importance of consistent ash preparation.

4. Conclusions

This study investigated the different pelleting types used during the ash fusion test to develop an optimised preparation technique with enhanced accuracy and reliability. This method was then applied to a range of other fuels to verify the technique and assess the significance of ash preparation. A modified version of the AAFT was used to generate the AFT profiles and highlight key temperatures. The novelty of the work identifies a new pelleting method for the AAFT and identifies its impact on a range of industrially used coals and biomass. The

findings which can be drawn from this study are:

- For the first time it has been shown that cones and the hand pressed pellets do not have good repeatability and delay the response of the IDT when compared directly to the MPPs.
- The 5 mm MPP provided excellent repeatability, identified the 2nd earliest IDT (after the 10 mm MPP), gave the least error and displayed the greatest change in H/Hi to enable easier identification.
- Reducing maximum ash particle size to < 72 μm and increasing the pressure of the pelleting process can produce a 120 °C reduction in the IDT, with an average reduction of 40 °C.
- A reduced ashing temperature for coal and biomass resulted in no evidence to support ashing temperature plays a role in IDTs, and thus volatiles lost during ashing played no part in the change in IDTs.
- Preparation of ash and pelleting method has a greater impact on biomass ash compared to coal ash.
- An optimised advanced ash fusion test (OAAFT) has been developed.

The optimised version of the AAFT creates a standardised test that is repeatable and gives the best representation of the first point of melting. This test will be useful to generate comparable results to aid in boiler operation and fuel selection decisions which might help prevent premature outages and the sourcing of unsuitable fuels.

Acknowledgements

This work was supported by the Engineering and Physical Sciences Research Council (EPSRC), Centre of Doctoral Training in Carbon Capture and Storage and Cleaner Fossil Energy [grant number EP/L016362/1] and the International Young Scientist Research Fellowship, the Natural Science Foundation China (NSFC) [grant number 51650110508]. A special thanks to Carbolite-Gero for supplying the CAF furnace and the continual support throughout the research. The authors would like to thank all those involved in the project for their support and assistance.

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