HIGH CARBON PULVERIZED FUEL ASH AS A FUEL ADDITIVE IN CLAY BRICKS

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KEYWORDS: High carbon flyash; flyash as a solid fuel additive in clay bricks; variability in high carbon content flyash; use of carbonaceous wastes in clay brick manufacture.

SUMMARY

This investigation commenced with the sampling of high carbon PFA being produced as a by-product arising from the operation of a carbon-separation process-plant at Didcot A power station. The sampling exercise took place over a number of days and the set of samples collected were then analyzed to evaluate their properties and variability in to judge the potential suitability of this material as a solid fuel additive for commercial brick manufacture – with particular reference to use at the Desford brickworks of Hanson Building Products Ltd.

The investigation was complicated by the difficulties experienced due to the inconsistent running of Didcot A power station during the latter part of the overall sampling period, within which, the collection of samples was divided into two separate phases. During the first period, the power station was operating continuously and the analyzed properties of the material collected showed a good level of consistency. However, during the second collection period, the station's operation was interrupted by irregular shut-down’s and the test results of the samples obtained were found to be detrimentally affected. This was particularly noticeable in relation to their carbon contents that were found to fluctuate considerably. This was of concern as consistency would be a key requirement for its use.

A complimentary study was also carried out on a stockpile of the PFA located in the power station storage area. The same testing/evaluation procedures used earlier were applied again, using bulk samples obtained by vertically trenching across the stockpile. The results from the individual trenches showed an agreeable level of consistency. This ‘evening-out’ of properties (particularly carbon content) is considered to be a result of the stockpile emplacement procedure, which follows a layer-upon-layer ‘build-up’ pattern, thereby creating a stable layered structure in which individual ‘days-make’ material overlays earlier material. The evidence of the testing results obtained, suggest that if this stockpile were to be subsequently recovered/removed by vertical (downward) excavation, it would result in even further effective homogenizing, thereby yielding a
'product' of high consistency and likely to be well within the boundary of specification required by Desford brickworks for use as a solid-fuel additive for brick making.

The concluding part of the report addresses the planned full-scale factory trial of the Didcot high carbon by-product at Hanson’s Desford brickworks, to be held in the Spring of 2009. The purpose of this study was an evaluation of the influence of this PFA on the ceramic properties of the bricks being produced at this factory and in particular its suitability to be used as a replacement for the coal fines currently added as a fuel additive (sourced from Rossington coal-mine).

This empirical evaluation was carried out in two phases. The first involved an assessment of the overall ceramic behaviour of the Desford brick-body when containing increasing levels of both the Rossington coal-fines and Didcot high carbon PFA in parallel on an equal heat-value input. As the calorific value of the Rossington material is approximately three times that of the Didcot PFA, the respective levels of w/w addition were proportioned accordingly. Overall, similar patterns in ceramic behaviour were recorded for both experimental products. However, the set containing the PFA was observed to exhibit more pronounced property changes than that containing the coal fines. This difference can be attributed to the three-fold addition of the PFA required to achieve equivalent heat-input. The results confirmed the ability of the Didcot high carbon PFA to be used in the same role currently provided by the Rossington coal fines.

In the concluding part of this laboratory evaluation, clay bodies containing the two alternative carbonaceous materials were used in an experiment reproducing the forming method employed at the Desford brickworks (extrusion wire-cut). Both materials were introduced on a like-for-like c/v basis (necessitating three times the weight of PFA to match the Rossington material). After making and drying, bar-samples of the extruded products were divided and two representative batches were sent to the Desford brickworks to be fired through the tunnel kiln with standard bricks under two different firing environments. A third batch was fired in Staffordshire University's ceramics laboratory in an electric kiln following a similar firing profile to that operating at the brickworks.

Subsequent ceramic testing revealed the product containing the Rossington material to be marginally superior in ceramic properties. However, the difference in magnitude between this and the PFA products suggests that the performance of the mix-design containing 15% w/w of high carbon PFA would comfortably meet market requirements. Moreover, in addition to offering an alternative carbonaceous additive, the results achieved confirmed that a product of this sort can not only be successfully fired in a commercial kiln, but also possesses a number of supplementary advantages to be considered when comparing it with the current use of coal fines. These are noted at the conclusion of the report.
INTRODUCTION

This research and development was led by RWE npower with support from Staffordshire University, Akristos Ltd and Hanson Building Products Ltd.

The content of the investigation report is presented in the form of three interconnected 'stand-alone' sections (work-packages) these collectively address all the technical issues surrounding the potential future success of this by-product in commercial brick manufacture. They are arranged in the document in the following order:

Section one:
An investigation of the quality and consistency of the high carbon PFA being produced as a by-product of the carbon-separation process-plant installed at Didcot power station.

Section two:
An evaluation of the quality and consistency of the stockpiled high carbon PFA produced as a by-product of the carbon-separation process-plant installed at Didcot power station.

Section three:
A laboratory programme involving ceramic mix-design development, production and testing to confirm the potential success of introducing Didcot high carbon PFA into the clay mix-design used at Desford factory – in preparation for a planned full-scale factory proving trial in 2009.
SECTION ONE

AN INVESTIGATION OF THE QUALITY AND CONSISTENCY OF THE HIGH CARBON PFA BEING PRODUCED AS A BY-PRODUCT OF THE CARBON-SEPARATION PROCESS-PLANT INSTALLED AT DIDCOT POWER STATION.

1.0 Introduction

Various carbonaceous materials are added to commercial clay bricks during their manufacture mainly for one of two of the following reasons:

a) To reduce the amount of premium-grade fuel (normally gas or oil) required for heating the kiln. This procedure works because the carbonaceous matter ignites and liberates additional energy from within the bricks during the firing process.

b) To promote a localized low-oxygen atmosphere at the interfaces between close-set packs of bricks during their firing. Once in the firing zone this carbonaceous additive oxidizes, thereby causing a decrease in the oxygen level in the kiln gases and promoting localized ‘reduction’ conditions. The outcome is the development of variable colours and textures on the outer surfaces of the bricks (such as blue/black-spotting and dark-patching). These visual effects enhance their aesthetic appeal, thereby allowing them to command a higher selling price.

1.1 Origin and characteristics of Rossington coal-fines

To achieve the above effect, Hanson Brick currently use a fine coal residue obtained from a coal preparation plant operating on the site of the Rossington colliery near Doncaster. During coal processing ‘fines/dust’ arises from the crushing and screening of freshly mined coal. After removal by washing, it is hydro-cycloned, then centrifuged, to reduce its water content. It is then stockpiled ready for sale. From samples provided to Staffordshire University, it can be broadly characterized as being: <5.0mm in sizing, with a typical heat-value of 18.0 GJ/tonne and moisture content of 24-28% (WWB).

1.2 Origin and characteristics of Didcot high carbon PFA

This material is the by-product of an electrostatic carbon-separation process-plant developed by STI (Separation Technologies Inc) operating at Didcot power station. The process can be summarized as follows.

After the PFA exits the power station boilers, it is captured by electrostatic precipitators and deposited in holding silo’s. From here, a proportion is conditioned with water and trucked to the ash holding site for storage/sale into markets such as engineering fill. However, a proportion is diverted as dry ‘raw feed’ and held in a silo prior to being fed into the carbon-separation process-plant. After carbon-separation two streams of PFA, a low carbon stream and high carbon stream, are directed into separate hoppers and subsequently blown into dry storage silos.
The low carbon PFA material (which now complies with BS EN 450-1:2005+A1:2007) is stored in its dry condition ready to be removed by powder tanker for delivery to construction sites as a concrete additive (pozzolan). The ‘carbon-rich’ by-product is conditioned with water to 7-8% (WWB), enough for dust suppression and returned to the coal conveyor for re-burn or conditioned with water to 25-28% (WWB) and sent to a designated ‘high carbon’ stockpile within the power station’s ash storage area awaiting re-use.

2.0 Variability study of Didcot high carbon PFA

As with all additives used by the ceramic industry for commercial brick manufacture, it is essential to monitor the level of variability of its diagnostic properties over time. Previous studies by Staffordshire University relating to the potential application of a wide range of industrial and municipal by-products as brick making additives, has confirmed the importance of closely measuring these properties. Significant fluctuations in calorific value (c/v), or variations in physical/mineral composition, can seriously affect the firing behaviour and fired properties of bricks containing such by-product materials. In this context, the following sample collection programme was conducted in co-operation with the RWE npower Projects Engineer based at Didcot.

2.1 Sample collection

The sample collection procedure carried out involved taking ~4/5kg ‘grab samples’ from the outlet point of the ‘high carbon’ discharge silo of the carbon-separation process-plant (after it had been conditioning with water). These were each put into sealable plastic tubs and then dispatched to the University for analysis.

The original proposed sampling timetable in February 2008, was subsequently found impracticable to implement. This was due to the intermittent running schedule of Didcot power station during the time originally allocated for this undertaking. As a practical compromise, two batches of five daily samples, were collected at two separate times (denoted First and Second Sampling Periods). The first took place in June 08 and the second in September/October 08.

2.2 Testing procedures undertaken

On arrival at the University ceramic laboratories, the following procedures were carried out on each of the ten submitted samples to establish their characterization pattern.

- % Moisture content on arrival
- % Loss-in-weight on firing
- Calorific value
- Laser particle sizing
- Firing behaviour (using Simultaneous Thermal Analysis)

The details of each of these testing procedures are described below:
a) % Moisture content

Weighed amounts (~200g) of each sample were placed in evaporating dishes and dried to constant weight in a laboratory fan-drier, then reweighed. Their moisture contents were then calculated on a wet-weight basis (WWB) i.e. expressing the moisture contents as a percentage of the total weight of the 'as-received' sample. Each sample moisture content was determined in triplicate and the three results then averaged to produce a single representative value.

b) % Loss-on-ignition (LOI) on firing

Weighed amounts of (~100g) of each sample were prepared in triplicate. Each was placed in a ceramic crucible. These were then placed together in an electric furnace programmed rise to 800°C, then held for 2 hours. After cooling, they were re-weighed and their percentage loss in weight (loss-on-ignition) values calculated. The three separate values obtained for each sample was then averaged to produce a single representative value.

c) Calorific values

Representative sub-samples of each of the 10 samples were dispatched to the Oil and Fuel Laboratory of RWE npower's Central Support Laboratories for calorific values to be measured by LECO analyzer.

d) Laser particle sizing

Full particle-size measurements of each of the ten samples were carried out using a Malvern 'MasterSizer'. This instrument operates by measuring the diffraction-pattern produced by the interference of a laser beam with particles (in this case PFA) dispersed and suspended in a solution of water which is constantly agitated (ultrasonically) to prevent particle settling.

In terms of civil engineering nomenclature, granular solids are classified according to their specific grain-size diameter. Thus, 'clay' is recognized (by convention) to be material of <2.5μm in diameter, 'silt' ranges between 2.5-62.5μm, 'fine sand' between 62.5-250μm and 'medium sand' between 250-500μm ..... and so on - up to boulder sizes (in excess of 256mm). To provide a comparison platform, each of the Malvern plots for the individual high carbon PFA samples have been recorded in accordance with this criterion and the results shown in Table 2. In addition, their full particle size curves as measured by the Malvern instrument are also provided (Fig. 3a,b).

e) Simultaneous Thermal Analysis (STA)

To compare the combustion behaviour of the samples and detect any differences in their individual firing patterns during heating (important if used as a body-fuel in brick manufacture) the following analytical procedure was carried out using a Netzsch
Simultaneous Thermal Analyzer (Model STA 409 EP). Two different values are measured at the same time by this instrument: a) Differential Thermal Analysis (DTA) and b) Thermal Gravimetric analysis (TG). Alternate samples within the collected suite of (ten) samples were submitted for evaluation. This analytical method is widely employed in the ceramics industry as it has the ability to record the influence of increasing temperature on the physical and chemical properties of the raw material submitted for testing.

The results of testing procedures (a–c) carried out on the ten samples, are recorded in Table 1 and presented in graphic form in Figures 2a-c. Table 2 provides the laser particle-sizing results according to civil engineering nomenclature. Additionally, their respective full size-distribution curves are shown in Figs. 3 a,b. The STA plot results are shown in Fig. 4 a-b.

<table>
<thead>
<tr>
<th>Sample identity number</th>
<th>Sample collection date</th>
<th>% Moisture as sampled (W/W)</th>
<th>% LOI</th>
<th>Calorific value (kJ/kg)</th>
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<td></td>
</tr>
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</table>

Table 1: Compiled properties of collected samples of the Didcot high carbon PFA samples
Table 2: Showing the particle-sizes composition of the Didcot high carbon PFA samples

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<tr>
<th>Sample identity number</th>
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<th>% &lt;2.5 μm (Clay)</th>
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<th>62.5-250μm (Fine sand)</th>
<th>250-500μm (Medium sand)</th>
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<td>5.05</td>
<td>56.67</td>
<td>37.91</td>
<td>0.37</td>
</tr>
</tbody>
</table>

3.0 Discussion of results

The discontinuous sampling opportunities and relatively small number of samples finally obtained (10), does not allow a statistical assessment of variability to be made. However, the sporadic operation of Didcot (particularly over the second sampling period), provides a good indicator of the typical level of variation that may be expected for this material on a day-to-day basis for the remaining life of the power station.

The % moisture content values listed in Table 1 (and also shown graphically in Fig. 2a) display a noticeably contrasting pattern when the results from both sampling periods are compared. Thus, as can be seen, the level of conditioning water being added on exiting the carbon-separation process-plant remains relatively consistent during the first sampling period - ranging between 22.30-24% (WWB). However, the second sampling period material displays a much wider fluctuation, ranging between 18.27-39.49% (WWB). The commercial implications, if this material were being trucked off-site to Desford brickworks direct from the carbon-separation process-plant, are that it would contain inconsistent levels of carbonaceous matter, due to the varying amounts of water present.

The Loss-on-ignition (LOI) results listed in Table 1 and also shown graphically in Fig. 2b, display relatively consistent values for the first sampling period, but wide variations among the samples for the second sampling period. Without further and more extended sampling
Figure 2a: % Moisture contents of 'high-carbon' PFA sample sets from Didcot

Figure 2b: % Loss-on-Ignition values of 'high-carbon' PFA sample sets from Didcot
Figure 2c: Calorific values of 'high-carbon' PFA sample sets from Didcot

and evaluation, no definitive explanation is currently apparent. However, it should be noted that the first sampling period carried out over five consecutive days, coincided with a period of continuous running of the power station. During this time, it can be assumed that it would have maintained a stable running state. In contrast, the second sampling period was forced to be conducted during a period of discontinuous running and consequently took fifteen days to complete. The erratic results obtained possibly reflect either unstable firing conditions in boiler operation giving rise to different levels of carbon in the PFA, or the inability of the carbon-separation process-plant to operate successfully under such irregular operation.

The calorific values measured for each individual sample (expressed in kJ/kg) are recorded in Table 1 and also shown graphically in Fig. 2c. These results confirm the same contrasting pattern of values between the samples obtained during the first collection period and those obtained during the latter collection period. The results broadly mirror the LOI values, insomuch that as expected, higher LOI results reflect higher c/v values. The variation range of the results returned by the second sampling period is extreme, fluctuating widely from 3437 to 9778 kJ/kg. They are also by and large much higher than those recorded for the first collection set. Again, an explanation for this pattern of result must be concluded to be most likely to be due to unstable firing conditions in the power station boilers over this latter period.

The results of the overall particle size measurement of the samples from both sampling periods expressed in terms of engineering nomenclature are provided in Table 2. Although slight differences in proportional size-composition are exhibited between individual samples, the underlying pattern confirms them to all fall dominantly within the silt-fine sand category of sediments. Moreover, as shown in Figs. 3a,b displaying the full
particle-sizing profiles of each of the samples (measured by Malvern MasterSizer) all show very similar overall size profiles, indicating good packing characteristics and a good level of sizing compatibility.

Daily sample 1 (12.05.08)

Daily sample 2 (13.05.08)

Daily sample 3 (14.05.08)
Daily sample 4 (15.05.08)

Daily sample 5 (16.05.08)

Fig 3a – Malvern results: First sampling period
Finally, the **STA analyses results**. In Fig 4a the plotted results of three alternate samples (1, 3 and 5) from the first sampling period are shown and in Fig 4b samples 6, 8 and 10 taken during the second sampling period are presented for direct comparison.

In essence, their thermal behaviour patterns* are shown to closely replicate each other in overall profile. The only noticeable departure shown is the much more pronounced levels of thermal activity displayed in samples 6 and 10 (second sampling period) where the amount of carbonaceous matter is shown by the recorded evidence of their thermogavimetric profiles to be far higher. This is confirmed from the results of their respective LOI values (Table 1) which shows an average value of between 24-28% for samples 1, 3, 5 and 8 compared with these latter two samples which show values of >38%. 

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* STA = Thermogravimetric Analysis
Daily Sample 1 (12/05/08)

Daily Sample 3 (14/05/08)

Daily Sample 5 (16/05/08)

Figure 4a Selected STA results: First sampling period
Fig 4b Selected STA results: Second sampling period
4.0 Conclusions

This first part of the investigation has examined the composition and variability of the high carbon PFA produced as a by-product of the carbon-separation process-plant. The overall results were mixed with those samples obtained under steady power station operating conditions (first sampling period) demonstrating a good level of compatibility in properties. However, those collected during the subsequent (second sampling period) reflected the intermittent operation of the power station at that time. Of particular concern in this context was the high level of variability in carbon content found in the samples, as this is the essential property of interest for use as a fuel additive at the Desford brick factory.

From the overall evidence obtained it is considered likely that if the high-carbon residue PFA was drawn off from the carbon-separation process-plant and taken directly to the brickworks on a daily basis, the level of consistency (primarily of carbon content) would be unacceptable while the station was operating on a start/stop basis.

The following part of the investigation (Section Two) was undertaken to evaluate whether material excavated from the stockpile of this high carbon material possessed an acceptable level of consistency.

* A more detailed analysis of the thermal behaviour of this material is given in Section Two, ‘STA tests’,
SECTION TWO

AN EVALUATION OF THE QUALITY AND CONSISTENCY OF THE STOCKPILED HIGH CARBON PFA PRODUCED AS A BY-PRODUCT OF THE CARBON-SEPARATION PROCESS-PLANT INSTALLED AT DIDCOT POWER STATION.

1.0 Introduction

The high carbon PFA stockpile at Didcot power station has been identified as potentially offering a source of body-fuel for use in brick manufacture. However, an essential requirement for the successful commercial use of all such carbonaceous additives is ‘consistency’ of certain essential physical and chemical properties. To confirm the acceptability of the Didcot material within this context, a sampling programme was carried out over its storage stockpile to procure representative material for testing.

The sampling undertaking was a combined operation between Akristos Ltd, RWE npower and Staffordshire University. Akristos is a well known commercial supplier of ceramic raw materials to the brick industry and has also built-up a reputation for the supply of quality assured by-product feedstocks (secondary raw-materials) as sustainable alternatives to many traditional virgin raw materials. The company has acquired extensive experience in stockpile evaluation and consequently was ideally placed to co-ordinate this preliminary activity.

2.0 Description of the site sampling procedure

The Akristos field sampling operation took place on the 4th June 2008. The stockpile which averages 8-10 metres in height overall, was arbitrarily divided into six broad sampling zones (see Fig. 1).

Figure 1: Aerial view of Didcot PFA storage facility showing location of trench samples taken across the ‘high carbon’ PFA stockpile for evaluation (positioning by GPS).
A Komatsu bucket-excavator (Fig. 2) was then used to dig trenches across the stockpile at each of these points. Trench 1 was dug down to the underlying solid ground at an estimated depth of 7-8 metres and in doing so, intersected a rubble-zone of contaminating material towards the base. To avoid re-encountering this problem again, subsequent trenches were only dug to approximately 3 metres depth (Fig 3). During extraction, the excavated material was deposited in piles by the side of the holes (Fig. 4). Each pile was then selectively sampled to obtain a bulk product representative of the full depth of the trench (Fig. 5). The six collected samples were then placed in 5.0 litre sealable plastic-tubs for onward transport to the University.

![Excavator used for stockpile sampling](image1)

**Figure 2: Excavator used for stockpile sampling**

![Full depth trench](image2)

**Figure 3: Full depth trench**

![Dug material formed into a pile](image3)

**Figure 4: Dug material formed into a pile**

![Sampling excavated (bulk) PFA](image4)

**Figure 5: Sampling excavated (bulk) PFA**

3.0 Staffordshire University laboratory evaluation

On arrival at Staffordshire University, the contents of each plastic container were first hand-mixed to achieve further homogenization. Smaller ‘sub-samples’ were then obtained from each of these original bulk samples by sub-dividing the contents of each container to further reduce its bulk-volume, while still maintaining its ‘representative’ composition.

This was accomplished using an Endecotts ‘sample-splitter’ (Fig. 6). The function of this equipment is its ability to divide any introduced powdered material into two separate
(but equal) parts. This is achieved through its internal construction which consists of a series of channel-ways which form 'slots' (or chutes) alternately splitting and subdividing (left and right). The original sample is poured in at the top and subsequent collected at the bottom. The bottom of each channel slopes at $60^\circ$ with six alternate slots leading into one of two basal collection boxes.

Figure 6: Endicott sample splitter with collection boxes at the base

Due to the design, these two boxes receive equivalent amounts of the starting sample. Applying this procedure by repeated passings, the full content of each plastic container was progressively divided and then subdivided into halves during this progressive operation. To achieve this, weighed amounts of 1.0kg were taken from the bulk amount of PFA in each container and then poured into the splitter. Each time, one of the two halves discharging at the bottom was discarded while the other was stored. This same procedure was repeated for the next weighed amount ... and so on, with the retained halves subsequently mixed together and passed through in the same way, until approximately 0.5 kg of final material (representative of the whole original bulk sample) remained.

The following analytical procedures were then carried out on each of the six 'representative' container samples prepared in the above way.

3.1 % Moisture content

The % moisture content of each sample was determined in triplicate. The procedure used involved weighing (~100g) amounts into porcelain crucibles at their 'as-received' moisture contents. These samples were then dried in a laboratory fan-oven set at $110^\circ$C for six hours and then re-weighed. The three determined % (wet weight basis) moisture values obtained from each sample were then averaged. The results are shown in Table 1.
3.2 Full particle-size analysis

Particle-size measurements of each of the six PFA samples were undertaken using a Malvern 'MasterSizer'. This instrument operates by measuring the diffraction pattern produced by the interference of a laser beam with particles (in this case PFA) dispersed and suspended in a solution of water, which is constantly agitated (ultrasonically) to prevent particle settling. The results are shown plotted as histograms in Figure 7a,b.

3.3 % Retention on 45μm mesh sieve

As a supplementary test for assessing the relative 'coarseness' of the samples, a weighed amount of 100gm (pre-dried) material was washed through a BS 350 mesh (45μm sieve). The residue retained on the sieve is then dried and re-weighed and calculated as a percentage of the total dry weight of the original sample. This procedure is commonly used in the cement and concrete industries as a rapid indicator for assessing this same parameter and is also part of BS EN 450 standard. The results are shown in Table 1.

<table>
<thead>
<tr>
<th>Trench sample number</th>
<th>Sampling date</th>
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<th>% LOI</th>
<th>Calorific value (kJ/kg)</th>
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<td>47.3</td>
<td>27.84</td>
<td>6154</td>
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</table>

(Trench sampling undertaken by Akristos Ltd)

Table 1: Comparative results obtained from Didcot 'high carbon' PFA stockpile sampling

3.4 % Loss-on-ignition

To determine the amount of combustible matter associated with the PFA, a quantity of each sample was first pre-dried. Amounts of 20g were then weighed out (in triplicate) into ceramic dishes. The samples were then heated in an electric furnace to 800°C and held for 2 hours. After final cooling in a desiccator, they were re-weighed and their percentage mass-loss calculated. The three separate values obtained for each sample were then averaged. The results are shown in Table 1.
3.5 Calorific values

Further amounts of each of the six samples were dispatched to the Oil and Fuel Laboratory of RWE npower’s Central Support Laboratories for their calorific values to be measured by LECO analyzer. The results obtained are shown in Table 1.

3.6 Simultaneous Thermal Analysis (STA)

To compare the combustion behavior of the individual trench samples and detect any differences in behaviour during heating (important if used as a body-fuel in brick manufacture), Differential Thermal Analysis (DTA) and Thermal Gravimetric analysis (TG) were carried out simultaneously on alternate samples taken from across the stockpile. This analytical technique is widely employed in the ceramics industry as it has the ability to record the influence of increasing temperature on the physical and chemical properties of any raw material being evaluated. The instrument used for this evaluation was a Netzsch Simultaneous Thermal Analyzer (Model STA 409 EP). The resulting firing plots are shown in Fig. 8.

4.0 Discussion

In the following assessment, each measured parameter is considered in turn. The examination begins with the % moisture contents determined for each sample. These values were found to vary across the stockpile ranging from between 26-32% (WWB). These results are acceptably consistent in terms of typical stockpile moisture variations of other ceramic raw materials such as clay and sand etc. PFA possesses good drainage properties and this level of moisture variation is well within expected boundaries. If being trucked regularly to a brick factory, weighbridge records would be likely to reflect little variation from load to load. This would allow storage and mix-proportioning to be carried out without difficulty.

Particle sizing of the deposit was also found to be very consistent across the stockpile. This is shown in the almost identical trench-sample histograms presented in Figure 7a,b.

The supplementary sieve-analysis undertaken (% retention on a 45μm mesh) provided values ranging between 47-55%, additionally confirming a satisfactory level of consistency in particle grading across the sampled deposit.

% Loss-on-ignition values are shown to range between 27-36% resulting from the oxidation of the combustible matter associated with the PFA, but also including smaller weight-losses associated with chemisorbed moisture and water from the breakdown of any hydrated salts during the heating procedure.

The calorific values show a broad level of agreement over the sampled area. A rough relationship is found between sample LOI values and corresponding calorific values for
individual trench samples, apart from that recorded from Trench 1. As noted earlier (Section 2.0) this first excavated trench intersected contaminating material towards the

Figure 7a: Showing Malvern particle-size analysis profiles of Trench samples 1-3
Trench sample 4

Trench sample 5

Trench sample 6

Figure 7b: Showing Malvern particle-size analysis profiles of Trench samples 4-6
Figure 8: Comparison of Differential Thermal Analysis plots of three selected Trench samples.
base. Although this was avoided through selective sampling, it is possible that a small trace may have remained to explain this higher than expected value.

The **STA tests** were carried out in a neutral firing atmosphere over the temperature range 20°C-1200°C at a ramp rate of 6°C/minute. The plots of the three samples tested on this instrument are shown together in Figure 8. The Differential Thermal Analysis (DTA) behavior is indicated by a blue plot-line. Exothermal reactions (heat given off) plot upwards and endothermic reactions (heat absorbed) plot downwards. The mass loss (TG) of the sample occurring during the firing is shown by a red line. In each sample the overall DTA plots show only two thermal events. An initial endothermic event beginning at about 100°C which indicates the dehydroxylation of a small amount of chemisorbed moisture still held in the sample, together with (perhaps) the disassociation of an even smaller amount of hydrated sulphates. The second event is a large exothermic reaction that takes place between (600-800°C) as the carbonaceous matter associated with the PFA progressively burns away. At the same time, a similarly pronounced loss in mass is recorded by the TG plot over this same temperature range. The pattern produced by this prolonged combustion reaction (and the gases evolved) is dependent on the nature of the carbonaceous matter. The plot displays a first shoulder, peaking at around 600°C, followed by a later higher peak at around 800°C before it rapidly falls back, with no further thermal events occurring. It is likely that the combustion of surviving grains of coal is responsible for the initial peak with de-volatized carbon (coke) or graphitic carbon oxidizing at the somewhat higher temperature and thereby extending the upward profile of the plot-line.

A side-by-side comparison of the three STA plots shown in Figure 8 reveals them to be virtually identical and confirms the consistent composition of the deposit, which is also shown to be free from any other contaminating mineral matter.

5.0 Conclusions

In assessing the overall results obtained from the current sampling, testing and evaluation programme of the Didcot high carbon PFA stockpile material, it should be recognized that due to the limited number of samples obtained and examined, no statistical analysis of individual property variations has been possible. Thus, it should be appreciated that the study undertaken was specifically targeted towards achieving an ‘overview assessment’ of the quality and consistency of this deposit according to a range of relevant criteria. In broad terms, the results obtained confirm a good level of overall consistency throughout the stockpile. If this deposit were subsequently recovered/removed by vertical (downward) excavation, it would provide even further effective mixing, thereby yielding a ‘product’ considered to be well within the specification required by the brickworks for use as a solid-fuel additive to the bricks.
SECTION THREE

A LABORATORY PROGRAMME INVOLVING CERAMIC MIX-DESIGN DEVELOPMENT, PRODUCTION AND TESTING TO CONFIRM THE POTENTIAL SUCCESS OF INTRODUCING DIDCOT HIGH CARBON PFA INTO THE CLAY MIX-DESIGN USED AT DESFORD FACTORY – IN PREPARATION FOR A PLANNED FULL-SCALE FACTORY PROVING TRIAL IN 2009.

1.0 Introduction

As result of the potential interest in the use of Didcot high carbon PFA a future full-scale factory proving trial is under consideration. This will be carried out in cooperation with the Technical Manager of Hanson Building Products Ltd at their Desford factory near Leicester. In preparation for this undertaking, a preliminary laboratory investigation has been undertaken at Staffordshire University to pre-assess the influence that the high carbon stockpile PFA will be likely to have on the ceramic properties of the bricks scheduled for production during the trial.

Desford brickworks manufactures a range of facing bricks using a basic raw material known as Keuper Marl (geologically designated as Mercia Mudstone) which is excavated from an adjacent quarry. However, in addition, smaller quantities of other types of clay are imported into the factory and variously blended with it to achieve an expanded product range comprising different surface colours and finishes. One particular brick (known as the ‘buff’ product) requires a 5% w/w addition of carbonaceous material to be added to the Keuper Marl. During firing, its oxidization produces localized ‘reducing’ conditions which promotes the development of a buff colour product with dark ‘patching’ on its faces.

The carbonaceous additive currently used at Desford for this product is a fine coal residue obtained from Rossington colliery near Doncaster (approximately 70 miles distance). The origin and characteristics of this material have been previously described (Section One, 1.1).

2.0 Experimental procedure undertaken

To obtain the firing evidence required, two sequential laboratory-based ceramic trials were carried out as described below.

2.1 Mix design considerations (levels of carbonaceous additive)

As noted above, the Desford factory currently uses coal fines to enhance the visual appeal of one of its range of bricks. At the factory, this carbonaceous material is added to the clay-feed from a separate storage hopper. Both materials then travel together on a conveyor belt that delivers them to the mixing-plant where they are blended together. It should be noted that the proportioning is not too exacting. However, even with this lateral variation occurring, the company has found this situation to be acceptable in
terms of product requirements. The amount of Rossington coal-fines added to the clay (calculated back from the amount used annually) is approximately 5% weight-for-weight.

Calorific values have been undertaken by RWE npower's Central Support Laboratories on supplied samples of both the Rossington coal fines and Didcot high carbon PFA which have yielded the following results:

a) Average value of the Rossington coal-finals samples = 18.10 GJ/tonne
b) Average value of the Didcot 'high carbon' stockpile PFA (excluding Trench 1) = 6.60 GJ/tonne

In order to match the heat-input of the Rossington coal fines in the Desford 'buff' product, a addition factor of x3 was applied to the Didcot PFA. This was done on the basis that its c/v is approximately one-third that of the Rossington material. Therefore, by using three times as much in the range of mix-designs prepared, an (approximate) equivalent 'heat-value' is achieved.

<table>
<thead>
<tr>
<th>Rossington coal slurry mix-designs &amp; Codes</th>
<th>% C/F replacement level (DWB)</th>
<th>Rossington C/F (g) (dry)</th>
<th>Desford clay (g) (dry)</th>
<th>Total amounts (g) (dry)</th>
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</thead>
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<tr>
<td>R2</td>
<td>2%</td>
<td>2</td>
<td>98</td>
<td>100</td>
</tr>
<tr>
<td>R4</td>
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<td>6</td>
<td>94</td>
<td>100</td>
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<tr>
<td>R8</td>
<td>8%</td>
<td>8</td>
<td>92</td>
<td>100</td>
</tr>
<tr>
<td>R10</td>
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<td>10</td>
<td>90</td>
<td>100</td>
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</table>

<table>
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<tr>
<th>Didcot high carbon PFA mix-designs &amp; Codes</th>
<th>% PFA replacement level (DWB)</th>
<th>PFA (g) (dry)</th>
<th>Desford clay (g) (dry)</th>
<th>Total amount (g) (dry)</th>
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</thead>
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<tr>
<td>D6</td>
<td>6%</td>
<td>6</td>
<td>94</td>
<td>100</td>
</tr>
<tr>
<td>D12</td>
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<td>12</td>
<td>88</td>
<td>100</td>
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<td>D18</td>
<td>18%</td>
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<tr>
<td>D24</td>
<td>24%</td>
<td>24</td>
<td>76</td>
<td>100</td>
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<tr>
<td>D30</td>
<td>30%</td>
<td>30</td>
<td>70</td>
<td>100</td>
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</tbody>
</table>

| Control clay | 0     | 0     | 100     | 100     |

Table 1: Mix-designs used for the preliminary evaluation of ceramic properties of Desford Keuper Marl bricks containing coal fines versus PFA additions at equivalent c/v levels (three-fold additions of PFA were made to compensate for its lower C/V compared with the coal fines).

The preliminary fabrication work produced two parallel sets of test-pieces containing increasing (but equivalent heat-values) of these two separate carbonaceous additives. Table 1 shows the mix-designs based on 100g batch-weights for producing sets of (three-each) 20g test-pieces (disks) of each of the addition levels indicated. A 100g mix
of pure Desford clay (i.e. containing no added carbonaceous material) was also produced as a control.

2.2 Test piece fabrication and firing

The making, firing and testing procedures carried out are described as follows:

Individual dry mixes were initially prepared in accordance with the mix-designs shown in Table 1. Each mix was then moisturized with 12% water (DWB) and then homogenized by hand mixing in a bowl. These damp mixtures were then brushed through a 1.20mm aperture (14 BS Mesh) sieve to granulate them. In turn, each granulated mix was then weighed-out into 20g amounts and poured into a 38mm diameter cylindrical die (Fig. 1a). These were then compacted in a manually operated hydraulic press (Fig. 1b) at a forming pressure of 9.96 kN (~1 tonnef) to produce the sets of test discs (Fig. 1c) required.

The sets of discs were then placed in a laboratory fan-oven and dried overnight. They were then measured, weighed and set (on-edge) in a programmable electric kiln (Fig 2). The (neutral atmosphere) firing was set to rise at 100°C/hour with a 1 hour pause at 600°C (for carbon burn-out), then progressed on to a top temperature of 1065°C (the same temperature as the Desford kiln reaches for firing its buff product). This temperature was then held for a 1 hour soak, after which the kiln was allowed to cool back to ambient temperature.

Figure 1a: Die used for making discs  Figure 1b: Forming under hydraulic pressure

Figure 1c: 20g pressed disc product
2.3 Testing of fired discs

The fired disc sets were then re-measured and re-weighed and their ceramic properties determined as follows:

a) % Loss in weight – calculated from the % weight difference between the dry unfired disc weights and their weights after firing.

b) % Dry to fired volume shrinkage – calculated from the volume reduction (shrinkage) of the discs from their dry to fired states.

c) Unfired/fired bulk density – obtained by dividing the weight of the discs by their volume

d) % Water absorption – this was determined by boiling the discs in water for 1 hour, then re-weighing them to record the % amount of water that they had absorbed.

e) Fired strength – each of the discs was crushed in a motorized Monsanto tensometer with a compression cage attachment (Fig 3a, b) and their diametral strengths calculated by the following formula expressed as N/mm$^2$

$$2 \times \text{Breaking force (in Newton's)}$$

$$\pi \times \text{Disc diameter (mm)} \times \text{Disc depth (mm)}$$
Figure 3a: View of Monsanto tensometer

Figure 3b: Close up view of Mansanto compression-cage holding disc

The values obtained from each of the three discs of each separate mix-design were then averaged and the mean results recorded (Table 2). These results are also presented graphically in Fig. 4a-f for visual comparison.
<table>
<thead>
<tr>
<th>CODES</th>
<th>Dry bulk density (g/cc)</th>
<th>% Fired loss in weight</th>
<th>% Fired linear shrinkage</th>
<th>Fired bulk density (g/cc)</th>
<th>% Fired water absorption</th>
<th>Fired strength (N/mm^2)</th>
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<td>1.66</td>
<td>1.46</td>
<td>30.20</td>
<td>2.56</td>
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Table 2: Shows the comparative ceramic results of the mix-designs containing Rossington coal fines and Didcot high carbon PFA (D). NOTE: the row highlighted in bold red is the 'interpolated' values of the current Desford brick body containing 5% Rossington coal fines.

3.0 Discussion of testing results

The behavior pattern of the test-pieces in response to the increasing additions of these two carbonaceous wastes is demonstrated by the ceramic testing results obtained, which are discussed individually below.

3.1 Unfired bulk densities

The unfired bulk density of the control body (100% Keuper Marl) is shown in Fig. 4a to be the highest, with both 'carbonaceous additive' bodies displaying decreasing values as their replacement level is increased. This is to be expected as both the coal fines and PFA have lower bulk densities than the clay and as their level is increased the net bulk density of the host body will naturally decrease. Moreover, neither additive possesses any plasticity. As a result, this detrimentally affects particle packing efficiency during the pressing operation, producing more internal voids and consequently further lowering bulk density values. The decline in density within the set of experimental bodies containing the PFA are seen to be more pronounced. This is a result of the fact that the w/w substitution levels used are three times greater by weight than that of the corresponding coal fines test-pieces.
3.2 Fired bulk densities

The bulk density values of the two sets of experimental discs fired to 1065°C (the factory firing temperature) are shown in Fig. 4b. They follow the same pattern of decreasing values with

Figure 4a: Unfired bulk density of experimental disc sets

Figure 4b: Fired bulk density (1065°C) of experimental disc sets
Figure 4c: Loss-in-weight on firing experimental disc sets to 1065°C

Figure 4d: Linear firing shrinkage of experimental disc sets
increasing levels of the additives recorded previously for the unfired products - but they also show somewhat lower density values compared with their unfired state. With many brickmaking raw materials the firing process significantly densifies the products from their unfired to fired states. However, the action of firing the Keuper Marl is unusual inasmuch that the mineralogy of this raw material frequently contains a significant amount of lime and magnesia. On heating the breakdown of these minerals causes it to release carbon dioxide, leading to the development of additional internal voids, producing characteristically porous bricks with relatively low densities. The reduced density of these products is also influenced by the presence of the carbonaceous
additives, which burn away during firing, leaving voids at the sites that they previously occupied.

3.3 % Loss-in-weight on firing

The loss in weight as a result of firing the sets of test-pieces to 1065°C mirror the pattern shown by their fired bulk densities described previously. In Fig. 4c the mass-weight losses shown, are a combined result of both mineral decomposition of the ‘host-clay’, supplemented by the oxidation of the inter-mixed carbonaceous matter contained by the coal fines and PFA. The gross influence on weight loss shown by these two combustible materials can be clearly observed with reference to the 100% Keuper Marl control product. In its natural state, this material contains no combustible matter in its composition. Hence, the weight loss recorded (10.8%) is entirely due to the decomposition of its clay minerals and the breakdown of associated carbonates during the firing process. Using this value as a yardstick graphically shows the influence on weight-loss brought about by the two carbonaceous additives as their amount is increased in the mix-designs. The close compatibility in loss of weight values displayed by the respective plots of these two materials at each incremental addition, confirms the good level of calorific-value matching brought about by the three-fold addition of PFA compared with coal fines, on a weight-for-weight basis.

3.4 % Linear firing shrinkage

Due to the unusual firing characteristics of the Keuper Marl host clay (see 3.2), the linear firing shrinkages (shown in Fig. 3d) are overall very low. The control is less than 1% and although the test-bodies containing increasing replacement levels of coal fines and PFA are shown to promote progressive increases, the levels (for reasons explained previously) are all modest compared with that expected of many other common brickmaking raw materials. The PFA addition set of test products are shown to possess a more pronounced level of shrinkage compared with the coal fines set. This is a reflection of the greater amount used (three times more) compared with counterpart coal fines addition products.

3.5 % Water absorption

On comparing the test-disc sets containing greater levels of PFA (Fig. 4e) a progressive pattern of increasing porosity is displayed. In each case, as the PFA replacement level goes up, the water absorption value of the products rises accordingly. This is due to the increasing level of carbon which burns-out leaving a larger amount of open-pores capable of being filled with water. When the water absorption values of the set of test-pieces containing Rossington coal fines is compared with these results, it can be seen that they are somewhat lower - although in the same way increase as more coal fines are introduced. This difference in magnitude is explained as being due to the greater amount of PFA present, which in all mix-designs (as noted previously) is three-times greater than the amount of coal fines at comparable c/v levels.
3.6 Fired strength

Fig. 4f shows a comparison of the strengths achieved by the two series of test-products. A trend of decreasing strengths is shown by both as the level of carbonaceous addition increases. An explanation of this pattern can be linked with the pattern of increasing water absorption values referred to above. It follows the normal ceramic relationship between porosity and strength i.e. fired products containing higher water absorption values (more internal voids) produce weaker (more open) ceramic micro-textures and consequently possess lower tensile strengths.

This general pattern of behavior can be compared with the control body which contains no such combustible material to burn out and consequently produces a denser and more robust ceramic product, possessing a higher strength and lower water absorption value than the two experimental products as shown both in Figs. 4e and f.

3.7 Conclusions

This preliminary ceramic work was carried out to compare the impact of replacing Rossington coal fines with high carbon PFA on the physical properties of a Keuper Marl brick body. Overall, similar patterns in ceramic behavior were shown by both materials as the amounts of replacement was increased i.e. decreasing bulk densities, increasing loss-in-weight on firing, greater levels of volume contraction, higher water absorption values and a reduction in strength. In each case the experimental discs containing PFA show more pronounced results. However, this can be attributed to the three-fold weight-for-weight 'matching c/v' needed for the PFA compared with the coal fines. In summary a decline in the ceramic properties compared with the control is brought about by the use of both these carbonaceous additives. However it should be noted that the desired enhancements in surface colours of the Desford bricks cannot be attained without carbonaceous additives of this type. Moreover, the existing ceramic properties of the standard Keuper Marl product are significantly in excess of those required for satisfactory building purposes, thereby allowing a significant degree of leeway, which favours the introduction of the Didcot PFA.

4.0 Advanced investigation (simulation of factory production conditions)

The extended laboratory work reported below was additionally undertaken to replicate the brick making procedure carried out at Desford Brickworks. This was progressed to confirm that the encouraging results achieved with the earlier disc test-piece study, could also be satisfactorily replicated at this factory using the making process operating there.

4.1 Mix designs development

The choice of addition levels of the two carbonaceous additives (to compare performances) was based on mix-design considerations explained earlier in sub-section 2.1 of the current Section. A 5% (w/w) addition of Rossington coal fines represents the
amount of this material currently used to produce colour-enhancement for the Desford buff-fired bricks. The addition of 15% w/w Didcot high carbon PFA was used as it is equivalent in net calorific value to 5% of the Rossington material. However, it should be noted that there is some leeway in the addition level used at the brick works due to the imprecision of the handling/proportioning equipment installed. Consequently, two further addition levels of the PFA containing 5% less and 5% more w/w than the target 15% w/w level, were also produced to access the influence of such lateral variation on the ceramic properties of the resulting products.

<table>
<thead>
<tr>
<th>Mix Codes</th>
<th>% Addit. of Rossington coal fines</th>
<th>% Addit. of Didcot high carbon PFA</th>
<th>Total weight of carbon additives in mixes (kg dry)</th>
<th>Total weight of clay in mixes (kg dry)</th>
<th>Total weight of mixes (kg dry)</th>
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<tr>
<td>XII</td>
<td>0</td>
<td>15%</td>
<td>1.8</td>
<td>10.2</td>
<td>12.0</td>
</tr>
<tr>
<td>XIII</td>
<td>0</td>
<td>20%</td>
<td>2.4</td>
<td>9.6</td>
<td>12.0</td>
</tr>
</tbody>
</table>

Table 3: Showing mix-design (dry w/w) proportioning of the experimental extruded products

4.2 Production of extruded test-bar (replicating the Desford making process)

The making method used for brick manufacture at Hanson’s Desford Brickworks is known as wire-cut extrusion. In this process, the clay body is first milled and (if required) the Rossington coal fines can be added and mixed-in with the clay. Sufficient ‘tempering water’ is then added and the mix mechanically tempered, to produce a plastic-mass in readiness for extrusion. This product is then passed into an auger extruder. Here it undergoes two-stage processing. Firstly, it is conveyed by screw-auger into a vacuum chamber where remaining air trapped in it is removed. This de-aired product is then carried-forward by a second screw-auger which both compacts it and forces it under high pressure through an oil-lubricated die of rectangular cross-section. The rectangular ‘column’ of clay that emerges from the mouth of this die is subsequently cut into separate bricks by a wire-cutter that slices vertically down through the fresh column. After this, the bricks are transported to the dryer to remove most of the water, before being subsequently fired through the factory tunnel kiln.

This same making procedure was reproduced at the University using a Rowden laboratory-scale vacuum extruder with a 38.9mm x 26.50mm die to produce solid bar extrusion (see Fig. 5). This machine possesses all the features of the full-sized
brickworks extruder at Desford (albeit being much smaller) and produces results that can be reliably projected across to the performance/behaviour of the full-sized machine.

4.3 Preparatory mixing procedures

The four mix-designs (each 12kg in dry weights) were first prepared by weighing-out the components. These were then placed in a laboratory Z-blade mixer for preliminary blending/homogenizing. Sufficient water was subsequently added to each mix in turn to bring them to an appropriate plastic state for extrusion. In each case, the actual water addition was dictated ‘by-feel’ during mixing to determine optimum results. Each body in turn was then tempered in a Z-blade mixer to ensure homogenous blending. The amount of water used to achieve this optimum plastic state in each of the mixes are listed in Table 4.

Figure 5: Rawdon laboratory extruder used to produce experimental bars containing Rossington coal fines and Didcot high-carbon PFA (insert shows close-up of plastic column emerging from the die-mouth of the extruder).

4.4 Extrusion performance of experimental test-bar products

As described above, the Rowdon extruder produced a de-aired column forced out by the machine’s auger-screw through a 38mm x 25mm rectangular cross-section die. With each successive mix-design, the plastic column emerging from the die-mouth of the extruder was cut into lengths of approximately 250mm. The upper faces of the bars were then indented with 100mm spacing marks, so that their shrinkage’s after drying
could be measured for comparison. The mix codes were also impressed on the upper surface of each bar, which were all numbered sequentially. A hand-penetrometer was used during this stage of the forming operation to record the 'stiffness' of the extruded column (see Table 4). This is a subjective measurement, but allows plasticity differences to be compared. Approximately eighteen bars of each of the four mix-designs were produced under a steady vacuum of 23 bars Hg. A perfectly smooth extruded column was achieved in each of the four cases.

4.5 Unfired property testing results

After the extrusions were completed, the individual sets of freshly produced bars were left on pallets for 24 hours in the laboratory for preliminary drying (Fig. 6). Subsequently, they were transferred to a laboratory fan-oven set at 90°C to dry to constant weight,

Figure 6: Showing freshly extruded bars undergoing preliminary drying

after which linear drying contraction and unfired bending strength were recorded with the individual bar results averaged to produce a single value for each of the four mix-design products. The results (together with the earlier values relating to extrusion properties) are shown in Table 4 and discussed below.

<table>
<thead>
<tr>
<th>Mix codes</th>
<th>% Forming moisture (WWB)</th>
<th>Column stiffness (kg/cm²)</th>
<th>% Linear shrinkage (wet to dry)</th>
<th>Unfired strength (kgf/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>R</td>
<td>15.7</td>
<td>11.0</td>
<td>5.7</td>
<td>29.0</td>
</tr>
<tr>
<td>XI</td>
<td>17.3</td>
<td>10.5</td>
<td>5.6</td>
<td>26.1</td>
</tr>
<tr>
<td>XII</td>
<td>17.6</td>
<td>11.0</td>
<td>4.9</td>
<td>25.7</td>
</tr>
<tr>
<td>XIII</td>
<td>18.3</td>
<td>11.0</td>
<td>4.8</td>
<td>25.9</td>
</tr>
</tbody>
</table>

Table 4: Showing comparative unfired properties of the experimental products
4.5.1 % Forming moisture

The level of water required to produce optimum extrusions shows a small progressive increase as more non-plastic material (the two carbonaceous materials) are introduced into the basic mix-design.

4.5.2 Column stiffness

The results obtained (although subjective) show a very similar level of plasticity for all the products. As noted above, all four extrusions were found to be highly satisfactory.

4.5.3 % Linear shrinkage

The comparative contractions of the four experimental bodies show a regular pattern of decrease as the level of non-plastic material introduced into the mix-designs is increased. This is a result of the ‘groging effect’ i.e. the non-plastic material produces an internal reinforcing structure which resists the normal contraction behaviour of the host clay.

4.5.4 Unfired strength (modulus-of-rupture)

This property was determined on a sample of three dried bars taken from each of the sets of four experimental products. These bars were broken transversely using a Howden Tensometer test-rig (see Fig. 7). The results obtained were averaged to provide a mean value for each of the products. The results are shown in Table 4.

Figure 7: Showing the Howden Tensometer measuring modulus-of-rupture strength of unfired bars
The results show a predictable decrease in strength as the amount of non-plastic carbonaceous additive is increased. However, the reduction is modest and unlikely to promote an increase in handling damage in their unfired state.

4.6 Fired properties

The firing of these experimental products has been undertaken using three different kilning procedures:

a) Laboratory firing (electric kiln).

b) Factory firing at Hanson Brick’s Desford factory (red-firing): undertaken in w/b 17th Nov. 2008.

c) Factory firing at Hanson Brick’s Desford factory (buff-firing).\(^9\)

The properties of firings a) and b) are compared below. The a) firing took place in a laboratory electric kiln (neutral atmosphere) programmed to the following schedule:

At 50\(^0\)C/hour to 600\(^0\)C (halt for 1 hour for carbon burn-out),
Then 50\(^0\)C/hour to 1065\(^0\)C (soaked for 1 hour),
Kiln switched off and allowed to cool back to ambient.

In contrast the b) firing was undertaken in the Desford tunnel kiln, where the bars were placed on the normal kiln cars set with bricks. These pass through the kiln during a three day cycle reaching a top temperature of (in the current case) 1030\(^0\)C within the firing zone. The results of both firings are compared in Table 5

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\(^9\) The final firing c) to yield a buff product, is scheduled to take place in the Spring of 2009 when the Desford factory is re-opened.
<table>
<thead>
<tr>
<th>Mix codes</th>
<th>% Linear shrinkage (Dry-Fired)</th>
<th>% Water absorption (boiled)</th>
<th>Fired strength (kgf/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>R (Lab. Fired)</td>
<td>0.20</td>
<td>19.1</td>
<td>99.80</td>
</tr>
<tr>
<td>R (Works fired)</td>
<td>0.14</td>
<td>18.9</td>
<td>90.60</td>
</tr>
<tr>
<td>XI (Lab. Fired)</td>
<td>0.23</td>
<td>21.7</td>
<td>85.00</td>
</tr>
<tr>
<td>XI (Works fired)</td>
<td>0.16</td>
<td>20.9</td>
<td>88.66</td>
</tr>
<tr>
<td>XII (Lab. Fired)</td>
<td>0.33</td>
<td>23.6</td>
<td>83.13</td>
</tr>
<tr>
<td>XII (Works fired)</td>
<td>0.30</td>
<td>24.2</td>
<td>73.40</td>
</tr>
<tr>
<td>XIII (Lab. Fired)</td>
<td>0.48</td>
<td>26.2</td>
<td>72.15</td>
</tr>
<tr>
<td>XIII (Works fired)</td>
<td>0.40</td>
<td>27.7</td>
<td>57.31</td>
</tr>
</tbody>
</table>

Table 5: Showing the comparative fired ceramic properties of the laboratory and factory fired test bars

For additional visual comparison, this data has also been produced in graphic form (Fig. 8a-c). The individual results are discussed below.

4.6.1 Firing shrinkage

As previously noted, the mineralogical composition of the Keuper Marl results in a fired product exhibiting little shrinkage until it reaches the top end of its firing range (vitrification point), whereupon rapid fusion and structural collapse rapidly take place. Consequently, when being fired, commercial ware of this kind is purposely kept well below this upper threshold temperature. All the percentage linear firing shrinkages displayed by the test bars as shown in Fig. 8a are below 1%. It is also seen that as the amount of the Didcot PFA in the mix-designs is increased, the level of shrinkage also marginally increases due to incipient fusion. In each case, the shrinkage of the factory fired bars is slightly less than those fired in the laboratory, confirming their lower firing temperature.

4.6.2 Fired water absorption

The percentage water absorption values of Keuper Marl bricks are characteristically high and this is reflected in the results obtained from the test bars (Fig. 8b). A modest trend in increasing values is recorded as the level of addition of Didcot PFA is increased.
Figure 8a: % Linear firing shrinkages of extruded bar mix-designs
(L = University laboratory firing, W = Works firing - Desford factory)

Figure 8b: % Fired water absorption values of extruded bar mix-designs
(L = University laboratory firing, W = Works firing - Desford factory)

Figure 8c: Fired strengths (modulus-of-rupture) of extruded bar mix-designs
(L = University laboratory firing, W = Works firing - Desford factory)
This is a result of the higher amount of the PFA used in these three mix-designs, which range from double to four times the amount of Rossington material used. Consequently, as it burns away during firing, it leaves an increasing number of open pore and void spaces capable of being filled with water and thereby increasing the level of water absorption. The slightly higher firing temperature of the laboratory fired products is reflected in the marginally lower values recorded for the factory fired set of bars. The increasing heat-work has promoted a higher level of shrinkage and consequently a 'tightening-up' of the microstructure, resulting in a reduction in pore volumes.

4.6.3 Fired strength

The slight decreases in the fired strengths of the test bars containing progressively greater levels of Didcot PFA (Fig. 8c) are due to the higher level of pore spaces created by the burning-out of the carbonaceous matter present. A ceramic product with a higher internal ratio of voids-to-solids will normally produce a lower fired strength. This trend is shown to continue to decrease as the level of Didcot PFA is increased.

4.6.4 Fired colour development

A dramatic difference in the fired colour of the two parallel sets of bars is shown in Fig. 9.

![Image of comparison between bars fired in laboratory electric kiln and Desford Brickworks gas-fired tunnel kiln](image-1)

Figure 9: Showing comparative colours of the sets of bars fired in the laboratory electric kiln (left) and through the Desford Brickworks gas-fired tunnel kiln (eight).

The rather 'anemic' pale-pink produced in the laboratory electric kiln contrasts markedly with the red and speckled appearance achieved with the same experimental products when fired through the Desford tunnel kiln. It should be noted that the firing schedule in place at Desford while these bars were being fired was the 'red' product. This is slightly lower temperature firing than the 'buff' product and also less reduction takes place in the kiln.
5.0 Conclusions of laboratory investigation

The two laboratory investigations carried out revealed similar patterns of properties results.

In the case of the initial disc test-piece undertaking, equivalent heat-value additions of the two alternative carbonaceous materials show that increasing the addition amount brings about a gradual reduction in physical properties when compared against the 100% Keuper Marl control (Figs. 4a-f).

Fired properties of shrinkage, water absorption and strength are frequently used by brick manufacturers as a yardstick for ‘new’ products by comparing them against their standard factory products. The second laboratory investigation involved replicating the forming method used at Hanson’s Desford brickworks (extrusion wire-cut). When the two carbonaceous additives were introduced on a like-for-like c/v basis, the results (Figs. 8a-c) marginally favour the product containing 5% w/w Rossington coal fines. However, the performance of this mix-design, when compared with the mix-design containing 15% w/w Didcot high-carbon PFA is relatively small.

In consideration of the market requirements for bricks of the kind being manufactured at Desford brickworks, the physical properties of a product containing 15-20% of Didcot high-carbon PFA would seem to fall well within the specifications demanded.

It should also be noted that there are a number of additional benefits of using PFA in brick manufacture. But in this same context, it should also be noted that many such benefits, only come into their own, if the ‘host’ clay brick body contains moderate to high levels of this material. The following brief information is therefore of only of limited advantage for use in bricks (such as those manufactured at the Desford brickwork) where a relatively small amount (15% w/w) is contemplated.

The potential benefits available if used in conjunction with a wide range of brick making clays, are highlighted below. The evidence is based on the cumulative (well documented) findings of extensive earlier work carried out by organizations including BRE, CERAM Research and Staffordshire University. The following brief listing will hopefully provide useful pointers in the event of growing environmental pressures encouraging the use of increasing amounts of PFA (and other industrial by-products) within the future UK brick industry.

1) Improvement in ‘safe’ drying speeds offering the benefit of reduced energy input.
2) Faster firing times with significant energy saving potential
3) Lower kiln emissions than traditional clay bricks
4) Improved frost-resistance to brick clays (such as the Keuper Marl) which are traditionally vulnerable to damage of this sort.
5) Lighter weight products allowing cheaper transport than normal clay bricks.
6) More thermally insulating than traditional clay bricks.
7) Promoting raw materials sustainability by partially replacing virgin clays.
REFERENCES
