

Effect of borate addition on the sintered properties of pulverised fuel ash

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Abstract

The effect of borate addition on the properties of sintered pulverised fuel ash (PFA) has been investigated. PFA from a major coal-fired power station in the UK has been formed into monolithic ceramic samples using dry powder compaction and sintering. Borax pentahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$), anhydrous borax ($\text{Na}_2\text{B}_4\text{O}_7$) and boric acid (H_3BO_3) were individually added to the PFA and the effects of firing between 1000 and 1200 °C have been investigated. Physical properties (density, water absorption and linear shrinkage), mechanical properties (bending strength and Vickers hardness), phase composition (X-ray diffraction) and sintered microstructure (scanning electron microscopy) are reported. Milling PFA containing 8% (by weight) of borax pentahydrate, to an average particle size of 7.25 μm and sintering at 1130 °C for 1 h, produces ceramics with properties comparable to those of commercial unglazed ceramic wall and floor tiles. The results indicate that PFA could be used to produce commercial ceramics using an economic manufacturing process, although current formulations display excessive shrinkage.

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1. Introduction

Coal-fired power stations are an important source of energy in many parts of the world. Coal combustion products include pulverised fuel ash (PFA), boiler slag and furnace bottom ash. In Europe the amount of coal combustion products (CCPs) produced in 2003 totalled 65 million tonnes, 68% of which was PFA. Pulverised fuel ash (PFA) is the fine, predominantly inorganic particulate residue that remains after pulverised coal is burnt [1].

Coal is used to generate approximately 33% of electricity in the UK and this produces 5.5 million tonnes of PFA annually. Approximately 50% of PFA in the UK is utilized while the remainder is disposed of in ash repositories or landfills at a current cost of approximately 30 euros per tonne (2005) excluding landfill tax [2]. PFA disposal is therefore a significant economic and environmental burden and there is an increasing

need to develop novel, economically viable and environmentally sound alternative reuse applications.

PFA is currently used mainly in concrete, autoclaved aerated concrete blocks, as a raw material for cement production and for the manufacture of lightweight aggregate. It has also been used in road construction, fill and ground remediation, grouting, as a filler in plastics, paints and rubber, and as an insulating material in doors and fire break walls [1–7]. Extensive research has investigated the use of PFA in glass-ceramics [8–12] and some work has also been completed on ceramic tile manufacture using PFA [13–15]. Potential additives to modify the sintering properties of PFA have also been investigated such as bentonite, waste glass and calcium and sodium carbonates [16–19]. PFA and borax solid waste have also been used at low levels to replace potassium feldspar in commercial tile bodies and gave promising results [14].

This paper reports on the effect of the addition of borates on the properties of sintered PFA. Borates are inorganic salts of boron formed by combination with oxygen and other elements. They are used in a wide range of applications in agriculture, detergents, wood treatment, fibreglass, glass and ceramics. Borates act as a flux and inorganic binder in tile bodies,

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increasing the dry mechanical strength and reducing the firing temperature, resulting in energy savings [20].

Systematic experiments have been completed using conventional ceramic processing technology based on powder pressing and sintering. The physical/mechanical properties, sintering behaviour and microstructural characterisation of PFA/borate samples are reported. Potential applications of the novel products as floor or wall tiles are discussed.

2. Experimental methods

2.1. Materials

2.1.1. Pulverised fuel ash

PFA was obtained from the Didcot A power station in Oxfordshire, UK. This is a 2000 MW coal fired power station that is currently using biomass (sawdust) to replace up to 5% by weight of coal, obtained from various sources worldwide. Most UK coal-fired stations, including Didcot A, burn hard or bituminous coals [2].

Table 1 shows the composition of PFA from Didcot A determined by XRF. The total weight percentage of silica, alumina and iron oxides in the PFA exceeds 70%, and the SO₃ content is less than 5% and therefore according to the American Society for Testing and Materials specification (ASTM C618-92a) the PFA from Didcot A is classified as a class F fly ash. The carbon content, indicated by the loss on ignition, is relatively high at 15.8 wt.%.

2.1.2. Borates

The inorganic borates used in this work were borax pentahydrate, Na₂B₄O₇·5H₂O (*Neobor*), anhydrous borax, Na₂B₄O₇ (*Dehybor*) and boric acid, H₃BO₃ (*Optibor*), and they were supplied by U.S. Borax Inc. [20].

Table 1
Analysis (weight% oxides) of PFA from Didcot A

| Oxides | wt.% |
|--------------------------------|-------|
| SiO ₂ | 43.48 |
| Al ₂ O ₃ | 27.26 |
| Fe ₂ O ₃ | 2.33 |
| CaO | 5.49 |
| MgO | 1.51 |
| K ₂ O | 0.57 |
| Na ₂ O | 0.15 |
| TiO ₂ | 1.37 |
| Cr ₂ O ₃ | 0.02 |
| Mn ₃ O ₄ | 0.04 |
| BaO | 0.24 |
| ZnO | 0.01 |
| PbO | <0.02 |
| P ₂ O ₅ | 1.17 |
| ZrO ₂ | 0.07 |
| HfO ₂ | <0.01 |
| CuO | 0.01 |
| SnO ₂ | <0.01 |
| SrO | 0.23 |
| SO ₃ | 0.48 |
| LOI | 15.79 |

Borax pentahydrate (Na₂B₄O₇·5H₂O) is a white odourless crystalline solid. It is not combustible, has a melting point of 200 °C and a specific gravity of 1.81. At 20 °C it has solubility in water of 3.8% and loses water when heated to form anhydrous borax (Na₂B₄O₇).

Anhydrous borax (Na₂B₄O₇) is a white odourless crystalline solid with a melting point of 742 °C and a specific gravity of 2.37. It is not flammable and has water solubility at 25 °C of 3.1%. It is a stable product but when wetted reacts exothermically to form hydrated borates.

Boric acid (H₃BO₃) is a white odourless, crystalline solid with a specific gravity of 1.51 and a melting point of 170.9 °C. It is not combustible and has water solubility at 20 °C of 4.7%. When heated it loses water, first forming metaboric acid (HBO₂) and on further heating converts to boric oxide (B₂O₃).

2.2. Production of sintered samples

Sintered PFA samples were produced using conventional powder processing methods for producing ceramics involving wet ball milling, drying, granulating, pressing and sintering. Initial experiments investigated the effect of 2% (by weight) additions of borates on as-received (unmilled) PFA. In subsequent experiments 200 g batches of PFA containing different levels of borates have been wet milled for various times in a polypropylene ball mill containing alumina milling media, with a water/solids ratio of 1. Milled slurries were oven-dried for 24 h at 105 °C and the dried cake was crushed to pass through a 500 μm sieve prior to pressing.

Cylindrical ‘green’ samples (20 mm diameter, approximately 5 mm high) were uniaxially pressed at 39 MPa using a steel die and hydraulic press. The same pressing pressure was used in all the experiments and at least three disc samples were prepared and fired for each test condition reported.

Samples were sintered at temperatures between 1000 and 1200 °C using a ramp rate of 10 °C min⁻¹ and a dwell time at the maximum temperature of 60 min. Samples were then allowed to cool to room temperature with the furnace.

2.3. Test methods

The particle size distribution of as-received PFA and PFA milled for 2, 4, and 6 h were determined using laser diffraction (Malvern Mastersizer particle size analyser). Fired density (ρ) was calculated using Archimedes’ principle and the linear shrinkage was calculated from the reduction in the sample diameter after sintering. The water absorption of samples was determined using the European Standard test method (EN 99) for determining water absorption of ceramic tiles.

A double-beam optical dilatometer (Misura 3 Optical Dilatometer, manufactured by Expert System Solutions) was used to analyse the sintering behaviour of 15 mm × 5 mm × 5 mm samples pressed at 39MPa using a ramp rate of 25 °C min⁻¹ up to a maximum temperature of 1600 °C.

Vickers hardness was determined using a Zwick/Roell ZHV micro hardness tester by applying a 2 Kg load for 10 s [21]. Three-point bend strength was determined on bar samples

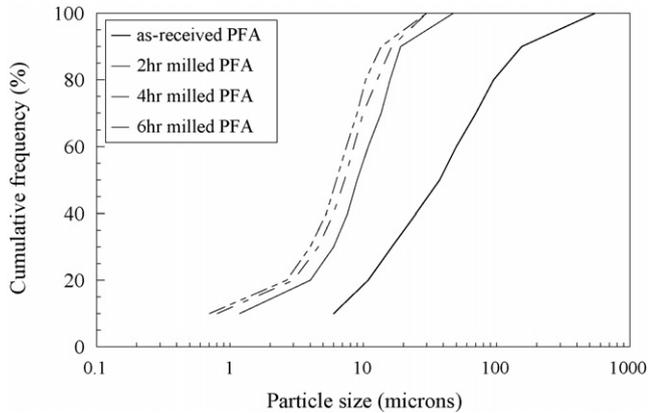


Fig. 1. Particle size analysis for as-received and milled PFA.

(50 mm × 4 mm × 4 mm) cut from rectangular plates prepared and sintered under identical conditions to the disc samples.

X-ray diffraction (XRD, Philips PW1700 series automated powder diffractometer) using Cu K α radiation with a secondary graphite crystal monochromator was used to analyse the crystalline phases present in as-received and sintered PFA samples. The microstructure of gold-coated sintered samples polished to 1 μ m was characterised by scanning electron microscopy (SEM, JEOL JSM-840A).

3. Results

3.1. Particle size distribution

Particle size distribution data for the as-received and milled PFA samples is given in Fig. 1. The as-received PFA had a mean particle size of 37.5 μ m and a fairly wide particle size range. The particle size distribution of milled PFA becomes much narrower and the mean particle size progressively reduces with increasing milling time. Milling PFA for 4 h reduced the mean particle size to approximately 9 μ m.

3.2. Sintered properties of as-received PFA

Sintered density data of samples made from as-received and 4 h milled PFA is shown in Fig. 2. The greater surface area of the milled PFA sample significantly increases the extent of

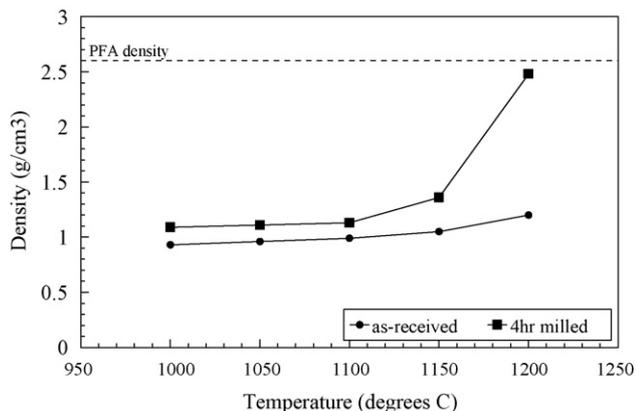


Fig. 2. Effect of 4 h milling on the sintered density of PFA.

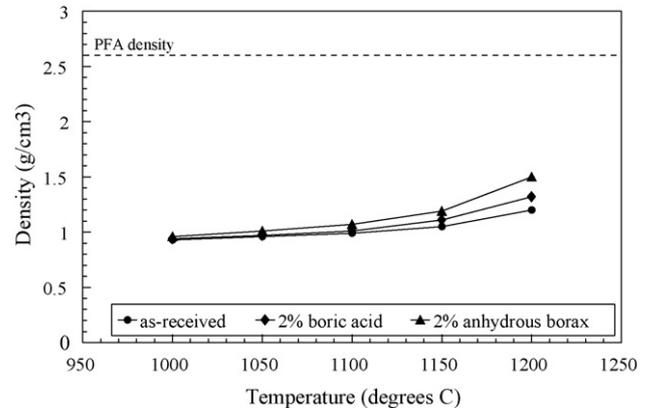


Fig. 3. Effect of borate addition on the fired density of as-received PFA.

sintering at temperatures greater than 1100 °C. The theoretical maximum density of sintered PFA is difficult to define accurately, but a reasonable estimate of the true density of the reactive part of the material is reported to be 2.50–2.60 g cm⁻³ [22]. Therefore the maximum density obtained for the 4 h milled PFA sample sintered at 1200 °C corresponds to a calculated porosity of approximately 3.9 volume%. The density of approximately 1.00 g cm⁻³ for the unmilled PFA sintered at 1200 °C corresponds to a sample with 61.5 volume% porosity.

3.3. Effect of the addition of different borates to PFA

The effect of adding 2% (by weight) by dry mixing different borates to the as-received PFA is shown in Fig. 3. Only relatively minor effects are observed and all the samples were ‘chalky’, poorly sintered and exhibited high water adsorptions ranging from 25 to greater than 50%. Anhydrous borax (Na₂B₄O₇) had a greater effect on density than boric acid probably because the amount of flux (boron oxide, B₂O₃) in a given amount is greater (68.5–69.4% compared to 56.25–57.0%) [18,19]. Anhydrous borax also contains some soda (Na₂O) which is an excellent flux in silicate systems. Addition of borax pentahydrate (Na₂B₄O₇·5H₂O) prepared by the dry powder mixing method for unmilled PFA caused samples to disintegrate during heating to the sintering temperature due to “puffing” of the borate on dehydration.

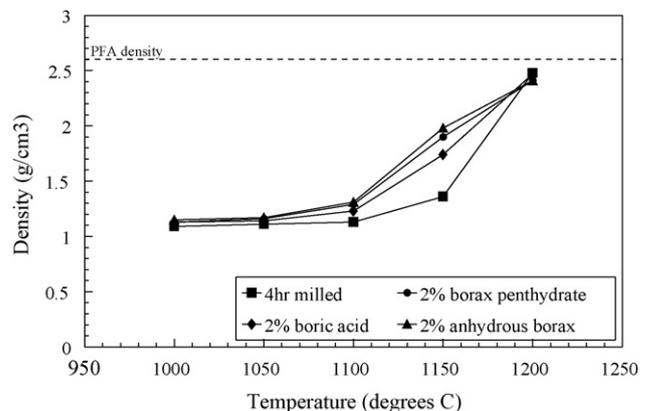


Fig. 4. Effect of different borates on the fired density of 4 h milled PFA.

The effect of 2% (by weight) of the different borates added to the PFA prior to milling is shown in Fig. 4. Adding the borate prior to milling ensures a homogeneous distribution within the PFA. The density of sintered samples increased with borate addition and the milling and subsequent oven drying allowed samples containing borax pentahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$) to be produced without showing any of the cracking associated with dry mixed samples. However at this level of 2% by weight addition the effects were relatively minor.

3.4. Effect of level of borate addition on sintered PFA

Fig. 5 shows fired density, water absorption and shrinkage data at a range of temperatures for 4 h milled PFA with

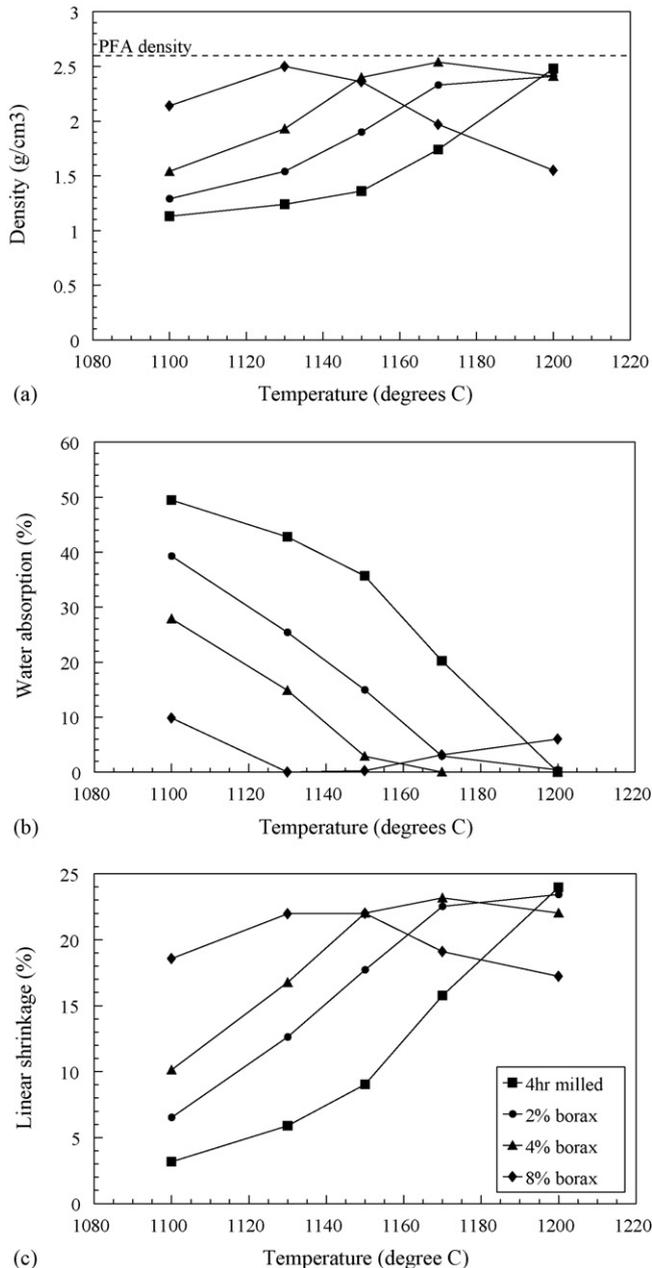


Fig. 5. Effect of different additions of borax pentahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$) on 4hr milled PFA: (a) sintered density; (b) water absorption ad; (c) linear shrinkage.

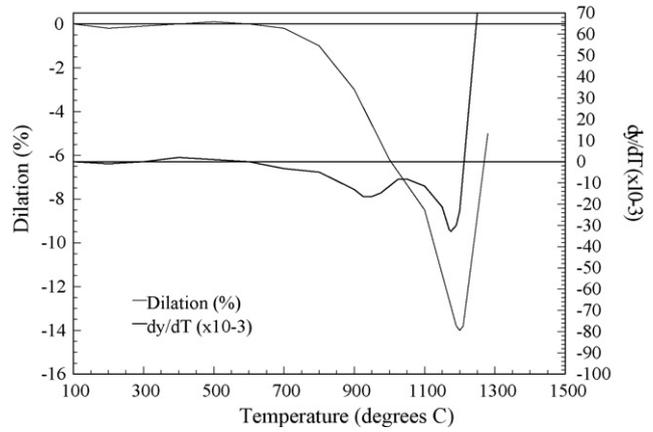


Fig. 6. Dilation (y) and dilation rate (dy/dT) for 4 h milled PFA/8% borax pentahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$), obtained by optical dilatometry.

increasing borax pentahydrate additions. The density curve is significantly shifted to lower temperatures with increased borate addition and higher densities were achieved at significantly lower temperatures. For samples prepared using 8% (by weight) borax pentahydrate, the density reached a maximum of 2.5 g cm^{-3} at $1130 \text{ }^\circ\text{C}$. Assuming the addition of borates does not significantly affect the theoretical maximum density of sintered PFA (2.6 g cm^{-3}), then the maximum density of 2.5 g cm^{-3} corresponds to a sample with approximately 3.8 volume% porosity. Sintering at temperatures above $1130 \text{ }^\circ\text{C}$ caused samples to exhibit a bloating effect associated with increased porosity and reduced density. Water absorption values (Fig. 5b) of samples with high borate content were also significantly reduced and samples made containing 8% (by weight) borax pentahydrate in the mix showed negligible water absorption when sintered at $1130 \text{ }^\circ\text{C}$. Comparable density and water absorption properties can be achieved for PFA containing no borate addition by sintering at $1200 \text{ }^\circ\text{C}$. Fig. 5c shows that high shrinkage values are observed, particularly for samples with high borate contents at all the sintering temperatures used and this is related to the low green density of pressed samples.

3.5. Optical dilatometry

Fig. 6 shows typical data obtained for dilation (shrinkage) and dilation rate against temperature for 4 h milled PFA containing 8% borax pentahydrate. The dilation curve clearly shows the bloating effect that occurs when samples are heated beyond the optimum sintering temperature. This technique

Table 2

Temperatures at which maximum shrinkage and maximum shrinkage rate occur for PFA samples containing different additions of borax pentahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$)

| Borax pentahydrate addition (weight%) | Temperature at maximum shrinkage rate dy/dT ($^\circ\text{C}$) | Temperature at maximum shrinkage ($^\circ\text{C}$) |
|---------------------------------------|--|---|
| 0 | 1250 | 1290 |
| 2 | 1230 | 1270 |
| 4 | 1210 | 1240 |
| 8 | 1180 | 1200 |

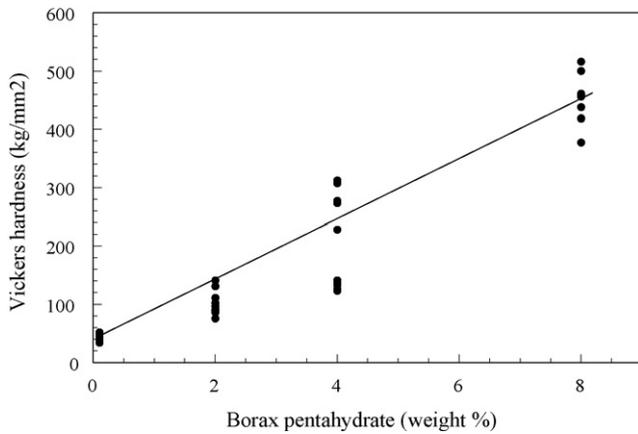


Fig. 7. Vickers hardness test data for sintered PFA samples made using different additions of borax pentahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$).

indicates the temperatures at which maximum dilation rate and maximum dilation occur. Results from the optical dilatometry testing of 4 h milled PFA containing increasing borax pentahydrate contents are summarised in Table 2. Under the rapid sintering conditions used in the dilatometer the temperature at maximum dilation rate decreases from 1250 to 1180 °C as the borax pentahydrate content in the mix increases from 0 to 8% (by weight).

3.6. Physical property testing

Vickers micro hardness test results for samples containing different amounts of borax pentahydrate fired at 1130 °C are shown in Fig. 7. To obtain reliable data 10 indentations were made on polished surfaces of each sample. The results clearly indicate that Vickers micro hardness of samples increases with increasing borax pentahydrate content. The highest value of Vickers micro hardness of 4.41 GPa was obtained for the sample made using 8% borax pentahydrate.

Three-point bending strength results are given in Fig. 8. Data points are based on testing five specimens. The strength clearly increases with increased borate content and the average strength for the 4% borax pentahydrate PFA samples was

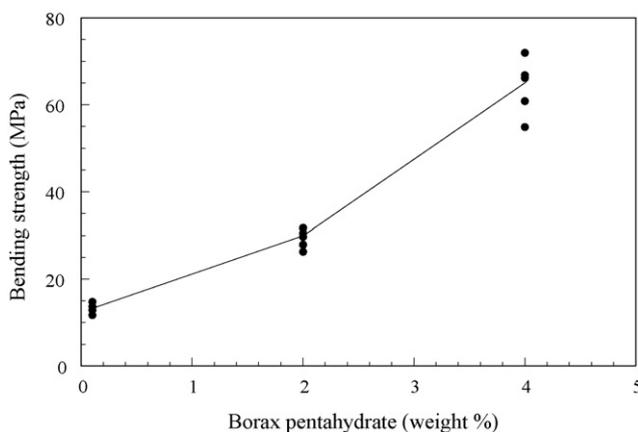


Fig. 8. Three point bend strength data for sintered PFA samples made using different additions of borax pentahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$).

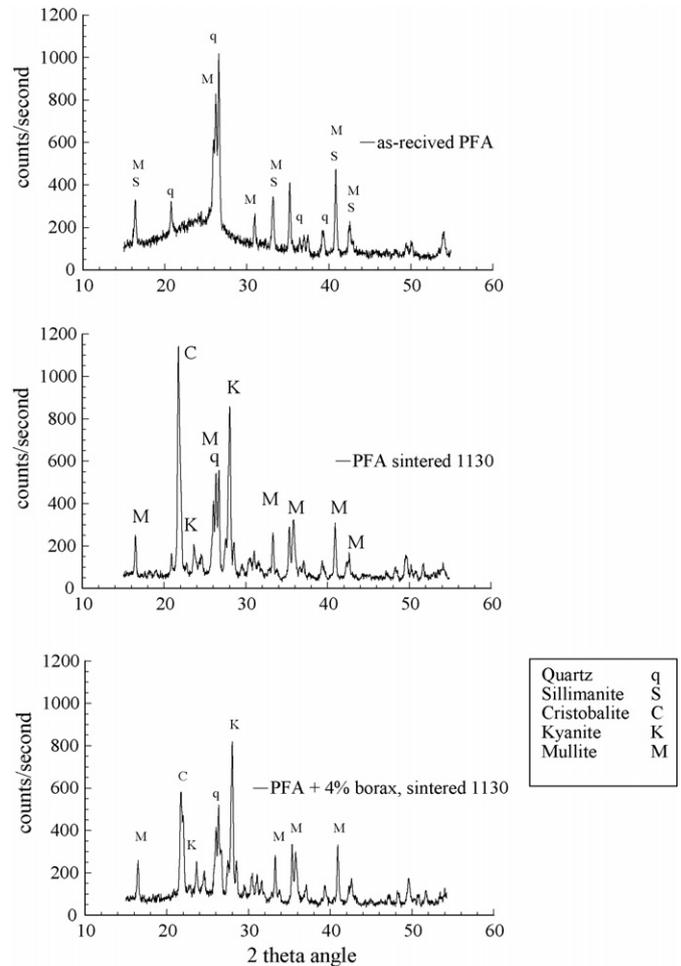


Fig. 9. X-ray diffractogram for as-received PFA and PFA samples sintered at 1130 °C.

64.1 MPa. Sintered PFA samples made with 8% borax pentahydrate were not tested because of defects on the surface of the samples that occurred due to bloating during sintering.

3.7. Microstructural analysis

XRD data is presented in Fig. 9. This shows that the major crystalline phases in as-received PFA are quartz (SiO_2), mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$) and sillimanite (Al_2SiO_5). When PFA was fired to 1130 °C, the amount of quartz decreases and cristobalite (SiO_2) and kyanite ($\text{Al}_2(\text{SiO}_4)\text{O}$) are formed. The XRD background level for sintered samples at 2θ angles between 15 and 25° was less than for the as-received PFA, and this indicates that sintered samples contained a significantly reduced amount of amorphous glassy phase. The addition of borax pentahydrate to the milled PFA resulted in sintered samples with reduced cristobalite and quartz, while the amount of mullite remained approximately constant.

Secondary electron images obtained by SEM of samples with varying proportions of borax pentahydrate fired to 1130 °C are shown in Fig. 10. These indicate a general decrease in the porosity with increasing borax pentahydrate content. This is in agreement with the density, water absorption, hardness and strength test data.

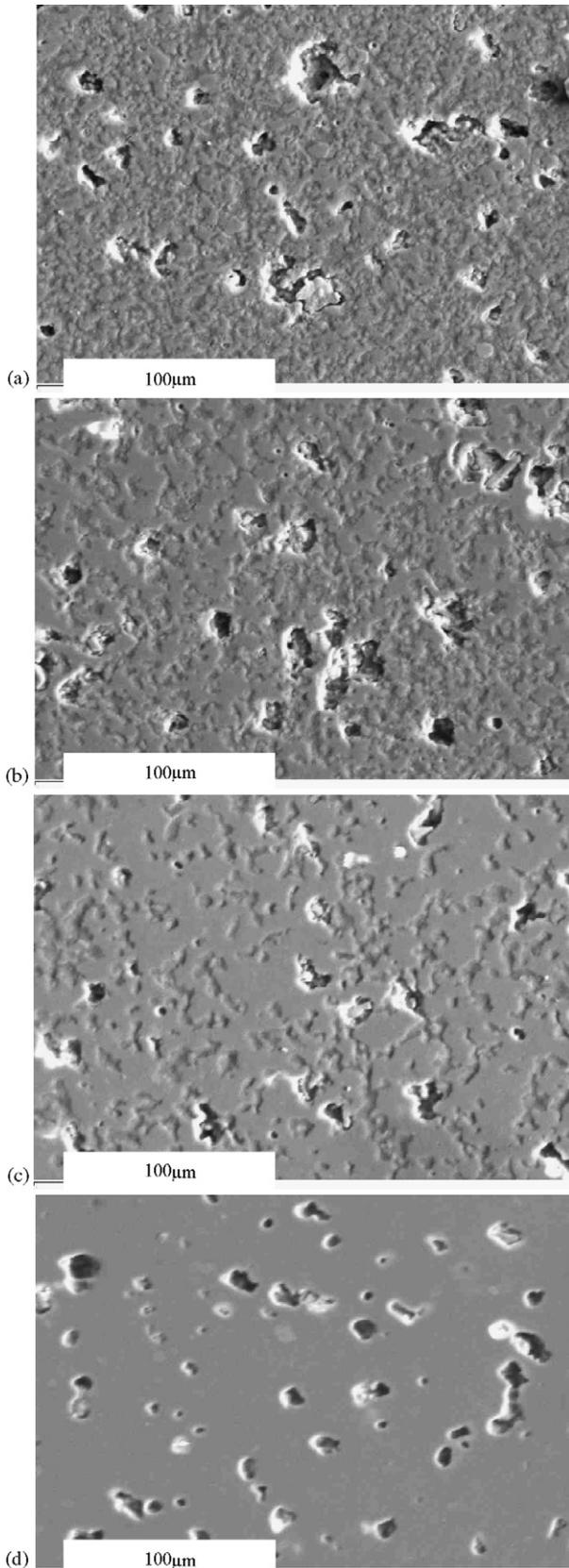


Fig. 10. Secondary electron images of polished surface of sintered PFA sample fired at 1130 °C containing different amounts of borax pentahydrate: (a) 0%; (b) 2%; (c) 4%; and (d) 8% (by weight).

4. Discussion

The properties of PFA ceramics clearly depend on the processing conditions, borate addition and the sintering temperature. To obtain potentially viable materials from PFA without any borate addition requires samples to be sintering at approximately 1200 °C. However the addition of between 4 and 8% by weight borax pentahydrate during milling reduces the optimum sintering temperature by at least 70 °C to approximately 1130 °C. This reduction in sintering temperature represents a significant saving in terms of equipment cost, energy and overall environmental impact of the manufacturing process.

Sodium borate is well-known as a good flux in silicate systems. The addition of sodium borate to these systems initiates the formation of a low viscosity melt at low temperatures, which facilitates sintering at a reduced temperature by enabling mass transport mechanisms to occur more easily. The phase diagram for the $\text{Na}_2\text{O}\cdot\text{B}_2\text{O}_3\cdot\text{SiO}_2$ system has a eutectic at a temperature below 600 °C and therefore in silicate systems containing sodium borate, the sintering process starts much earlier than in the same system without borate.

The results obtained in this study are particularly encouraging, as they show that that milled PFA can be sintered to produce a ceramic body with properties such as density, water absorption and strength similar or superior to those of commercially available wall and floor tile bodies. Table 3 provides summary data comparing sintered PFA ceramics with those produced from conventional raw materials. This also indicates that although the sintered PFA body has comparable physical properties to other commercial ceramic tile bodies the current PFA formulation and processing produces a body that exhibits excessive shrinkage during firing. This is a major practical difficulty that needs to be overcome as high shrinkage is normally associated with deformation and warping during firing. Ongoing research is currently addressing this issue.

The research highlights the potential for using PFA to form new types of ceramic products. This would significantly widen the applications for PFA, which remains a major European waste. However PFA disposal problems are also a major issue in many other countries. The largest producers of PFA are China and India, both of which are rapidly increasing coal fired power generation rates as they become increasingly economically competitive. These experiments show that the properties of sintered PFA can be comparable with those of standard commercial ceramic materials that are used for floor and unglazed wall tiles. There is an enormous demand for these types of products in many parts of the world and it is clear that ceramic products manufactured using PFA combined with borates have significant potential. However it is also clear that more research and development work is needed to optimise the PFA ceramic body and the manufacturing process. In addition pilot plant scale trials are necessary to assess potential commercialisation of the research reported in this paper.

Table 3
Comparison of the properties of sintered PFA and commercial ceramic tile bodies

| Property | Porcelain | White body | Red body | Monoporosa | PFA + 8% borax pentahydrate fired 1130 °C |
|--|-----------|------------|----------|------------|---|
| Water adsorption (%) | <0.5 | 1–3 | 3–6 | <12 | <0.5 |
| Shrinkage (%) | 7–8 | 6–7 | 5–6 | 0–1 | 21 |
| Fired density (g cm ⁻³) | 2.40 | 2.35 | 2.35 | 1.80 | 2.50 |
| Typical pressing pressure (kg cm ⁻²) | 300–450 | 250–300 | 150–200 | 200–250 | 400 |

5. Conclusions

- Conventional dry powder pressing manufacturing methods for ceramics can be used to produce high density and low porosity ceramics using pulverised fuel ash (PFA) obtained from coal-fired power stations.
- The addition of borates to PFA prior to milling improves the physical and mechanical properties of sintered PFA and significantly reduces the firing temperature. Optimum materials with respect to density, water absorption, strength and hardness were achieved by firing PFA samples, prepared using 8% by weight of borax pentahydrate, at 1130 °C for 1 h.
- The physical and mechanical properties of samples made from PFA and borax pentahydrate are comparable to those of commercial unglazed wall and floor tiles. PFA has potential to be exploited as a low-cost raw material for the commercial production of ceramic tiles. However, excessive shrinkage during firing is a problem and more work is needed to further optimise the PFA body and firing cycle.

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