



# Surface and bulk characterization of an ultrafine South African coal fly ash with reference to polymer applications



E.M. van der Merwe<sup>a,\*</sup>, L.C. Prinsloo<sup>b</sup>, C.L. Mathebula<sup>a</sup>, H.C. Swart<sup>c</sup>,  
E. Coetsee<sup>c</sup>, F.J. Doucet<sup>d</sup>

<sup>a</sup> Department of Chemistry, University of Pretoria, Lynnwood Road, Pretoria 0002, South Africa

<sup>b</sup> Department of Physics, University of Pretoria, Lynnwood Road, Pretoria 0002, South Africa

<sup>c</sup> Department of Physics, University of the Free State, P.O. Box 339, Bloemfontein 9300, South Africa

<sup>d</sup> Mineral Waste Beneficiation Group, Council for Geoscience, 280 Pretoria Road, Private Bag X112, Pretoria 0001, South Africa

## ARTICLE INFO

### Article history:

Received 18 June 2014

Received in revised form 13 August 2014

Accepted 13 August 2014

Available online 21 August 2014

### Keywords:

Coal fly ash

Bulk properties

Surface properties

Characterization

## ABSTRACT

South African coal-fired power stations produce about 25 million tons of fly ash per annum, of which only approximately 5% is currently reused. A growing concern about pollution and increasing landfill costs stimulates research into new ways to utilize coal fly ash for economically beneficial applications. Fly ash particles may be used as inorganic filler in polymers, an application which generally requires the modification of their surface properties. In order to design experiments that will result in controlled changes in surface chemistry and morphology, a detailed knowledge of the bulk chemical and mineralogical compositions of untreated fly ash particles, as well as their morphology and surface properties, is needed. In this paper, a combination of complementary bulk and surface techniques was explored to assess the physicochemical properties of a classified, ultrafine coal fly ash sample, and the findings were discussed in the context of polymer application as fillers. The sample was categorized as a Class F fly ash (XRF). Sixty-two percent of the sample was an amorphous glass phase, with mullite and quartz being the main identified crystalline phases (XRD, FTIR). Quantitative carbon and sulfur analysis reported a total bulk carbon and sulfur content of 0.37% and 0.16% respectively. The spatial distribution of the phases was determined by 2D mapping of Raman spectra, while TGA showed a very low weight loss for temperatures ranging between 25 and 1000 °C. Individual fly ash particles were characterized by a monomodal size distribution (PSD) of spherical particles with smooth surfaces (SEM, TEM, AFM), and a mean particle size of 4.6 μm (PSD). The BET active surface area of this sample was 1.52 m<sup>2</sup>/g and the chemical composition of the fly ash surface (AES, XPS) was significantly different from the bulk composition and varied considerably between spheres. Many properties of the sample (e.g. spherical morphology, small particle size, thermal stability) appeared to be suitable for its applicability as filler in polymers, although the wide variation in surface composition between individual particles may challenge the development of a suitable surface modification technique. The observation that the bulk and surface compositions of the particles were so intrinsically different, strongly suggested that surface characterization is important when considering compatibility between matrices when applying fly ash as filler in polymers.

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## 1. Introduction

Coal fly ash (FA) is one of the main residues produced during the combustion of pulverized coal in thermoelectric power stations. It originates from the lighter particles ascending with the flue gases during coal combustion, and is collected by electrostatic precipitators or bag filter systems. After precipitation, collected FA is safely

disposed of and managed on ash dumps or slurry dams; or it can be utilized in a number of applications. FA is usually classified by particle size, creating a relatively uniform fine gray powder of which the particles are predominantly spherical in shape. Depending on the source and type of coal being combusted, the FA composition may differ considerably, but its chemical nature remains predominantly glassy or amorphous [1].

The main source of power generation in South Africa is coal-fired thermoelectric power stations, which currently produce about 25 million tons of FA per annum [2]. Approximately 5% of the produced FA is reused, mainly as cement extender and in the production of

\* Corresponding author. Tel.: +27 12 420 5379; fax: +27 12 420 4687.  
E-mail address: [liezel.vandermerwe@up.ac.za](mailto:liezel.vandermerwe@up.ac.za) (E.M. van der Merwe).

concrete. In addition, SASOL Synfuels generates an additional 4 million tons of fine ash per annum [3]. The extensive amounts of FA that is produced each year pose significant environmental and economic threats due to the great need of environmentally safe and economically affordable ways of disposal and handling. There is a growing concern about pollution and increasing landfill costs and therefore there has been global interest in the utilization of FA for economically beneficial applications.

South Africa has a long history regarding the development of new applications for FA. The beneficiation of South African FA after it has been put through a process of air classification, electrostatic recovery and/or density separation has been described before [1], and specific size fractions of FA, with specific physical and chemical properties, may be used in suitable applications. The country's research and development involving FA includes its application in building and construction as cement extender and into lightweight aggregates [4], environmental rehabilitation in the form of counteracting acid mine drainage, wastewater treatment, and toxic element immobilization by zeolites [5–7], mine backfilling, the recovery of alumina [8,9], production of geopolymers [10,11] and hydrotalcites [12], soil amelioration [13], refractories and bricks, road stabilization, CO<sub>2</sub> capture [14], and as a processing aid and functional filler in polymers [15].

In order to take advantage of FA utilization efficiently, an accurate characterization of the chemical composition and mineralogy of a FA sample from a specific origin needs to be performed [16]. Furthermore, if the targeted applications require surface modification of FA, the properties of its surface need to be characterized appropriately.

Very few studies have specifically described the characterization of South African FA samples [2,17,18], and due to differences in origin and method of classification and/or separation, the physical and chemical properties of FA products will vary. Furthermore, the industrial application of FA will dictate which physical and chemical properties need to be characterized.

In order to understand the processes that can enhance the properties of FA for its industrial application into polymers, a detailed study of the surface, chemical and morphological properties of FA is necessary before experimentation according to scientific methods can take place. Apart from controlling the mechanical properties (e.g. toughness, stiffness, and strength) of polymer composites, the viscosity of the uncured mixture must be low enough to improve workability and permit the evacuation of air bubbles. Studies performed by Roulin-Moloney et al. [19] and Nakamura et al. [20] have shown that increasing the particle size of spherical silica particles in a silica-filled epoxy resin have a detrimental effect on the strength of the obtained composite. The mean particle size range studied by Roulin-Moloney was between 60 and 300 μm, while that of Nakamura ranged between 6 and 42 μm. On the other hand, the same authors have noted that although the use of filler particles with sizes less than 4.5 μm may improve the strength of the composite, a practical limit is enforced by the viscosity of the mixture. Smaller filler particles have a greater surface area and may thus considerably increase the viscosity of the uncured filler–polymer mixture. It is therefore clear that particle size will have a considerable effect on the properties of polymer–filler composites, and this effect will be material-specific. Due to the increased mechanical properties obtained upon decrease in filler particle size, an ultrafine coal fly ash sample was chosen for the purpose of this study.

In this paper, a comprehensive combination of complementary characterization techniques was used to determine the chemical composition, mineralogy and surface properties of a classified South African FA sample. These results serve as a strong foundation in our ongoing project where we are designing and testing dry and wet coating procedures aimed at modifying the surface properties

of FA particles according to specific requirements imposed by our targeted applications.

## 2. Materials and methods

### 2.1. Origin and description of the ultrafine fly ash sample

A representative sample of a classified, ultrafine FA sample was obtained from the Ash Resources Pty Ltd.'s ash beneficiation site at Eskom's Lethabo Thermal Power station, which is located between Vereeniging and Sasolburg in the Free State province of South Africa. This commercial-grade FA is classified on site using electrostatic precipitators and is specified to have a mean particle size between 3.9 and 5.0 μm, with more than 90% of the volume distribution of its particles having a diameter smaller than 11 μm. After classification it is marketed as a very fine, spherical, pozzolanic and highly reactive alumino-silicate with a low carbon content. The product currently finds application in the construction industry, but is also used in small quantities in the rubber and polymer industries.

### 2.2. Bulk characterization of the fly ash sample

#### 2.2.1. X-ray fluorescence spectroscopy (XRF) analysis

XRF was used to determine the bulk chemical concentrations of major elements in the FA sample. No milling was required prior to the analyses. The analyses were performed on an ARL9400XP+ XRF spectrometer (Thermo ARL, Switzerland). The loss on ignition (LOI) was determined by roasting the sample at 1000 °C for at least 3 h until a constant weight was obtained. A glass disk was prepared by fusing a mixture of 1 g of the fly ash sample with 6 g of Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> at 1000 °C.

#### 2.2.2. X-ray diffraction (XRD) analysis

The mineralogical composition of the FA sample was obtained by XRD. The XRD patterns were collected from 5° to 90° on a PANalytical X'Pert Pro powder diffractometer with X'Celerator detector and variable divergence and fixed receiving slits with Fe filtered Co-Kα radiation. The phases were identified using X'Pert Highscore plus software. The relative phase amounts were estimated using the Rietveld method (Autoquan Program). Twenty percent silicon (Aldrich 99% pure) was also added to each sample for the determination of amorphous content. The samples were then micronized in a McCrone micronizing mill, and prepared for XRD analysis using a back loading preparation method.

#### 2.2.3. Carbon and sulfur analysis

The total carbon and sulfur content of the FA sample was determined using an Eltra CS 800 Double Dual Range system. Between 50 and 200 mg of sample was weighed into a ceramic crucible. Iron and tungsten chips were added, mixed and then combusted in a stream of oxygen where carbon and sulfur was converted into CO<sub>2</sub> and SO<sub>2</sub> respectively. The concentrations of gasses were detected using four infrared absorption detectors. The instrument was calibrated using certified carbon and sulfur standards, Euronorm-CRM 484-1 Whiteheart malleable iron and Leco No 501-502. The reported result is the average of two measurements.

#### 2.2.4. Thermogravimetric analysis (TGA)

TGA analyses were performed on a Mettler Toledo TGA/SDTA 851e Thermogravimetric Analyzer. Approximately 20 mg of sample was placed in an alumina pan and heated from 25 to 1000 °C at a heating rate of 10 °C/min. Thereafter, the sample was kept isothermally at 1000 °C for 10 min. Air was used as the purge gas at a flow

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