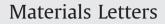
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A simple method for preparing super-hydrophobic powder from paper sludge ash

Charikleia Spathi^a, Neale Young^a, Jerry Y.Y. Heng^b, Luc J.M. Vandeperre^c, Chris R. Cheeseman^{a,*}

^a Department of Civil and Environmental Engineering, Imperial College London, Skempton Building, South Kensington Campus, SW72AZ, London, UK

chemically bonded to fracture surfaces.

^b Department of Chemical Engineering, Imperial College London, Skempton Building, South Kensington Campus, SW72AZ, London, UK

ABSTRACT

^c Department of Materials, Imperial College London, South Kensington Campus, London SW72AZ, UK

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1. Introduction

The European pulp and paper industry generates ~7.7 million tonnes per year of paper sludge [1]. Dewatered paper sludge is often combusted to reduce the amount of material requiring disposal and this produces paper sludge ash (PSA). Currently ~125 ktonnes of PSA are produced each year in the UK and this is a fine, highly alkaline (pH > 12) powder with moisture content less than 0.1% w/ w [2,3]. Approximately 70% of UK PSA is used in low-value applications such as land spreading where it neutralises acid soils [4]. Other uses include as a cattle bedding material and as an additive in waste effluent neutralisation processes. PSA has also been investigated as a potential hydraulic concrete admixture, although the porous PSA particles increase water demand and excess free lime causes durability problems [3,5,6]. There are increasing legislative, economic and social drivers for the development of higher value reuse applications for PSA [2,7].

Hydrophobicity is widely exploited in biological systems as in the self-cleaning mechanism used by super-hydrophobic leaves [8,9]. Water droplets remove particles and pathogens from the leaf, resulting in greater resilience to chemical and biological damage. This effect could provide considerable advantages in many engineering applications. For example, low cost hydrophobic coatings that produce self-cleaning building facades resistant to bio-deterioration, algal growth and general soiling would have significant advantages

* Corresponding author. Tel.: +44 20 75945971.

E-mail address: c.cheeseman@imperial.ac.uk (C.R. Cheeseman).

[10]. Other important uses for hydrophobic coatings include biofouling-resistant ship hulls and marine infrastructure, anti-icing surfaces and corrosion-resistance applications.

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Paper sludge ash (PSA) is a readily available waste material generated by the paper recycling industry.

This work reports on the production of hydrophobic powders by dry milling PSA in the presence of a

fatty acid surface functionalising agent. Optimum laboratory processing involves dry milling for 8 h with

a 4 wt.% addition of stearic acid and this produced a super-hydrophobic powder with a water contact angle of 153°. Different chain length fatty acids were investigated but stearic acid produced the highest

hydrophobicity. The super-hydrophobicity of PSA results from the micro-particulate texture induced by

dry milling with simultaneous formation of calcium stearate self-assembling surface monolayers

PSA has been dry milled in the presence of stearic acid in experiments designed to investigate the use of PSA as a bloating agent in sintered glass products [11]. This produced a hydrophobic powder that could not be formed into pellets when subsequently mixed with water. The optimisation of this effect is reported, with hydrophobicity assessed by measuring the water contact angle (WCA) and the interaction between PSA and stearic acid studied using Fourier transform infrared spectroscopy (FTIR).

2. Experimental

Paper sludge ash was obtained from a major newsprint recycling company operating in Southern England (Aylesford Newsprint Ltd). This facility produces 400,000 t per year of recycled newsprint from 500,000 t of waste paper fibre and produces ~70 ktonnes of PSA. The chemical composition of PSA was determined by X-ray fluorescence spectroscopy (XRF, Spectro 2000 Analyser) and the mineralogical composition of PSA was determined by X-ray diffraction (XRD, Philips PW 1830 diffractometer system) using 40 mA and 40 kV, Cu radiation.

The surface functionalising agents (SFAs) used to produce hydrophobicity were the saturated fatty acids capric acid ($CH_2(CH_2)_8COOH$), myristic acid ($CH_3(CH_2)_{12}COOH$), stearic acid ($CH_3(CH_2)_{16}COOH$) and behenic acid ($CH_3(CH_2)_{20}COOH$). Laboratory production of hydrophobic powders involved dry ball milling 500 g batches of PSA in the







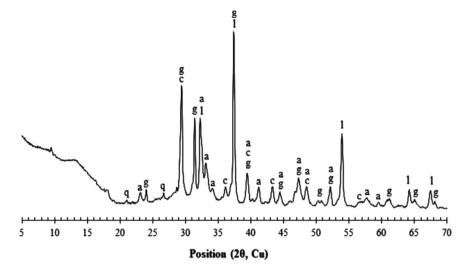


Fig. 1. X-ray diffraction data for as-received PSA indicating that the major crystalline phases present are: a: calcium silicate (Ca₂SiO₄), c: calcite (CaCO₃), g: gehlenite (Ca₂Al (AlSiO₇)), l: lime (CaO) and q: quartz (SiO₂).

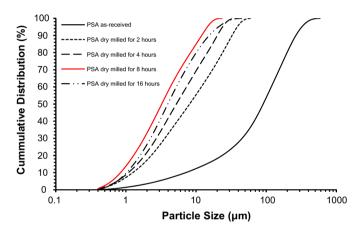


Fig. 2. Effect of milling time on the PSA particle size distribution.

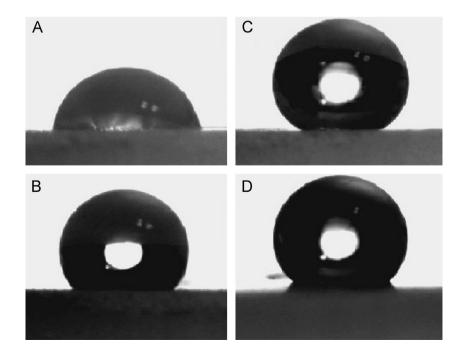


Fig. 3. Images of water droplets on hydrophobic surfaces used to determine the water contact angle (θ_c) by the sessile drop method. The droplets shown are for PSA dry milled for different times with 1 wt.% addition of stearic acid (A, 2 hours; B, 4 hours; C, 8 hours and D, 16 hours).

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