Young Researchers’ Forum in Construction Materials
Thursday 17 May 2012, SCI HQ, London, UK
Organised by SCI’s Construction Materials Group
Editorial: The role of the institutions in construction materials research

Mark Tyrer, Adrian Blacker & John Bensted
Construction Materials Group, Society of Chemical Industry,
14-15 Belgrave Square, London SW1X 8PS
(construction@soci.org)

1 Reduction of cement in dry cementitious mix for paving blocks

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2 Report on current straw usage in Great Britain and future availability for construction

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Andrew Wylie, Buro Happold Ltd., andrew.wylie@burohappold.com
Celia Way, Buro Happold Ltd.
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3 Lime stabilisation of soils; Implications for sulphate-induced heave

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4 Foamed concrete for sustainable construction

Kezban Ozlutas & Roderick Jones
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University of Dundee, Dundee, UK.
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5 Understanding and enhancing the moisture-damage performance of asphalt materials

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6 The effect of dewatering on strength and setting time of hydraulic mortar

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The University of Manchester, Manchester M13 9PL, UK.
(nidhal.aldefai@postgrad.manchester.ac.uk)

7 Protecting the UK’s critical infrastructure – Impressed Current Cathodic Protection on the Midland Links Motorways Network

C. Christodoulou¹, C. I. Goodier², S. A. Austin², J. Webb¹ & G. K. Glass³
¹AECOM Europe, 94-96 Newhall Street, Birmingham, B3 1PB, U.K.
²Loughborough University, School of Civil and Building Engineering, Loughborough, U.K.
³Concrete Preservation Technologies, University of Nottingham Innovation Lab,
Nottingham, UK, NG7 2TU
(christian.christodoulou@aecom.com)
8 The use of ternary binders in self-compacting concrete (SCC)

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University College London, WC1E 6BT, London, UK
(m.shams@ucl.ac.uk)

9 Design for Deconstruction in UK Timber Framed Dwellings. The Contemporary State of Play

Sophie Chisholm, Aedas R&D.
Primary research conducted at Oxford Brookes as part of the MSc SB: PD
(sophie.chisholm@gmail.com)

10 A rapid quantitative determination of polymer modified bitumen storage stability by FTIR

Ian M. Lancaster & Hussain Al-Khalid
Centre for Engineering Sustainability, University of Liverpool
Brodie Tower, Brownlow Street, L69 3GQ
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11 Structural limecrete: an investigation into the potential of hydraulic lime-concrete using pozzolanic and latent hydraulic additions

Ellen Grist, EngD Research Engineer, Ramboll
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12 Magnesium phosphate cement: A new material for waste encapsulation

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13 Compressive Strength, Hydration Products and Microstructure of Alkali-activated Fly Ash Manufacture with Pulsed Microwave Curing Method
Shi Shi¹, Yun Bai¹, Hui Li², P.A.M. Basheer³, Delong Xu²
¹Department of Civil, Environmental and Geomatic Engineering, University College London, Gower Street, London, WC1 6BT, United Kingdom
²Institute of Powder Engineering, Xi'an University of Architecture and Technology, Yanta Road, Xi'an, 710055, P.R. China
³School of Planning, Architecture and Civil Engineering, Queen's University Belfast, Stanmillis Road, Belfast, BT9 5AG, United Kingdom

14 The effect of different addition methods of lignosulfonate admixtures on the adsorption, zeta potential and fluidity of alkali-activated slag binders

Jun Ren¹, Yun Bai¹, Martyn J. Earle² and Changhui Yang³
¹Department of Civil, Environmental and Geomatic Engineering, University College London, London, UK
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Young Researchers’ Forum in Construction Materials

17th May 2012, Society of Chemical Industry, 14-15 Belgrave Square, London SW1X 8PS

The past, present and future chairmen of the Construction Materials Group at the SCI, share a few thoughts on the role of the institutions in the construction materials field and describe how meetings such as this attempt to unite new researchers in the subject.

Editorial

In recent years, the nature of research in construction materials and indeed, most disciplines, has changed markedly. This reflects the changing nature of industry across the world, where operating companies have evolved, often through acquisition and merger, to result in a commercially very competitive marketplace. The supply of materials is dominated by large, often multi-national companies, capable of satisfying the changing demands of the construction industry. Complementing these companies are a multitude of relatively small, specialist manufacturers supplying both end users and other suppliers alike. Contemporary with this in the UK, we have seen the former national laboratories all but disappear; those which remain, operate on a very different funding model to their earlier incarnations. Government funding for research is constrained at present, due to the sluggish economic climate and applications for university funding are subject to fierce competition. This is true at all levels; in national and international funding competitions and in both publically and commercially funded research.

One noticeable consequence is that the experience of early career scientists and engineers has also changed. There is greater incentive to remain focussed and work hard on the individual project, with somewhat reduced freedom to interact with people outside the researcher’s immediate peer group. For example, the network of an individual’s contacts seems less well developed than in the past, fewer researchers have a comprehensive understanding of where expertise and facilities are located and active participation in the institutions and learned societies is at an all time low. To their credit, the universities have a stronger infrastructure in post-graduate training than ever before. This manifests itself in numerous courses in research methods and in related fields such as statistics, intellectual property, publication of research etc. and these are largely of a very high quality.

To complement this, a number of institutions have combined efforts in promoting this meeting to act as a forum on which early career researchers can meet and exchange ideas and also present their work to a friendly audience of their peers. The meeting is deliberately wide in scope – embracing all construction materials – and the programme spans the range of major disciplines, materials and applications. If the event is well received, it is hoped that it will be hosted by another organisation in two years time, but again, co-promoted by many. It is important that this meeting is low in cost and relatively informal. Many presenters will be making their first public presentation whilst other, more established researchers, will seek to hone their presentation skills and hear about work outside their immediate field.
In the welcome and concluding remarks, we will remind delegates of the importance of participating in events – not just passively (as members or attendees) but as active contributors to the field. Those people working in academia are under great pressure to publish in well-respected journals and those working in industry contribute to the patent literature to consolidate their commercial leads. It is important that you also consider the ‘glossy’ magazines, aimed at a wider readership. Editors are usually delighted to receive contributions, from the shortest letter or application note, to large illustrated features. Once your patent or paper is secure – please consider telling your good news story to a wider audience. Reprints of such articles are often very useful to send out, so ask the editor for some copies or permission to reproduce. On a similar topic, most of the committees of the institutions welcome contributions from early career members. As either a research student, or new appointee in industry, you bring a new perspective to the committees and help shape the future of your institutions. If you are interested, ask the Chairman or Secretary if you could attend a committee meeting as an observer. By and large, you will be very welcome. Many committees co-opt members for a short period to help with a particular task, such as organising an event or conducting a review, so you can try your hand before making a longer term commitment.

Finally, consider this. Apart from filling your CV and getting your name known outside your firm or university, your committee work may see you sitting alongside your next employer!

With all good wishes for the future,

Mark Tyrer (Current Chairman)
Adrian Blacker (Chairman Elect)
John Bensted (Immediate Past Chairman)

Construction Materials Group
Society of Chemical Industry

The construction materials group extend thanks to our partners in co-promoting this event
### Welcome

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#### Session 3. Chairman: Adrian Blacker

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#### Session 4. Chairman: Mark Tyrer

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#### Concluding remarks and presentation of awards

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All delegates are welcome to attend the AGM. If you intend to seek nomination to this committee, please speak to one of the chairmen during the day.
Reduction of cement in dry cementitious mix for paving blocks
Ghassan Jalull, Eshmaiel Ganjian and Homayoon Sadeghi-Pouya
Department of Civil Engineering, Architecture and Building, Faculty of Engineering & Computing, Sir John Laing Building, Coventry University, Coventry, CV1 5FB

Abstract:
The production of Portland cement has significantly adverse effects on the environment due to the emission of carbon dioxide. Therefore reduction of Portland cement content will benefit the carbon footprint of concrete products. This paper presents the use of waste and by products materials such as basic oxygen slag (BOS), plasterboard gypsum (PG) and run-of-station ash (ROSA) to produce paving blocks by mixing in binary and ternary blends in 7 different groups of paste mixes and is compacted using a hydraulic press similar to factory process. Compressive and splitting tensile strength was measured on paving blocks and cubes specimens. Results confirmed that ROSA up to 50% by weight, BOS up to 70% by weight, combination of 40% GGBS and 40% BOS and combination of 5% PG by weight and 65% BOS, 15% ROSA and 35% GGBS can replace the Portland cement without having considerable impact on the desirable strength of producing paving blocks in accordance to the BS EN 1338: 2003.

1. Introduction:
Cementitious paste in the form of pre-cast paving blocks is widely used for a wide range of applications including that of exterior landscaping. Such blocks are available in a number of different shapes and colours. The appeal of paving blocks is that they are able to provide a hard surface which is visually attractive and easy to walk upon while at the same time allowing for easy maintenance and having a long life in use. They can therefore be used for the most heavy duty purposes such as car parks, bus terminal and stops, petrol stations, roundabout, Industrial estates, etc. and being able to cope with considerable loads as well as offering resistance to those forces that might shear the surface or otherwise damage it. Portland cement is the essential material that is used in all paving blocks. However, as is well known the production of every tonne of Portland cement releases approximately 1 tonne of carbon dioxide - a major contributor to the greenhouse gas emissions that are responsible for global warming. Considering the yearly Portland cement production of 1.6 billion tonnes, the cement industry itself is responsible for 7% of the total carbon emissions. (Turanli. 2004). Since 1970 ongoing attempts have been made by researchers to find suitable materials to partially replace Portland cement. A number of these materials, such as pozzolan, limestone and metakaolin occur naturally while others, such as fly ash, slag which has been produced by certain metallurgy processes, silica fume and others are the by-products of other industries. (Menéndez, et al. 2002). Therefore, using more waste materials will decrease their effect on the environment and help to save natural raw materials as well as reducing the overall energy required to produce a cementitious material and thereby reducing the emissions of carbon dioxide (CO₂) (Ganjian et al. 2007). The aim of this Research is to explore the possibility of using a mixture of different waste pozzolans to make paving blocks, and to reduce the percentage of Portland cement in the mixture.

2. Materials Used In This Research:
2.1 Plasterboard Gypsum (PG):
In this research the crushed plasterboard gypsum waste from demolition and construction sites was received from Lafarge plasterboard recycling plant in Bristol. Plasterboard used in the construction and repair of buildings as a facing for walls and ceiling, also it is used for forming certain structures,
for instance barriers. “Plasterboard waste can arise on construction sites for a number of reasons, including wasteful design, off-cuts generated during installation, damaged boards, and over-ordering” (Dunster, 2008). Crushed plasterboard gypsum waste from different sources, such as construction and demolition sites, was recycled and classified carefully to avoid some contaminants such as paper and glass.

2.2 Basic oxygen slag (BOS):
The basic oxygen slag in this research has been acquired from Tarmac UK (i.e. from the Corus plant at Scunthorpe). The creation of Basic Oxygen Steel Slag (BOS) is inevitable at this time because of the method of steel production. Apparently, it promotes the use of Basic Oxygen Steel Slag where it is possible; this is not only to avert the production of disagreeable slag accumulates, but also to decrease the necessity to reduce finite primary minerals. Therefore, the uses of basic oxygen steel slag (BOS) have been known for a long time and have been used in different fields under the Civil Engineering Corporation.

2.3 Run-of-station ash (ROSA):
Run-of-station ash for this research has been brought from Rugby Ash. Dry run-of-station ash (ROSA) comes from a power station and has been in paste mixes, and prepared as a fine powder.

2.4 Ordinary Portland cement (OPC):
The characteristics of cementitious mixes are dependent upon both the amounts and nature of those elements that go to make it up. The cement is the primary operative constituent of concrete and it is normally an expensive component and therefore its effective use is important if the greatest degree of economy and stability is to be the result for any specific paste mix. For this research the CEM1 cement was used as defined by European standard.

2.5 Grand Granulated Blast Furnance (GGBS):
The Ground Granulated blast furnace slag (GGBS) was obtained from Civil and Marine, a part of Hanson UK. The material was marketed under the standard BS 6699.

3. Experimental work and mix proportions:
In this research the aim was to find out the best way to achieve good consistent tests result for paving blocks made in the laboratory. A pressing action was used by making use of a compression machine. The mix designed used in this research was to obtain the highest compressive strength and tensile splitting strength of binary and ternary mixtures. Five different proportions of 7 groups of mixes were design and tested compressive and splitting tensile strength on paving blocks and cubes specimens. The water content for all groups of mixes was constant at 15 percent.

4. Casting, curing and testing:
Paving blocks in sizes of 190x100x76 mm, 190x100x80 mm and 190x100x75 mm and cubes of 50x50x50 mm were cast and fully compacted by use of a compression machine in one layer with 150 KN load. After casting, specimens were covered with polyten sheets to avoid any loss of water, by the following day all samples were de-moulded and stored in curing chambers with a constant air temperature of 22±2 °C and 98% relative humidity up to the date of testing.
The tensile splitting strength of paving blocks was determined in accordance with BS EN 1338: 2003. The minimum split tensile strength of 3.6 MPa is required for paving blocks strength. Blocks and cubes were tested at 14 and 28 days.

5. Results and discussion:
Results of compressive strength and split tensile strength of all groups are shown in figures 1 to 4 and table 1. The results show that 50% ROSA and 50% OPC produces the highest strength, and as the ROSA content increased the strength is reduced. The reason for this is that the ash particles act as filler without assisting gel production in cement paste matrix. In addition, using ROSA in ternary mixtures of OPC/ROSA/BOS and OPC/ROSA/PG satisfies the required Minimum strength criteria. On other hand, using 5% plasterboard gypsum in combination with OPC/ROSA/PG, OPC/BOS/PG and OPC/GGBS/PG as a partial replacement of cement showed satisfactory results for use as paving blocks.

The optimum range for GGBS for the ternary mix of OPC/GGBS/PG is between 25-35 percent in the study, and for ternary mix of OPC/GGBS/BOS is around 30-40 percent.

Furthermore, the results of mixes with BOS in OPC/GGBS/BOS, OPC/BOS/PG and OPC/BOS shows that up to 70 Percent of cement replacement can be achieved.

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### Table 1 Results for Compressive and Tensile strength after 14 and 28 days for groups from 3 to 7.

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<td>OPC60/BOS55/PGS 4</td>
<td>4</td>
<td>OPC60/BOS55/PGS 4</td>
<td>4</td>
</tr>
</tbody>
</table>
6. Conclusion:
The following conclusions can be drawn from this study:
1. It is possible to use waste minerals to achieve the Minimum tensile strength of paving blocks 3.6 MPa according to the British Standard BS BN1338: 2003.
2. The ternary materials (GGBS, ROSA and BOS) were more effective to reduce cement content.

References:
1. Introduction

Great Britain is moving towards a low carbon energy supply under the influence of EU directives (European Commission, 2011). This, along with the increased thermal performance of buildings, and increased efficiency in heating, cooling, ventilation, and lighting systems, will reduce the operational carbon of buildings, meaning their embodied carbon becomes a larger percentage of overall carbon emitted. This shifted focus to embodied carbon means that current building materials need to be re-evaluated to continue to reduce the energy used in construction. This may lead to low-embodied energy building materials to be more widely used, such as straw, a renewable co-product of the farming industry. Straw has many uses in Great Britain and it is the dynamic between these uses that may increase the competition for the resource.

2. Current uses of straw

In this paper, straw is defined as the dry stalks from crops after the grain and chaff have been removed. There are two families of straw; cereal (which include wheat, oats, and barley) and oilseed (linseed and oilseed rape). In 2007 annual straw production was estimated at 11.9 million tonnes (Turley & Copeland, 2008), and in 2010 close to 11 million tonnes (Biomass Energy Centre, 2011). As straw yield changes from crop to crop, and from year to year due to climate and changes in management, an average of 11 million tonnes will be taken. Figures from the UK and Great Britain have been compared on a like for like basis as Northern Ireland produced only 0.043 million hectares of arable crops in 2010 (AFBI, 2010).

Straw’s main use is for livestock as feed and bedding. Exact quantities of straw in feed are virtually impossible to calculate as each farmer has different practices, but it was estimated at 2 million tonnes of straw per year in 2007 (Turley & Copeland, 2008). The information on straw for bedding was derived from Great British livestock numbers along with the basic straw use for bedding in tonnes per livestock unit (DEFRA, 2006) and estimated at 6.2 million tonnes per year in 2007. It is currently estimated that 40,000 tonnes of straw is supplied to Great British mushroom growers (Turley & Copeland, 2008).

Straw is also used as a source of biomass. Ely power station in Cambridge is the largest straw burning power station in the world at 38MW and generating over 270GWh each year with an annual demand of 200,000 tonnes of straw (epr, 2011). Work on the 38MW Sleaford straw power station is starting April 2012 and will require approximately 240,000 tonnes of straw per year. The 40MW Brigg power station which would have used 240,000 tonnes of straw per year has currently been put on hold. On a smaller scale, there are plans for a straw-fired power station at Holderness and a combined heat and power plant in Goole, both in East Yorkshire with an annual demand of 64,000 tonnes and 43,000 tonnes of straw relatively (Turley & Copeland, 2008). In total, if all of these straw biomass stations were commissioned 587,000 tonnes of additional straw would be required as feedstock. Since the straw burning ban in 1992, excess straw has been ploughed back into the ground and it is now considered an important fertiliser (Keysoil, 2004).

3. Use of straw in construction

Straw construction takes many forms; the simplest of which involves using the bales of straw as load bearing elements. Straw bales are stacked to form walls on raised foundations where protruding spikes hold them in place, with more spikes or compression straps used to hold further courses of bales together. A continuous roof beam is usually placed on top of straw bale walls to give stability as well as an adequate surface on which to attach the roof. Load bearing straw bale construction is more common in the USA than Great Britain and there are state codes on how to design load-bearing and non-load bearing structures (Sher et. al., 1995) (International Code Council, 2011).
As a non-load bearing building material, straw is used as an insulative infill material in frame structures. Construction takes the form of stacking individual bales on site within timber frames or manufacturing prefabricated systems such as Green Panels® or ModCell®. As well as using baled straw for construction, there is strawboard; a technique where straw is subjected to high pressures and temperatures to release natural resins in the straw which binds the fibres together. These panels are then covered in paper or OSB. Commercial prefabricated strawboard panels such as Stramit Panels® and Durra Panels® are used as non-load bearing infill panels in frame structures.

To assess the capacity of the supply chain to fulfil the potential future needs of straw building in Great Britain a simple analysis, based on different building types, has been undertaken below.

**Amount of straw per m² floor area** - To estimate the potential demand for straw in construction, the straw requirements from three different buildings have been estimated. As there are no commercial strawboard manufacturing plants in Great Britain, case studies involving straw bales were looked into. The net usable floor area for each building has been compared with quantity of straw used in the external envelope of each building.

<table>
<thead>
<tr>
<th>Building</th>
<th>Description</th>
<th>Floor Area</th>
<th>Tonnes Straw used</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Balehaus, University of Bath</td>
<td>Prototype housing system made from ModCell® panels and 8 ‘straw boxes’ at the corners (Seguret, 2009)</td>
<td>71m²</td>
<td>Whole house uses approximately 4 tonnes of straw</td>
<td>0.0561 straw/m² Or 4 tonnes of straw/house</td>
</tr>
<tr>
<td>Eco Depot, York</td>
<td>Two storey commercial building made using 78No. 3x3.2 x0.48m ModCell® panels (Modcell)</td>
<td>2x1250m²</td>
<td>40255kg straw used (density of 112kg/m² assumed) (International Code Council, 2011)</td>
<td>0.0161 straw/m²</td>
</tr>
<tr>
<td>GE Sworder Auction House, Kent</td>
<td>Single storey straw bale commercial construction</td>
<td>1079m²</td>
<td>20 tonnes (Beckett, 2011)</td>
<td>0.018 tonnes straw/m²</td>
</tr>
</tbody>
</table>

Commercial space is measured in square metres of lettable floor space. The amount of straw needed per m² of commercial space has been estimated from the Eco-Depot and Auction House examples and so a value of 0.02 tonnes straw per m² usable floor area will be used. For domestic properties, the value of 4 tonnes of straw per house will be used.

The exact tonnage of straw per m² floor area will depend on the density of the straw, the type of straw, the shape of the building, and finally whether it is load-bearing or used as infill. The shape of the building is the most critical factor and the three examples given have high wall to floor areas which provides conservative estimates for the amount of straw needed. The auction house is one storey high, and the York Eco-Depot is two storeys high with a width to length ratio of 1:3. The Balehaus is a detached house and so has a greater wall to floor area compared to semi-detached and terraced housing.

**Construction Demand** - This paper aims to determine if there is an adequate supply of straw to meet construction demand in the future. As construction demand for housing and commercial properties in the UK varies year on year, large assumptions have to be made. To provide a conservative estimate on whether enough straw will be available for construction year on year, it will be assumed that all commercial properties and all housing built will use straw, and peak construction demand before the recession will be used.

The total floor space of commercial and industrial bulk class properties in England and Wales (Scottish data was unavailable) in 2008 was 608million m². Looking at data from the 10 years previously, this total area has fluctuated year on year, with an average increase of 3.6million m² (Communities and Local Government, 2009). This value will be taken to conservatively guess whether there is enough straw available for mainstream commercial construction in Great Britain.

To date, the peak number of houses completed in Great Britain was during 2007/08 at 203,220 houses (Communities and Local Government, 2011). It will be assumed that 100% of these houses will be built from straw.

<table>
<thead>
<tr>
<th>COMMERCIAL</th>
<th>Office and Retail Portfolio (millions m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>assume 100% commercial property built from straw</td>
<td>3.6/year</td>
</tr>
<tr>
<td>at 0.02 tonnes straw per m²</td>
<td>72,000 tonnes straw</td>
</tr>
<tr>
<td>HOUSING</td>
<td>Number of houses built in 07/08</td>
</tr>
</tbody>
</table>
On average, 11 Mt of straw are produced in Great Britain, most of which was used for agriculture and energy production; leaving a surplus of approximately 2 Mt. This is around twice the amount needed in the unlikely event that all commercial and domestic structures each year in Great Britain were built of straw. Although there may be enough physical straw to supply the construction industry, there are other issues that need considering:

Local variations in straw supply/demand balance - Large quantities of straw move from the arable East of Great Britain to the livestock-heavy West (Turley & Copeland, 2008), which suggests there is little potential for straw biomass feedstock in the West, and a surplus of straw in the East. This disparity in straw supply and demand means that for straw construction, the availability and cost of local straw can vary greatly depending on location.

Price - The price of straw is affected by the quality and quantity of straw available, which in turn depends on conditions during the growing season. The prices for ‘pick-up’ bales of barley or wheat straw have varied from £22 to £79/tonne in the last 10 years (British Hay & Straw Merchants, 2012). Currently surplus soil is re-ploughed into the ground where it improves the soil condition, lessening the need for fertilisers (Keysoil, 2004). This surplus straw now has a monetary value, and the minimum value for wheat straw is £47.76/t to be economically viable for farmers to sell it and buy fertiliser in its place (ADAS, 2009).

These minimum values may increase in the future as the price of fertiliser is temperamental and the price of diesel and petrol (required to run the machinery for administering fertiliser) is increasing, and could reach to £60/tonne (Booth, 2009). As the price of fertiliser reacts to the European and even global markets (EurekAlert, 2008) it is difficult to predict, and so difficult to predict how it will affect the price of straw in Great Britain.

A study conducted by Anglican Straw (Goodhall R., 2008) on straw as a viable biomass feedstock in the UK, stated that the straw would cost approximately £2/GJ if buying at around £35/tonne in 2008. This makes it comparable to coal at £1.50/GJ and much better than the £4.50/GJ for miscanthus or willow figures from 2007 (Goodhall C. E., 2007).

Considering that straw is now double that price, the cost per GJ can be assumed to be around £4.50/GJ in the UK (UK Coal Plc., 2010), following the predictions of ADAS(2009) and Booth E. et al (2009).

Where cost sensitivity of straw for biomass is high as fossil fuels still have a low cost per GJ, cost sensitivity for construction is low. In a ModCell® Panel, straw constitutes 60% of the volume but less than 2% of its retail cost (White, 2011), which is mainly in the lime render and labour. This means that if straw was to double in price, the cost of a ModCell® panel would be only marginally greater. Similar volumes of straw are involved in non-ModCell® construction as well and so these fractions are applicable across straw-bale construction.

Straw fuelled power stations - Even though the recent increases in straw price see it losing its competitive edge on coal, the EU directives to decrease the carbon intensity of UK energy could increase the number of straw fuelled power stations in the UK, especially since straw biomass achieves one of the highest load factors of any renewable energy plant at 90% (Edwards, 2006). This increase could lead to a battle for resources if the straw construction industry continues to grow. With straw construction, a relatively small amount of straw will be sourced in a one-off transaction for the building. Selling straw for biomass involves long term sourcing contracts, and so a more secure supply chain for the farmer. This could lead to farmers selling most of their straw to power stations, meaning there is less straw to sell for construction purposes.

Substitutions - Other insulation materials are a big threat to straw bale construction, sheep wool in particular (Penk, 2011). Sheep wool, similarly, has a low embodied carbon compared to traditional building materials, but unlike straw bale, sheep wool can be easily retrofitted into existing structures and it is more in keeping with traditional building techniques as it is installed in a similar way to fibreglass insulation. As 80% of existing UK housing stock will be standing in 2050 (House of Commons, 2009), retrofitting will be a big market for sustainable materials that straw will miss out on.

Straw-less Farming - An increase in the price of straw, fears of a straw shortage (BBC, 2011), plus innovations in livestock husbandry such as Tenderfoot®, a straw-less livestock bedding system, could lead to wide scale straw-less farming within Great Britain, reducing demand. Additionally, the
numbers of livestock in Great Britain are decreasing (UK Agriculture, 2010) and so the demand for straw for livestock may decrease further in the next 5 – 10 years.

5. Conclusion

There is potentially enough straw in the UK to increase straw bale construction to much larger scale than current practice, however there is growing competition for straw from other end users and straw costs are known to fluctuate yearly. Due to their high load factor and low carbon credentials, it is likely that more straw fuelled power stations will be built, thereby decreasing the supply of straw. At present power generation is likely to provide a more stable supply chain than construction and it is likely that farmers will enter into long term contracts with these power stations and so limit supply to other users.

Straw bale construction not only needs a supply of straw, but a supply of timber and lime plaster as well. The impact of scaling up and sourcing these building materials needs to be assessed. The disparity in straw supply throughout Great Britain could make it difficult to source local straw for large scale construction in the West.

Finally, the market for straw bale housing needs further investigation as it may be smaller than previously thought due to the increase in the use of sheep’s wool insulation and the difficulties in straw construction entering the retrofitting market. Also the uptake of hemp lime, CLT and other renewable materials for construction should be investigated as they are gaining popularity.

6. References

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(2009). Case Study 2 Straw incorporation since burning ban improves soil condition.
Lime Stabilised Soils; Implications for Sulfate Heave

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The University of Nottingham, Nottingham, NG7 2RD, UK

Introduction

The phenomena of sulfate heave, which can occur during the chemical stabilisation of susceptible soils, can cause dimensional instability and loss of mechanical strength, which can ultimately lead to the failure of the stabilised layer. The mechanisms of this deleterious process and its relationship to observed physicochemical behaviour are complex. This paper presents research undertaken at Nottingham Transportation Engineering Centre (NTEC) using a series of artificial lime stabilised sulfate bearing soils subject to two swell test procedures; their physical response; and the underlying phase transformations investigated using a range of advanced analytical techniques. The aim of which, is to further the understanding of the fundamental properties of sulfate heave.

Background

Soil stabilisation involves the addition of a hydraulic binder (and sometimes additions pozzolanic materials) to a soil containing clay minerals. The addition of the lime can first dry and modify the soil before raising the pH of the soil to >10.5 (a₁ and a₂) upon which the dissociation of aluminosilicates from the clay particles occurs (b):

a₁. \( \text{CaO} + \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2 + \text{heat} \)
a₂. \( \text{Ca(OH)}_2 \rightarrow \text{Ca}^{2+} + 2\text{OH}^- \) (pH >10.5)
b. \( \text{Al}_2\text{Si}_3\text{O}_10(\text{OH})_2\cdot n\text{H}_2\text{O} + 2(\text{OH})^- + 10\text{H}_2\text{O} \rightarrow 2[2\text{Al(OH)}_4^- + 4\text{H}_4\text{SiO}_4] + n\text{H}_2\text{O} \)

This then enables the solubilised clay particles to react with free calcium ions in solution to form calcium silicate hydrates (CSH), calcium aluminate hydrates (CAH) and calcium aluminium silicate hydrates (CASH), described here using general formulas.

These cementitious products are equivalent to those formed during the hydration of cement pastes. The insoluble hydrates form a cementitious matrix which fills void spaces within the soil to encompass unreacted clay particles. Subsequent curing of this cementitious matrix results in material with increased engineering performance in terms of dimensional stability, increased strength and stiffness.

If however, the insitu soil contains sulphur bearing minerologies such as gypsum (CaSO₄·2H₂O) and pyrite (Fe₂S) these can react with the lime and dissolved alumina to form expansive minerals such as ettringite – AFT (d); monosulfate – AFm (e) and in some special cases thaumasite.

c. \( \text{CaSO}_4 \cdot 2\text{H}_2\text{O} \rightarrow 2\text{Ca}^{2+} + \text{SO}_4^{2-} + 2\text{H}_2\text{O} \) (disassociation of gypsum)
d. \( 6\text{Ca}^{2+} + 2\text{Al(OH)}_4^- + 4\text{OH}^- + 3\text{SO}_4^{2-} + 26\text{H}_2\text{O} \rightarrow \text{Ca}_6[\text{Al(OH)}_2\cdot (\text{SO}_4)\cdot 2\cdot 26\text{H}_2\text{O} \) (formn ettringite AFT)
e. \( 4\text{Ca}^{2+} + 2\text{Al(OH)}_4^- + \text{SO}_4^{2-} + 4\text{H}_2\text{O} \rightarrow \text{Ca}_2\text{Al}_2(\text{SO}_4)(\text{OH})_{12} \cdot 6\text{H}_2\text{O} \) (formn monosulfate AFm)

Where deleterious sulfate reactions have contributed to this loss of durability, the formation of ettringite and thaumasite is often found to the cause. The formation of the highly expansive sulfate mineral ettringite, after the initial setting and hardening has occurred, (termed delayed ettringite
formation, [1] and [2]) can severely damage a stabilized soil layer and allied structures due to the expansive forces associated with its formation. This can result in a loss of structural integrity caused by microstructural damage to the cementitious matrix. The formation of thaumasite can not only cause damage due to expansive effects, but also breakdown the hydrated mineral elements associated with the strength giving properties of cemented materials [3]

**Experimental Method**

The research used a series of artificial soils blended in the laboratory with varying amounts of gypsum. Two clays were used: a kaolin (K) and a montmorillonite (M). The lime addition used was determined by the Eades and Grim test. Control soils were devised using the clay alone (K and M); the clay plus 5.0% sulfate (K5S and M5S); and the clay plus lime alone (K4L and M6L). The sulfated soils were dry blended with the lime and 0.5%, 1.5% and 5.0% sulfate for each clay to yield test mixtures (K4L0.5S, K4L1.5S, K4L5.0S......) The blended test soils were then compacted to their Optimum Water Content +2% in accordance with the method defined in the two swell tests:

a. UK Linear CBR Swell Test (BS1924-2)
b. European Accelerated Volumetric Swell Test (EN13286-49)

After the soils had been tested to the two swell tests, they were analysed using a combination of SEM/EDX, XRD and TGA to understand how the structure and phase composition changes in the soils as a function of starting composition and the conditions imposed by the swell tests.

**Results**

Examplar results of the swell testing are shown in Figure 1 for the kaolin series of soils subject to the UK linear CBR swell test (left) and the European accelerated swell test (right).

*Figure 1: Linear swell results of Kaolin soils at 28 days immersion (BS1924-2), left and after 7 days immersion (EN13286-49), right*

Under both tests, the degree of dimensional instability was proportional to the amount of sulfate in the lime stabilised soils. The degree of both linear and volumetric swell exhibited by the sulfated clays was proportional to sulfate content and followed the series K4L < K4L0.5S < K4L1.5S < K4L5.0S.
SEM-EDX images of the clays show interesting compositional changes based both the amount of sulfate in the soil mixture and the swell test used. Figure 2 and Figure 3 show typical morphology of the low and high sulfate kaolins (EN13286-49). In the low sulfate clay, the typical laminar morphology of monosulfate (AFm) was predominant, whereas in the high sulfate clay (K4L5.0S) the acicular crystal habit of ettringite (AFt) was found. Both was confirmed by EDS analysis.

**Figure 2** SEM/EDX of low sulfate kaolin (K4L0.5S) after testing to European Accelerated volumetric swell test (EN13286-49)

**Figure 3** SEM/EDX of high sulfate kaolin (K4L5.0S) after testing to European Accelerated volumetric swell test (EN13286-49)

Figure 4 shows the TGA plots of the kaolin soils subject to BS1924-2 (left) and EN13286-49 (right).

**Figure 4** Differential TGA plot of kaolin sulfate soils at 28 days (BS1924-2), left and 7 days (EN13286-49), right. 1 - desorption H2O, 3 - Ca(OH)2, 6 and 6a – ettringite (AFt), 7 – CAH (CaAH13), 8 – hydrogarnet (generic CSH), 9 – CAŠH: solid solution between CAŠH and monosulfate (AFm)

It can be seen in the left-hand figure that residual lime is still present in all the samples (3), whereas there is a much reduced peak in the right-hand plot. The most distinguishing feature is a large peak (7 or 9) and an accompanying shoulder peak (9) of the low sulfate kaolin (K4L0.5S) subject to the
European accelerated test, not present when tested to the UK linear CBR swell test. A small peak attributed to AFt formation was only detected in the high sulfate soil (K4L5.0S) irrespective of the test employed.

Discussion

The relationship between sulfate heave and the formation of ettringite in particular is complex. Several potential mechanisms have been suggested and the matter is as yet unresolved [4]. Quantification of AFt phases by XRD is extremely difficult due to its tendency to form amorphous gels in the pore solution and a high degree of preferred orientation of the elongated crystals [5] The author has undertaken extensive QXRD studies on the soils reported here and are to be published at a later date. Considering the data reported here, it is clear that a proportional relationship exists between the amount of gypsum in a soil and the amount of observed swell. It is suggested that this correlates to the amount of the AFt and AFm phases formed. These can be differentiated by SED-EDX analysis. AFt – acicular, needle-like crystals and AFm – large lamellar, plate-like crystals. XRD and further SEM/EDX analysis confirmed that AFt was formed in all lime stabilised sulfated soils. Considering Figure 4, it maybe that TGA is not a sufficiently sensitive technique to detect AFt at the concentrations formed in these soils.

Conclusions

Based on the work reported here and other work conducted by the author, the following conclusions can be reached:

a. For both swell tests (BS1924-2 and EN13286-49) the amount of sulfate heave follows the series: K4L < K4L0.5S < K4L1.5S < K4L5S.

b. The propensity to form AFt and AFm is based on a combination of the amount of sulfate in the starting mixture and the swell test employed in its assessment. This could have important implications on the behaviour of soils not only subject to a particular swell test, but also for their behaviour insitu.

c. TGA is not a sufficiently sensitive technique to not only quantify but detect ettringite (AFt) formation in the sulfated lime stabilised soils reported herein.

References

Abstract

Having the unique characteristics of cellular microstructure with high air content, lightweight (300 to 1600 kg/m³) and high workability (self-flowing and self-compacting), foamed concrete has become a well-known and widely used construction material over the past 20 years. Flexibility in design allows it to be used in various construction applications from insulation to road bases and ground supported slabs, as well as its most common use in large-scale void fill. Foamed concrete is produced by adding pre-formed foam to a ‘base-mix’ of cementitious mortar or paste, can, therefore, be designed to yield plastic densities ranging from 1600 down to 300 kg/m³. For cost considerations, surfactants used to produce foam tend to be protein based, although synthetics are also used. Foamed concretes are particularly sustainable, as they can be produced by using low embodied carbon dioxide cements, as well as recycled and secondary aggregates. Moreover, foamed concretes themselves can be easily reused as an aggregate, requiring minimal energy for crushing. This paper covers, in general, characteristics and durability properties of foamed concrete, its applications and role in the sustainable construction.

Introduction

Along with its unique characteristics of being lightweight, cellular and highly workable, foamed concrete brings solutions for various construction applications in an environmentally friendly way. Its flexibility in design results in production of foamed concrete at wide range of densities with air contents reaching up to 75-80%. Therefore, flexibility in design, in turn, increases diversity of foamed concrete applications.

Foamed concrete is produced by mixing pre-formed foam with a ‘base mix’ that is composed of cement, water and fine aggregates, whereas the use of aggregates is eliminated at densities below 600 kg/m³. Various cement types ranging from Portland cement to fly ash are used in the production of foamed concrete at range of densities in order to adapt the characteristics and the behaviour for a specific application.

Similarly, in order to produce the pre-formed foam, either protein-based or synthetic surfactants are used. Each surfactant produces foam with different structure and stability. Compared to synthetic surfactants, protein-based surfactants produce more stable foam with relatively closed structure as well as fully formed bubbles [2]. Although, both types are used, protein-based surfactants are more likely to be employed, since they are cheaper, more sustainable and result in more stable foam.
Properties of Foamed Concrete

Typically foamed concretes show compressive strengths ranging from 1 to 10 N/mm²\(^2\)\(^1\). However, compressive strength is greatly influenced by the type of cement, fine aggregate/filler and water/cement ratio. Accordingly, compressive strengths from 0.5 to 30 N/mm²\(^2\) are reported for densities ranging from 300 to 1600 kg/m³.

Foamed concrete is known to have good durability properties, showing enhanced resistance against both aggressive chemical environments and freeze/thaw cycles. It is the cellular structure of foamed concrete that provides space for the expansions caused by freeze/thaw cycles, explaining the reported lower resistance of higher density foamed concretes\(^6\).

Having good thermal and sound insulation properties, foamed concrete increases its attractiveness. Roughly speaking, thermal conductivity values from 0.1 to 0.6 W/mK are reported for densities ranging from 400 to 1600 kg/m³ (see Table 1). Along with its good thermal performance at elevated temperatures, foamed concrete is an incombustible material, offering an alternative for being used in fire walls\(^3\),\(^4\).

Table 1. Typical properties of foamed concrete\(^1\),\(^3\),\(^4\)

<table>
<thead>
<tr>
<th>Dry density (kg/m³)</th>
<th>Compressive strength (N/mm²) – 7 days</th>
<th>Thermal conductivity (W/mK)</th>
<th>Drying shrinkage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>400</td>
<td>0.5-1.0</td>
<td>0.1</td>
<td>0.30-0.35</td>
</tr>
<tr>
<td>600</td>
<td>1.0-1.5</td>
<td>0.11</td>
<td>0.22-0.25</td>
</tr>
<tr>
<td>1000</td>
<td>2.5-3.0</td>
<td>0.23-0.30</td>
<td>0.15-0.18</td>
</tr>
<tr>
<td>1400</td>
<td>6.0-8.0</td>
<td>0.50-0.55</td>
<td>0.07-0.09</td>
</tr>
<tr>
<td>1600</td>
<td>7.5-10.0</td>
<td>0.62-0.66</td>
<td>0.06-0.07</td>
</tr>
</tbody>
</table>

Applications

Given its wide range of densities as well as ease to place and excavate, foamed concrete has broad application areas. Some common applications of foamed concrete are listed as void filling, insulation, pre-cast blocks and trench-fill foundations. A recent example application of foamed concrete as a road foundation was near to the Thames in London, where an access road was to be built on a peat soil that was prone to settle. In order to meet the design requirements against the risk of flooding, it was needed to raise the road level. Use of conventional pile foundations would bring a solution, perhaps it would be an expensive option. Therefore, foamed concrete was used as an economical and practical option to replace the excavated made ground on the peat layer causing no increase in net stress, therefore, no settlement\(^1\).

Sustainable construction

Given its high air content and low amount of aggregate content foamed concrete is a sustainable material. As well as the wide range of densities, which directly affects the air content of the material, flexibility in the use various constituent materials such as low embodied CO₂ cements and recycled and secondary aggregates (RSA) further increase its contribution to the sustainable construction. The difference between the embodied CO₂ of Portland cement (930 kg CO₂/tonne) and fly ash (4 kg CO₂/tonne) clearly shows the huge amount of contribution made to the sustainable construction when low embodied CO₂ cements are used (see Table 3). Moreover, recycling/reuse potential of foamed concrete minimizes the demolition wastes without requiring high energy inputs for processing, since foamed concrete has low bonding energy. It is also reported that, performance of reused foamed concrete increases with the number of crushing cycles\(^5\) (see Table 2).
Table 2. Effect of crushing on the performance of reused foamed concrete [5]

<table>
<thead>
<tr>
<th>Number of crush/recycle</th>
<th>Foamed Concrete Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Plastic density (kg/m³)</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>1035</td>
</tr>
<tr>
<td>1</td>
<td>985</td>
</tr>
<tr>
<td>2</td>
<td>1020</td>
</tr>
<tr>
<td>3</td>
<td>1010</td>
</tr>
<tr>
<td>4</td>
<td>990</td>
</tr>
<tr>
<td>5</td>
<td>1015</td>
</tr>
<tr>
<td>6</td>
<td>1030</td>
</tr>
</tbody>
</table>

Use of RSA in foamed concrete

Primary aggregates, one of the main constituents of concrete, has considerably high impact on the environment, therefore, minimizing the use of primary aggregates by substituting them with recycled and secondary aggregates is vital [8]. Increased use of recycled and secondary aggregates that are either wastes or by-products of various processes, has recently brought a new aspect to foamed concrete production maximizing its sustainability. However, it should be emphasized that heterogeneity of the RSA materials needs to be analysed, since it has a vital effect on the characteristics and the behaviour of foamed concrete.

RSA materials used to replace the primary aggregates in foamed concrete are listed as demolition fines/silt (DF), incinerator bottom ash (IBA), glass fines (GF), foundry sand (FS), China Clay sand (CCS), conditioned fly ash (CFA) and crumb rubber (CR) (see Table 3).

Table 3. Mean particle sizes and sources of RSA materials [6]

<table>
<thead>
<tr>
<th>Type</th>
<th>Mean particle size (mm)</th>
<th>Source</th>
<th>Embodied CO₂ (eCO₂) per 1 m³ of foamed concrete (kg CO₂/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DF</td>
<td>0.8</td>
<td>Construction demolition and excavation waste</td>
<td>Density= 1000kg/m³, 100% PC 70% PC-30% FA</td>
</tr>
<tr>
<td>IBA</td>
<td>2.5</td>
<td>Municipal solid waste incineration</td>
<td>281 198</td>
</tr>
<tr>
<td>GF</td>
<td>0.5</td>
<td>Building flat glasses</td>
<td></td>
</tr>
<tr>
<td>FS</td>
<td>0.18</td>
<td>Sand used in the metal casting industry</td>
<td>281 198</td>
</tr>
<tr>
<td>CCS</td>
<td>0.6</td>
<td>Fines from China Clay waste</td>
<td></td>
</tr>
<tr>
<td>CFA</td>
<td>0.02</td>
<td>Coal combustion (water addition for conditioning)</td>
<td>Density= 300kg/m³, 100% PC 70% PC-30% FA</td>
</tr>
<tr>
<td>CR</td>
<td>1.0</td>
<td>Shredded truck tyres</td>
<td></td>
</tr>
<tr>
<td>FA</td>
<td>0.02</td>
<td>Coal combustion (conforming to BS 450)</td>
<td>186 130</td>
</tr>
</tbody>
</table>

Performance of RSA foamed concrete

RSA materials can successfully replace the primary aggregates in foamed concrete, as long as the heterogeneity and the particle density of the RSA material are analysed. In most cases, incorporation of RSA in foamed concrete results in comparable performance with primary aggregate foamed concrete, however, variations in the characteristics and the performance tend to occur with changing material heterogeneity and density.
In terms of fresh properties, flow characteristics of RSA foamed concrete is comparable with sand foamed concrete except from CR foamed concretes which is not self-flowing. Given its low particle density, crumb rubber reduce the workability, however, increasing the cement content, hence the water content, overcomes this issue.

When hardened properties are considered, cube strength of RSA foamed concrete is similar to the sand foamed concrete, whereas being low at early ages and significantly high at later ages in the case of conditioned fly ash. Material density of RSA materials is an important factor affecting strength, given RSA materials with low density and water absorption result in higher strengths at all ages compared to the high density and water absorption materials. In terms of thermal conductivity performance, RSA foamed concretes perform same with sand foamed concrete, suggesting that thermal conductivity is more dependent on the density of foamed concrete.

Although variations occur with changing RSA type and level of primary aggregate replacement, drying shrinkage strain of RSA foamed concrete is mostly higher. Even though it is difficult to generalise, RSA foamed concretes show similar resistance against sulfate attack which should be taken into account for void filling applications in sulfate-bearing soils. Results for drying shrinkage and resistance to sulfate attack suggest further work [6].

Conclusions

Foamed concrete has now become a familiar construction material that utilizes a comparatively complex interaction of Portland cement chemistry with that of protein foams. There is much still to be understood about this interaction, particularly with respect to chemical refinement between cement/filler type and the surfactant. However the material has been successfully used in small and mega projects for the past 20 years.

The paper focuses on sustainable construction, both in terms of the use of RSA, and the more effective reuse of the material itself. In addition, some data on embodied CO₂ is provided. An example of a clever application of the material overcoming a geotechnical problem is presented, showing the engineers are finding new ways of exploiting the unique properties of foamed concrete.

References

UNDERSTANDING AND ENHANCING THE MOISTURE-DAMAGE PERFORMANCE OF ASPHALT MATERIALS

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South Kensington Campus, London, SW7 2AZ, UK
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Introduction
It is thought that a major cause of the failure of the road surface is due to moisture attack on the asphalt mixture, which can lead to interfacial debonding between the bitumen and the aggregate particles and can lead ultimately to cracking [1]. Numerous laboratory test methods have been developed to identify the durability of asphalt mixtures and their response to moisture [2, 3]. However, many of these methods are too complex to allow differentiation between the response of bitumen binders and the response of aggregates and the interfacial interactions to moisture-damage. A more fundamental and direct measurement of both adhesive and cohesive properties in asphalt system is increasingly needed. Direct measurements of the bitumen-aggregate adhesion and the binder cohesion will allow the measured values of the fracture energies to be correlated with the moisture-damage performance of the corresponding asphalt system. The peel test [4] has been shown to be a suitable method to study the adhesion and fracture properties of viscoelastic asphalt materials. The analysis of the peel strength and fracture surfaces provided essential information on the fracture mechanisms operating in the asphalt.

Materials
Limestone was selected as it is known to possess good moisture resistance [5], and was therefore used here as the standard aggregate material as it has given consistent performance in tests. In this work, 40/60 pen bitumen is used. A 1200 series aluminium alloy strip of thickness 0.2 mm was used as the flexible peel arm.

Experimental Method

Peel joint manufacture and peel test procedures
In this work, the aggregate formed the rigid adherend, which was bonded to the peel arm (flexible adherend) using the bitumen binder as the adhesive. The thickness of the adhesive layer was controlled at 0.25 mm via a continuous undulate metal wire spacer placed along the bondline.

The peel test was conducted at controlled ambient conditions, 20 ± 2 °C and 50 ± 5 % relative humidity. During the test, as shown in Figure 1, the peel force as a function of the displacement to initiate and propagate a peel fracture was recorded.

![Figure 1. 90° peel testing, (a) before test and (b) during test.](image-url)
Moisture conditioning of bitumen-aggregate joints

Moisture was introduced into the peel joint after bonding the aggregates by submersing the whole specimen into water for a period of time, shown in Figure 2. In this way, the water permeates into both the bitumen binder and the aggregate simultaneously. This method can closely simulate the effect of moisture on an asphalt mixture.

Figure 2. Schematic diagrams, post-bond moisture conditioning: whole bitumen-aggregate joint.

Results and Discussion

Fracture strength and energies

The tests were conducted at a speed of 10 mm/min, which has been selected as the standard test speed for this medium penetration grade bitumen at 20°C to ensure stable failure. After the initiation stage, the peel force shown in Figure 3 (a) remained at a consistent value, indicating the peel fracture progressed steadily. This was consistent with the fracture surface, shown in Figure 3 (b), in which a uniform layer of bitumen residue can be seen, indicating cohesive failure occurred within the bitumen binder throughout the bondline. This result was reproducible and was typical for a joint made by using 40/60 pen bitumen and limestone with the peel force of 23 ± 2 N. Fracture energy, $G_{Adv}$ value of the dry specimen was calculated [4] to be 633 J/m².

Figure 3. Peel results for a dry joint (40/60 pen bitumen -limestone): (a) the peel force and (b) the fracture surface.

Effects of moisture

To investigate the sensitivity of the measured peel force and the resulting fracture energy to moisture, peel tests were undertaken using water conditioned bitumen-aggregate joints. They were conditioned in water for up to 10 days. Figure 4 shows an example of post-bond moisture conditioned peel test results. Apart from introducing water into the specimens, this specimen was
made of the same materials as the specimen in Figure 3 and they were tested at the same conditions. It can be clearly seen that the peel force decreased in the water conditioned specimen. This indicates that the joints suffer a reduction in strength with the presence of moisture. There is very little bitumen residue on the aggregate surface in Figure 4 (b), signifying an interfacial fracture (between the aggregate and bitumen) in the moisture conditioned specimen. This suggests that the moisture reduces the adhesion between the aggregate and the bitumen.

The values of the fracture energy and the locus of failure for the peel joints are summarised in Table 1. The peel force and the \( G_A \) values for the water conditioned specimens were much lower than those of the dry specimen. The peel force and the corresponding \( G_A \) values decreased with increasing conditioning time. This result can quantify the extent of the loss of the strength in the bitumen-aggregate joints by moisture-damage. A dimensionless energy ratio combining the two fracture energies, \( \frac{G_A}{G_{A0}} \), can represent the moisture sensitivity of the joints. When the specimens were post-bond conditioned for 10 days, the low peel force of 4 N gives a \( G_A \) value of 69 J/m², which is only 11% of that of the dry specimen.

<table>
<thead>
<tr>
<th>Sample Description</th>
<th>( P ) (N)</th>
<th>( G_A ) (J/m²)</th>
<th>( \frac{G_A}{G_{A0}} )</th>
<th>Locus of fracture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry specimen</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Conditioned specimen</td>
<td>1 day</td>
<td>10</td>
<td>212</td>
<td>0.33</td>
</tr>
<tr>
<td></td>
<td>5 days</td>
<td>8</td>
<td>160</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>7 days</td>
<td>8</td>
<td>160</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>10 days</td>
<td>4</td>
<td>69</td>
<td>0.11</td>
</tr>
</tbody>
</table>

To differentiate the respective responses of the cohesive and adhesive properties of the bitumen-aggregate system to moisture-damage, an aluminum substrate was used to replace the aggregate. By this way, the effect of moisture-damage on the cohesive property of the bitumen binder can be studied as the aluminum substrate does not take in water. As shown in Figure 5, cohesive failure took place in the post-bond conditioned bitumen-aluminum specimen, with the peel force remaining as high as the dry bitumen-aluminum specimen, as listed in Table 2.
Table 2. Comparison of peel forces and fracture energies of dry and conditioned 40/60 pen bitumen/aluminum joints.

<table>
<thead>
<tr>
<th>Sample Description</th>
<th>P (N)</th>
<th>$G_A$ (J/m$^2$)</th>
<th>$G_A/G_{A0}$</th>
<th>Locus of fracture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry specimen</td>
<td>23</td>
<td>633</td>
<td>1</td>
<td>Cohesive</td>
</tr>
<tr>
<td>Conditioned specimen</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1 day</td>
<td>23</td>
<td>633</td>
<td>1</td>
<td>Cohesive</td>
</tr>
<tr>
<td>5 days</td>
<td>22</td>
<td>597</td>
<td>0.94</td>
<td>Cohesive</td>
</tr>
<tr>
<td>7 days</td>
<td>22</td>
<td>597</td>
<td>0.94</td>
<td>Cohesive</td>
</tr>
<tr>
<td>10 days</td>
<td>23</td>
<td>633</td>
<td>1</td>
<td>Cohesive</td>
</tr>
</tbody>
</table>

Conclusions

A peel test protocol has been successfully established for the testing of bitumen-aggregate system to study the fracture mechanism of the road materials at both dry and wet conditions. This test protocol enables the examination of the impact of moisture-damage on both the cohesion and adhesion of an asphalt system. It has been found that moisture-damage is mainly attributed to a reduction in adhesion between the bitumen and the aggregate. High cohesive strength of the bitumen binder remains under the presence of moisture.

Acknowledgements

The authors thank Prof G. D. Airey and his group in Nottingham Transportation Engineering Centre, University of Nottingham, UK, for providing bitumen and aggregates and for their support and useful discussions. The authors acknowledge EPSRC and industrial collaborators for funding the project and providing materials.

References


Figure 5. Peel results for a post-bond conditioned (10 days) joint (40/60 pen bitumen-aluminum): (a) the peel force and (b) the fracture surface.
THE EFFECT OF DEWATERING ON STRENGTH AND SETTING TIME OF HYDRAULIC MORTAR

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1. Introduction

In masonry construction jointing mortars, renders and plasters are applied in the wet state in thin layers to absorbent substrates such as brick and are dewatered before the setting process begins. The absorption of water from fresh mortar by clay brick depends on both the sorptivity of the substrate and desorptivity of the fresh mix [1]. Many researchers [1-3] have shown that lime based mortars possess high water retaining ability in comparison with cement based mortars.

Water retention is the ability of a fresh mortar to retain its mix water when placed in contact with an absorbent substrate. In 2002, Hall and Hoff [4] derived the theoretical basis and quantified the water retaining ability of fresh mortars by a well-defined parameter, the desorptivity $R$, obtained from the gradient of a plot of volume of water absorbed per unit area versus the square root of time.

The present work reports an investigation of the effect on mortars of dewatering by clay brick using an absorbent mould of the same dimensions as the standard steel test mould (40x40x160 mm). The properties examined are initial and final setting time; and compressive and flexural strength.

2. Mould design

Calculations were carried out to determine the thickness of brick and the time required to dewater a wet mortar mix in a brick mould of the same dimensions as a standard steel mould. A theoretical model which defines the time to dewater a freshly mixed mortar is described in [5]. Since the mould is constructed of sufficient thickness of brick, the wet mortar is fully dewatered prior to setting. Golden purple brick was used for the mould construction as it has relatively high sorptivity. Figure 1 shows the brick mould and a schematic representation of the flow of mix water from the wet mix to the absorbent mould.

![Figure 1: (a) the brick mould, (b) schematic representation of dewatering.](image_url)

3. Experimental work

The binder materials used in the experimental work were CEM1 Portland cement and St Astier natural hydraulic lime NHL5. NHL5 was chosen because it is the most hydraulic of the natural
hydraulic limes and its setting is mainly by hydration reactions with the carbonation reaction having less effect. Mortar sand was dried at 105 °C, and the sieved fraction of 300-600 μm was used in all mixes. A single mix design of 1: 2: 0.78 (binder: sand: water) by volume was examined in this study. This mix proportion is recommended by the manufacturer to give optimum workability [1]. The mass of both binder and sand required to produce the mix proportions by volume were calculated after careful determination of density values. To ensure consistency, a standard mixing regime was used.

Initial and final setting times were determined using the Vicat method (BS EN 196-3:2005 and A1:2008). The initial setting time is defined as the time elapsed since the mixing water was added to the binder to when the needle penetration is 4 mm from the base of the mould. The final setting time is defined as the time when there is less than 0.5 mm penetration of the needle. Setting time was determined for the dewatered and non dewatered cement and lime mortars at two different temperatures: 15°C and 25°C. For the dewatered mortars, Vicat measurements were not begun until the wet mix had been fully dewatered. This time was established by calculations.

Flexural strength measurements were carried out according to BS EN 196-1: 2005. Six prism samples of each mortar mix at each age were tested: three dewatered samples (brick mould) and three non-dewatered (steel mould). Following the flexural test, each bar produced two samples which were then used for compressive strength measurement. The compressive strength was measured on one of these two pieces.

4. Results and discussion

Comparison of the results obtained from the Vicat test for initial and final setting time of dewatered and non-dewatered cement and hydraulic lime mortars at 15 and 25°C are shown in figure 2. The results show clearly that the lime initial and final setting times are longer than for cement. this is because the initial setting time is the time needed to change from a fluid to a rigid stage [6], and the final setting time represents the end of workability and the beginning of hardening. Setting of hydraulic lime is slower than cement mortar because the dominant calcium silicate present in hydraulic lime is C₂S and not C₃S. Also, both initial and final setting times are reduced for both by dewatering. The initial setting time of dewatered cement mortar at 15°C was reduced from 3.92 h to 0.75 h which represents an 80% reduction. The final setting time was reduced by a similar amount: from 6.2 h to 2.4h and represents 61%. The dewatering effect on setting time of lime mortar is nearly the same. The initial and final setting of NHL5 mortar were reduced by approximately 6 hours at 15°C which represents 77% and 61% reduction of the initial and final setting time respectively. Also it is well known that setting time is affected by temperature, it reduces as the temperature rises. The results in figure 2 also show that the reduction in setting time of dewatered CEM I and NHL5 mortars is greater at 25°C than at 15°C. Although the Vicat test does not give an obvious link to hydration and it is not a fundamental measure of setting time, the test is, however, entirely suitable for the comparative results carried out in this work. It is worth noting that in the development of new more sophisticated setting time test methods [7-9], the fundamental issue that the dewatered mortars are different to the non-dewatered mortars is not addressed.

The strength results presented in figure 3 show the average values (of three) of flexural (R_f) and compressive strength (R_c) of both dewatered and non-dewatered lime and cement mortars at 7, 28, 56 and 90 days. Dewatered samples show a higher strength at all ages than the non-dewatered samples of cement and lime mortars. In each case the strength is increased following dewatering in the wet-state. The higher increase in both compressive and flexural strength of dewatered cement samples is at 7 days (37 % and 27% respectively. Figure 3 (a) & (b)). For NHL5 flexural strength of dewatered samples at all ages is 3-4 times the non-dewatered as shown in figure 3 (c). The compressive strength of dewatered NHL5 samples at 28 days is more than 3 times that of non-
dewatered samples and at 56 days is more than 4 times but shows little further increase thereafter as shown in figure 3 (d). The water retentivity of Portland cement mortar is much less than that of natural hydraulic lime NHL5 mortar [1-3], therefore the loss of water from cement mortar on dewatering is higher than the loss from the equivalent NHL5 mortar. The strength of cement at early age (7 days) increased by this abstraction of mix water therefore the difference is higher for the cement mortars at early age.

Figure 2: Initial and final setting time for dewatered and as-mixed (non-dewatered) mortars at 15 and 25°C. CEM I mortars (a) & (b), and NHL5 mortars (c) & (d).

Figure 3: Flexural and compressive strength of dewatered and non-dewatered mortars. CEM I mortars (a) & (b), and NHL5 mortars (c) & (d). (Note y axis scales).
5. Conclusions

- It is possible to dewater mortars to the same extent that they will be dewatered in use by a suitably designed and constructed brick mould.
- The initial and final setting times of both cement and hydraulic lime mortars are greatly reduced by dewatering.
- Dewatering results in a significant increase in both flexural and compressive strength.
- The higher the desorptivity, \( R \), the more the water releasing the mortar and therefore the greater the effect of dewatering on both setting time and strength.
- The standard steel mould used in testing the strength of mortars gives values that underestimate the strength of mortars in masonry. Therefore there are implications for standards: mould material, procedure, and time to testing should all be considered. Currently lime mortars are tested at 28 days as for cement mortars, and clearly this time requires consideration.

6. REFERENCES


ABSTRACT

The Midland Links Motorway Viaducts (MLMV) are located around the UK’s second largest city - Birmingham - and comprise 21 kilometres of elevated bridge motorway structures. Impressed Current Cathodic Protection (ICCP) has been used to prolong the life of more than 700 concrete structures in the MLMV, in a significantly sustainable manner, by reducing the need to remove chloride contaminated (but otherwise structurally sound) concrete.

The present study was initiated after identifying that some of the ICCP systems were reaching the end of their design life, whilst others had deteriorated or been vandalised, therefore requiring a significant level of maintenance, and cost. The objective of this work was to collate evidence from field structures to support preliminary laboratory results that the application of ICCP to a reinforced concrete structure over a period of time can transform the protective environment around the reinforcement.

The results can indicate when repairs to ICCP systems are likely to be critical; provide new evidence for determining the design life; reduce the requirement to replace systems at the end of their functional life; and the interval between planned maintenance of existing systems may be extended with corresponding reduction in monitoring frequency and costs. The work is unique and novel as it is the only ICCP field trial across Europe on full-scale motorway structures. It also contributes to sustainability as the results form a basis for an improved maintenance strategy.

INTRODUCTION

Impressed Current Cathodic Protection (ICCP) is a well-established repair method for corroding reinforced concrete elements with a track record of more than 30 years worldwide. The single largest application of ICCP in Europe is in the United Kingdom on the Midland Links Motorway Viaducts where over 700 concrete structures are currently protected.

Long-term monitoring of field structures suggests that after steel passivity has been induced then the protection current may be interrupted. The technical reason for this is that the application of ICCP has resulted in an increase in the reservoir of inhibitive hydroxide ions at the metal surface which will stifle the corrosion process.

However, a recent report by the Transportation Research Board, U.S.A. (2009) surveyed National Transportation Agencies in the USA to identify where ICCP is used, the reasons for its selection and...
explanations why it is not used by other States. They concluded that the technique is not used because of disappointing past experience, being more expensive than other options, and monitoring and maintenance was a significant burden.

In addition, recent experience in the UK has demonstrated that several ICCP systems were now reaching the end of their design life while others were suffering from material deterioration (Figure 1) or vandalism. This has resulted in several structures being in need of refurbishment.

**Figure 1: Typical material deterioration**

This work sought to identify the existence of long-term effects from the use of ICCP in a number of field structures. The objective was to systematically collect data from in-service structures that can be compared to published laboratory testing and hence establish if field evidence exists for the effect of long-term ICCP application (Christodoulou et al. 2010).

**METHODOLOGY**

Figure 2 illustrates a typical arrangement of the sub-structure for the Midland Links Motorway Viaducts. Approximately 700 reinforced concrete crossbeams have so far been protected with ICCP to extend their service life due to chloride contamination. Ten crossbeams were selected for testing based on the age, condition and environmental exposure of the ICCP system and chloride contamination of the crossbeams.

**Figure 2: Typical structural arrangement of the Midland Links Motorway Viaducts**

The protection offered by the ICCP system was switched off for a period of 52 months and the crossbeams were monitored for signs of corrosion onset. Corrosion activity was assessed based on: a) corrosion potential measurements, b) polarisation resistance and c) impedance testing for
corrosion rates. All of the techniques are well established techniques in the field of corrosion assessment and in particular employing impedance analysis on site structures was another unique and novel feature of the current work.

RESULTS
From the monitoring data over a period of 52 months, there were no evidence of corrosion activity of the reinforcement. Steel potentials remained stable in general without any great fluctuations indicating passive condition of the reinforcement. Corrosion rates calculated both from polarization resistance and impedance analysis returned extremely low values indicating the absence of corrosion (Figure 3). This is despite the fact that the tested crossbeams did not receive any protection for the past 52 months and they retained high residual levels of chlorides which posed a significant corrosion risk to the structures.

Furthermore, it was demonstrated that impedance analysis can be successfully employed on site to assess the corrosion condition of reinforced concrete structures. The technique requires a very large data set for analysis which significantly improves accuracy and eliminates errors. In addition, measurements are taken when there is no electrical current passing through the structure, which eliminates errors in the calculation of the corrosion rate due to interference of the concrete resistivity.

![Figure 3: Corrosion rate monitoring over a period of 52 months](image)

DISCUSSION
At the start of the study all the structures were assessed for their corrosion risk. It was found that the structures located at Lath Lane and Ray Hall Lane (C1/21, see Figure 3) were the two at most risk due to high residual chloride contamination. Chloride sampling results showed that these two structures had more than 40% of their test locations with chlorides greater than 1% by weight of...
cement and about 60% 66% of their test locations with more than 0.4% chlorides by weight of cement at the depth of the steel.

With reference to the steel potentials and the corrosion rates from polarisation resistance testing over a period of 52 months, the data suggests that there is no significant corrosion activity on the structures. More specifically it can be observed that a poorly performing system, as illustrated by Error! Reference source not found., it had been capable of inducing and maintaining steel passivity even after protection has been lost.

The results of this field study can help improve the asset management strategy of Maintenance Agencies. When considering the repair of old ICCP systems the passivation of the reinforcement from the previous system should be taken under consideration. Therefore, new ICCP systems need only to be designed for corrosion prevention rather than corrosion protection. In addition, other forms of corrosion management should be considered, such as monitoring only, concrete repairs, galvanic anodes etc (Christodoulou et al. 2009). Alternatively, the failed ICCP systems can just be periodically monitored until corrosion activity becomes significant and the ICCP system can then be renewed. Overall, this approach should result in reduced refurbishment and maintenance costs of ICCP systems.

CONCLUSIONS
The site data presented here is consistent with the laboratory and other results, indicating a persistent protective effect after the interruption of the ICCP systems. More specifically we conclude:

1) After 52 months with no ICCP current, all the structures investigated have remained passive, including cases where 60% of the test locations exceeded 1% chloride concentration at the depth of steel reinforcement. This supports previous laboratory evidence suggesting that ICCP does not only arrest ongoing corrosion but it also prevents future corrosion.
2) The polarisation resistance, steel potential and impedance data show that ICCP protects reinforced concrete structures not only by shifting potentials to negative values (i.e. pitting potential model) but also by transforming the steel-concrete interface.
3) The absence of corrosion should be taken into account when repairing old CP systems. Replacement anode systems need only to be designed for corrosion prevention rather than corrosion protection. Other forms of risk management include just having corrosion rate monitoring on its own as opposed to a repair of the ICCP system.

REFERENCES
The use of ternary binders in self-compacting concrete (SCC)

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Introduction

In recent years concrete producers and users have made great strides in reducing the material’s carbon footprint. Measures have included more efficient manufacture of Portland cement, whose production accounts for about 5% of the total of man-made greenhouse gas emissions [1], and the increasing use of blended cements with significant quantities of fly ash (FA), ground granulated blastfurnace slag (GGBS) and limestone powder (LP). There is a wealth of information regarding the use and behaviour of these additions in concrete, dating back several decades [2-5]. FA and GGBS both participate in the hydration reactions and can increase long-term strength, reduce heat of hydration and improve durability. However their use results in lower strength at earlier ages. The effects of limestone powder, which is nearly inert, are mainly physical. Advantages are accelerated hydration reaction at early age due to the increased surface area for reaction (nucleation) and improvement in consistence (filler effect); disadvantages are reduction in long-term strength (dilution effect) and worsened durability characteristics. However over the years the use of limestone powder in concrete has to a significant extent been side-lined due to the superior advantages of ggbss and fly ash with respect to strength and durability of the concrete.

Over a similar period advances in admixture technology has enabled concrete with enhanced workability (or consistence), such as self-compacting concrete (SCC), to be developed and increasingly used [6]. Advantages include reduced concrete placing times, reduced noise and improved quality of construction. Such concrete requires high quantities of Portland cement or similar powder together with admixtures such as superplasticisers to achieve a satisfactory combination of fluidity and stability. There is therefore the potential for using greater quantities of FA, GGBS and LP than in conventional non-vibrated concrete.

The majority of the studies in this area to date have been concerned with binary blends of the powders (Portland cement + secondary/filler powder). However, being able to achieve satisfactory properties with ternary and or even quaternary blended binders creates greater opportunities for the reduction of Portland cement and a significant amount of research has also been carried out on such concretes [7]. However there is a relative lack within this of rigorous studies of concrete with Portland cement, LP and either FA or GGBS as the third binder. Most of the studies on this have been carried out only within the past 5 years [8]. There were a number of earlier studies on SCC with ternary binders, but these were more focused on the application of the concrete rather than looking at the variations and effects on the fresh, hardened mechanical and durability properties of the concrete [9].

This study is therefore examining the effect of a range of ternary binder compositions (consisting of Portland cement, limestone powder and ggbss or fly ash) on the hardened mechanical and durability properties of SCC mixes. 100% Portland cement and binary mixes with each of the three additions have also been tested for comparison. The initial stages of the work established suitable optimum combinations of fine and medium grade limestone powder for the filler effect (75/25 fine/medium), and suitable concrete proportions for mid-range self-compacting properties i.e. a slump flow of 650 – 750mm, a V-funnel time of less than 8s, a J-ring step height of less than 15mm.
This paper summarises the results obtained thus far on compressive strength and rapid chloride permeability of all mixes at ages of up to 91 days.

Materials

The materials used were a CEM I 52.5N Portland cement, a Class F fly ash, GGBS and 3 batches of limestone powder with varying fineness. The fine aggregate was river sand (0/4) and the coarse aggregate was uncrushed 4/10 and 10/20 marine aggregates. A high-range water reducing superplasticiser (sp) and a viscosity-modifying agent (vma) were also used.

Experimental Methods

All the concrete mixtures prepared had the same volume fraction of coarse aggregates (35%), fine aggregate (30%), water (18%) and binder (sum of all powders) (17%). During the study 20 SCC mixes with various binder compositions were prepared and tested. The fresh properties of plastic viscosity and yield stress (using the two-point workability test) and slump flow, V-funnel time and J-ring step height (with the appropriate BS EN 12350 tests) were measured for each mix. For the hardened mechanical properties, 100mm cubes, 200x100mm dia. cylinders and 100x100x500mm prisms were cast for testing. After casting the specimens were moist cured at room temperature (20 ± 1°C) for 24 h and then demoulded and immersed in water at 20 ± 1°C until testing. At 1, 3, 7, 28, 56 and 91 days, the ultrasonic pulse velocity and compressive strength of the cubes (average of 3 cubes) and the resonant frequency test (for dynamic elastic modulus) of the prisms was measured. The cylinder splitting strength was determined at 28 and 91 days (average of 3 cylinders), and the sorptivity at 7, 28 and 91 days (average of 3 cubes). An extra cylinder was cast and cut into 3 discs of 50mm thick for performing the rapid chloride permeability (RCP) test according to ASTM C1202 [10] at 7, 28 and 91 days. Only the results of the cube compressive strength and the RCP test are presented and discussed in this paper.

Results

Compressive strength

<table>
<thead>
<tr>
<th>Mix</th>
<th>Binder composition (% by volume)</th>
<th>Cube Compressive Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PC LP GGBS FA 1 3 7 28 56 91</td>
<td>Age (days)</td>
</tr>
<tr>
<td>1</td>
<td>Control 100% - - - - - - - -</td>
<td>33.3 49.6 59.7 66.4 70.1 73.6</td>
</tr>
<tr>
<td>2</td>
<td>90% 10% - - - - - - - - - -</td>
<td>26.3 43.5 50.3 59.1 74.2 73.6</td>
</tr>
<tr>
<td>3</td>
<td>80% 20% - - - - - - - - - -</td>
<td>25.4 36.1 40.8 47.0 53.3 66.2</td>
</tr>
<tr>
<td>4</td>
<td>70% 30% - - - - - - - - - -</td>
<td>20.5 29.8 41.2 49.5 57.5 64.4</td>
</tr>
<tr>
<td>5</td>
<td>GGBS 80% - - - - - - - - -</td>
<td>25.2 43.5 54.4 59.4 70.9 73.6</td>
</tr>
<tr>
<td>6</td>
<td>60% - 40% - - - - - - - - -</td>
<td>16.5 32.3 42.2 62.6 64.9 69.8</td>
</tr>
<tr>
<td>7</td>
<td>40% - 60% - - - - - - - - -</td>
<td>11.1 25.0 35.4 46.1 56.4 60.9</td>
</tr>
<tr>
<td>8</td>
<td>20% - 80% - - - - - - - - -</td>
<td>5.7 21.8 30.0 44.0 46.4 48.1</td>
</tr>
<tr>
<td>9</td>
<td>Fly Ash 80% - - - - - - - -</td>
<td>28.2 43.5 45.9 56.5 67.9 71.0</td>
</tr>
<tr>
<td>10</td>
<td>60% - - 40% - - - - - - - -</td>
<td>16.9 29.9 37.5 50.2 58.7 61.3</td>
</tr>
<tr>
<td>11</td>
<td>40% - - - 60% - - - - - - -</td>
<td>4.1 12.2 16.6 28.0 32.8 37.2</td>
</tr>
<tr>
<td>12</td>
<td>- - - 80% - - - - - - - - -</td>
<td>1.4 3.6 4.9 7.6 10.9 14.3</td>
</tr>
<tr>
<td>13</td>
<td>TB-GGBS 64% 16% 20% - - - - -</td>
<td>14.0 31.1 40.1 54.1 70.8 76.6</td>
</tr>
<tr>
<td>14</td>
<td>48% - 12% 40% - - - - - - -</td>
<td>7.8 26.0 38.6 54.3 70.2 73.3</td>
</tr>
<tr>
<td>15</td>
<td>32% - 8% 60% - - - - - - -</td>
<td>5.8 22.2 34.7 49.2 60.3 62.7</td>
</tr>
<tr>
<td>16</td>
<td>16% - 4% 80% - - - - - - -</td>
<td>3.3 19.0 29.7 42.0 49.5 50.1</td>
</tr>
<tr>
<td>17</td>
<td>TB-Fly Ash 64% 16% - - - - -</td>
<td>14.4 33.4 38.5 65.5 68.1 73.5</td>
</tr>
<tr>
<td>18</td>
<td>48% - 12% - - - - - - - - -</td>
<td>6.3 18.3 25.0 41.8 48.0 51.2</td>
</tr>
<tr>
<td>19</td>
<td>32% - 8% - - - - - - - - -</td>
<td>2.0 8.4 12.2 25.6 31.0 34.6</td>
</tr>
<tr>
<td>20</td>
<td>16% - 4% - - - - - - - - -</td>
<td>0.6 2.1 3.1 6.7 9.1 10.2</td>
</tr>
</tbody>
</table>

Table 1: Cube compressive strength of the SCC mixes
The results of the cube compressive strength for all the mixes are given in Table 1. As mentioned above, the use of ggbs and fly ash in concrete results in the reduction in early strength and an improvement in the long-term strength. This is evident from the results of the binary mixes 5-8 (ggbs mixes) and 9-12 (fly ash mixes); as the replacement levels increase the early-age strengths (1 and 3 days) progressively decrease. Looking at the long-term (91 day) strength, the results show that the ggbs mix with a replacement level of 40% (mix 6) and the fly ash mix with 20% replacement level (mix 9) both have the same strength as the control mix (mix 1) whereas at higher replacement levels both ggbs and fly ash achieve strengths lower than the control mix. This confirms that the optimum replacement levels for ggbs and fly ash are 50% and 30% respectively. As for the limestone mixes (mix 2-4), as the addition level increases both the early-age strength and long-term strength decrease (due to the dilution effect) and are, at all ages, lower than the control mix apart from mix 2 (10% replacement level) which at 91 days surpassed the control mix by 4MPa. With the ternary binder (TB) mixes (13-20), mixes with ggbs (13-16) had lower strengths relative to binary binders with same replacement levels up to 28 days however at ages 56 and 91 day the strengths of the ternary binder mixes surpassed that of the binary mixes at all replacement levels (by about 4 MPa) with mixes 13 and 14 (20% and 40% ggbs) also surpassing the strength of the control mix (by 6.5 and 3 MPa respectively). As for the ternary binder mixes with fly ash, all the mixes gave lower compressive strength relative to the binary mixes at all ages with the exception of mix 17 (20% fly ash) which had a higher compressive strength (up to 9 MPa higher) than its corresponding binary binder mix at 28, 56 and 91 day.

RCP test

Table 2 shows the RCP test results obtained for the different concrete mixes. It can be seen that all binary mixes with limestone powder gave a higher value of charge passed relative to the control mix meaning the concrete more permeable to chloride ions and hence less durable. Binary mixes with ggbs and fly ash generally had lower chloride permeability, with mix 6 (40% ggbs) and mix 9 (20% fly ash) performing better than other mixes; these are close to their optimum replacement levels. With the ternary binder mixes, the permeability increased due to the presence of limestone for mixes with 20 and 40% replacement, for both ggbs and fly ash. Mixes with ggbs and fly ash at replacement levels of 60 and 80% showed improvements chloride permeability.

<table>
<thead>
<tr>
<th>Mix</th>
<th>Binder composition (% by volume)</th>
<th>Charge passed (Coulombs)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PC LP GGBS FA Age (days)</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>Control 100% - - - 0%</td>
<td>5650 2774 1704</td>
</tr>
<tr>
<td>2</td>
<td>90% 10% - - 20%</td>
<td>8806 3636 2603</td>
</tr>
<tr>
<td>3</td>
<td>80% 20% - - 20%</td>
<td>12180 10090 4929</td>
</tr>
<tr>
<td>4</td>
<td>70% 30% - - 30%</td>
<td>5486 4712 2513</td>
</tr>
<tr>
<td>5</td>
<td>GGBS 80% - 20% - 20%</td>
<td>5044 2857 2026</td>
</tr>
<tr>
<td>6</td>
<td>60% - 40% - 40%</td>
<td>4114 1454 611</td>
</tr>
<tr>
<td>7</td>
<td>40% - 60% - 60%</td>
<td>3135 1594 700</td>
</tr>
<tr>
<td>8</td>
<td>20% - 80% - 80%</td>
<td>1240 621 626</td>
</tr>
<tr>
<td>9</td>
<td>Fly Ash 80% - 20% - 20%</td>
<td>5762 2403 585</td>
</tr>
<tr>
<td>10</td>
<td>60% - - 40% - 40%</td>
<td>9206 2701 793</td>
</tr>
<tr>
<td>11</td>
<td>40% - - 60% - 60%</td>
<td>8333 2283 1056</td>
</tr>
<tr>
<td>12</td>
<td>20% - - 80% - 80%</td>
<td>7548 4560 3205</td>
</tr>
<tr>
<td>13</td>
<td>TB- GGBS 64% 16% 20% - 20%</td>
<td>7257 3491 2347</td>
</tr>
<tr>
<td>14</td>
<td>48% 12% 40% - 40%</td>
<td>2237 1448 990</td>
</tr>
<tr>
<td>15</td>
<td>32% 8% 60% - 60%</td>
<td>829 573 446</td>
</tr>
<tr>
<td>16</td>
<td>16% 4% 80% - 80%</td>
<td>371 362 227</td>
</tr>
<tr>
<td>17</td>
<td>TB- Fly Ash 64% 16% - 20%</td>
<td>9821 1839 1223</td>
</tr>
<tr>
<td>18</td>
<td>48% 12% - 40% 40%</td>
<td>9454 1903 951</td>
</tr>
<tr>
<td>19</td>
<td>32% 8% - 60% 60%</td>
<td>10570 2750 1009</td>
</tr>
<tr>
<td>20</td>
<td>16% 4% - 80% 80%</td>
<td>11500 3290 2878</td>
</tr>
</tbody>
</table>

Table 2: RCP test results
Discussion and conclusions

The use of limestone powder as a replacement of Portland cement (binary binder) results in the reduction in compressive strength at all ages (above 10% replacement) and also makes the concrete less durable in terms of chloride permeability with higher charge passing through it (RCP test) (lower resistance to ionic movement). Both ggbs and fly ash binary binder mixes showed a reduction in compressive strength at early ages however an increase was seen at later ages (up to 91 days) in addition to reduced chloride permeability improving the durability of the concrete resulting in a lower value of charge passing. The greatest improvements, in both compressive strength and chloride permeability were shown in the mix with 40% ggbs and the mix with 20% fly ash replacement. As for the ternary binder mixes, all mixes showed a reduction in compressive strength up to 28 days relative to their binary mixes with all the ggbs mixes and mix with 20% fly ash showing improvements thereafter. The values of charge passed (at 91 days) increased for mixes containing ggbs and fly ash at 20 and 40% (however still lower than the control mix) relative to the binary binder mixes which could be attributed to the presence of limestone powder; however the values were lower at replacement levels of 60 and 80%.

In conclusion, the use of limestone powder in a ternary binder mix with ggbs and fly ash does offer advantages in both the compressive strength of the concrete and improves chloride permeability. This could be due to the filler effect and the nucleation of hydration by the limestone powder increasing the rate of reaction of Portland cement consequently increasing the rate of the secondary reaction between the calcium hydroxide produced and the ggbs or fly ash.

The results and those of the other tests have yet to be fully analysed, and it is expected that clearer and more comprehensive conclusions will emerge, which will hopefully give a sound basis for the use of ternary blends in self-compacting concrete, and by implication, other high consistence concretes.

References

Introduction

As we move closer and closer to zero (operational) carbon homes, the question of embodied carbon will become increasingly more important in the domestic carbon debate and the construction industry at large. In 2008, England produced 165.1 million tonnes of waste across the domestic, industrial and commercial sector. The largest contributor was the construction, demolition and excavation sector, generating 49% of that figure and estimated to account for around 3% of all direct UK emissions [1].

Although the Coalition have abandoned the new homes target of 3 million by 2020 set by Labour, affordable housing developments are on the agenda to be pushed over the next 10 years [2]. With this in mind, it is more important than ever that we find a way to retain the embodied energy and resources that are locked into these new homes. One solution to this is design for deconstruction (DfD). One of the major manifestos in design for deconstruction is on the lifespan of a building versus the durability of the materials or products it is comprised of [3]. These discrepancies can be overcome with intelligent design that enables building fabric to be removed unadulterated. Stewart Brand’s layering diagram (figure 1) goes some way to explaining the solution, illustrating the independent layering of building elements to facilitate the maintenance, upgrade or removal for reuse of components [4]. Timber frame construction is the fastest growing method of construction in the UK. There are many drivers for this, not least the huge environmental benefits of utilising this material. A typical 100 square metre timber framed dwelling saves approximately 4 tonnes of carbon dioxide when compared to an equivalent masonry dwelling, which is equivalent to the emissions produced by driving 14 000 miles [5].

Figure 1: Shearing layers of change, Stewart Brand [3]
Recent Political Drivers

There have been two major step changes to the political backdrop in the last 7 years. Following London’s successful Olympics bid in 2005, the London Organising Committee of the Olympic Games and Paralympic Games Ltd (LOCOG) launched a raft of sustainability targets under their ‘One Planet 2012’ philosophy in which waste and materials play a leading role [6]. Ambitious, yet deliverable, key performance indicators (KPIs) were outlined for material reuse, reclamation and recycling in both the demolition phase and the construction phase and the LOCOG and the Olympics Delivery Authority (LDA) release regular reports on their progress [ibid]. In 2010 the new Coalition Government pledged to be the ‘greenest Government yet’ [7]. The UK’s capacity for renewables has grown under the Coalition Government from virtually none to delivering almost 10% of the total electricity requirements last year. [8] The recent Clean Energy Ministerial, hosted in the UK and co-chaired by Energy Secretary, Edward Davey, has begun to address global operational energy and carbon emissions with fervour [9]. The Coalition’s extensive overhaul of Policy has also begun to focus on embodied carbon. Material reuse has been included in many Government documents: ‘2010 Strategy for Sustainable Growth’, [10] ‘Securing Britain in an Age of Uncertainty: The Strategic Defence and Security Review,’ [11] as well as the Climate Change Act 2008 [12].

Aim

This paper aims to outline the new political context for design for deconstruction and reuse of building materials. It will describe the methodology utilised to carry out the body of underlying research and goes on to discuss some of the pertinent issues uncovered throughout the collation of the thesis.

Primary Research Thesis

The research base for this discussion paper was undertaken as part of an MSc in Sustainable Buildings: Performance and Design at Oxford Brookes and with the help of Stewart Milne Timber Systems who provided open access to the Sigma Home factory and drawing documentation for use as a case study. A context review investigated existing sustainable housing codes and legislation in the UK to determine any drivers or barriers to DfD and the accompanying literature review discussed the viability of the reuse of existing timber elements, the theory of DfD and historic precedents. These exercises facilitated an inventory of DfD principles and a SWOT analysis that outlined the strengths, weaknesses, opportunities and threats of DfD [13]. The case study analysis began with an examination of the manufacturing process which was tested against the inventory of principles developed under the literature review. The design and construction of the case study was summarised from drawing packages, specifications and information gleaned from conversations on site at the factory. Once the information had been collated, key areas were appraised for potential for DfD improvement as a process driven exercise and embodied energy calculations were performed for each component to verify the appropriateness of the appraisal. The aim was to identify a suitable evaluation process for the creation of a DfD hierarchy in a project. Upon identification of DfD sensitivity, a series of solutions were designed and tested against the Stewart Milne manufacturing process and the deconstruction criteria as defined earlier in the research. This detail was then costed to check the financial viability of the DfD detail against the existing one [ibid].
Consumer Product Agreements vs. Lifespan of Buildings

Extended producer responsibility involves a shift from selling a product to selling a service and is sometimes referred to as dematerialisation. An example of a company employing this ethos is Interface, a carpet manufacturer in the USA. The business model is such that they no longer sell carpet but lease it to clients, who return it at the end of its useful lifespan for melting and re-spinning. This eradicates the waste in this system, while providing the raw materials for the next product [14]. Due to the adaptability of this concept and the solutions for minimising waste that this business model facilitates, it is anticipated that similar legislation will be introduced into the construction industry in the future. Introducing this model into housing is a little more complicated as each house is expected to have a minimum service life of 60 years. This means that there are few economic drivers for the company in terms of material recovery. However if a maintenance extended producer responsibility model was implemented, this would generate revenue through the inherent need for adaptation in the home [15].

Materials

There already exists a body of work produced by many DfD proponents globally, many of them in the US. A large amount of the DfD detailing and specification work carried out so far has focused on layers such as the external finish [16]. Since the structure of a building tends to have the longest lifespan and is often comprised of the largest members, an alternative approach was adopted for the research and the timber structural elements of the case study were chosen for DfD analysis. An interview with the owner of a reclamation yard highlighted key points for consideration that are appropriate for DfD most notably; reclaimed materials do not come with structural guarantees or warranty which means that specimens that would be suitable for structural purposes are rarely utilised as they need to be signed off by a structural engineer. The second significant point is that, as a rule, smaller, softwood timber members have replaced larger, hardwood timber members; softwood being considered a less superior material. In light of this, DfD emphasis must be attuned to modular, component deconstruction - i.e. of wall, floor and roof cassettes - as opposed to individual member deconstruction in the contemporary world.

Infrastructure

The main barriers to a DfD paradigm shift are the absence of product demand and a framework to support DfD which would include a network of storage and distribution centres. This type of network does already exist in the form of the reclamation industry and Salvo-Mie and BioRegional and the National Community Wood Recycling Project have been working for some years to establish both Reclamation Megayards and ReIY shops as models for reuse infrastructure [17]. Site waste management plans are a recent driver with the potential to make deconstruction a principal part of a project. The difficulty posed by the recording of material information and history is a major barrier to the full implementation of this. If the information were to be kept within the building documents, it would give rise to the possibility of losing information. Another solution is to tag each member, and this would require a robust central framework to hold the data [18]. While this has been seen as a far-fetched solution in the recent past, this process is becoming increasingly more financially feasible, with some companies already offering the service [19]. This has many potential links with the current Building Information Modelling (BIM) ethos.
Conclusion

The context for deconstruction and material reuse is one that changes rapidly. The legislative landscape has evolved dramatically since the outset of the research at the end of 2009 due to a refocus from a new Government and the introduction of site waste management plans. Models for a supporting infrastructure for reuse are being tested and an economically feasible capacity for documenting material history is becoming a reality. Consumer pressure may be the last piece in the jigsaw to motivate an authentic change in approach to design for material reuse.

References


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A rapid quantitative determination of polymer modified bitumen storage stability by FTIR

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* Corresponding author

Introduction

Polymer modification of bitumen is widely used to improve both the high and low temperature properties of asphalt. However, it is known that commonly used polymers, such as styrene-butadiene-styrene (SBS), only have limited compatibility with bitumen [1], with the precise degree of incompatibility dependent upon bitumen origin and grade, polymer type and concentration, and binder storage conditions. Many commercially available modified binders are stabilised by proprietary processes, but testing is still required as part of quality control and CE-marking to ensure their performance, with the reference test method giving an empirical indication of polymer stability in the bitumen. In this study, a method is described using Fourier Transform Infra Red (FTIR) spectroscopy, which provides a quantitative measure of the polymer stability and is also faster and more practical to perform.

Polymer modified binder production

A commercially available linear SBS copolymer, Kraton 1101, was used to modify 50/70 penetration grade bitumen. Bitumens from Venezuelan, Middle Eastern, and Russian crude sources were all modified using SBS polymer contents of between 2.5% and 7.5% w/w, which is typical of the range used in paving applications. The modified binder did not include any additional stabilising package so that later storage stability testing would highlight the direct effects of polymer concentration and crude type. The polymer was added to the bitumen at 180°C to produce a 4 kg batch in a heated mixing vessel and subjected to high shear mixing for two hours at 180°C before removing from the vessel, subdividing into smaller containers and allowing to cool prior to subsequent testing.

Classical empirical testing

The polymer modified binders were tested using the classical empirical tests of penetration, softening point and dynamic viscosity, reported in table 1.

<table>
<thead>
<tr>
<th>Bitumen origin</th>
<th>Venezuelan</th>
<th>Venezuelan</th>
<th>Venezuelan</th>
<th>Middle East</th>
<th>Russian</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polymer content</td>
<td>2.5%</td>
<td>5.0%</td>
<td>7.5%</td>
<td>5.0%</td>
<td>5.0%</td>
</tr>
<tr>
<td>Penetration at 25°C (100g, 5s)</td>
<td>46 dmm</td>
<td>42 dmm</td>
<td>31 dmm</td>
<td>40 dmm</td>
<td>31 dmm</td>
</tr>
<tr>
<td>Softening Point</td>
<td>56.4°C</td>
<td>84°C</td>
<td>93°C</td>
<td>77.6°C</td>
<td>84.5°C</td>
</tr>
<tr>
<td>Dynamic viscosity at 150°C</td>
<td>540 cP</td>
<td>1210 cP</td>
<td>2570 cP</td>
<td>1320 cP</td>
<td>1230 cP</td>
</tr>
</tbody>
</table>

Table 1 Empirical testing of polymer modified binders

With increasing polymer content in the binder the softening point (SP) increases, but in a non-linear manner. Other SBS polymer contents were used to modify the bitumen in addition to the ones reported in table 1. These are shown in figure 1 to demonstrate the non-linear increase in SP. At low polymer contents, typically less than 4%, the polymer domains are discrete within the bitumen leading to relatively modest increases in SP. However, at higher polymer contents the polymer domains become continuous leading to a sharp increase in the SP. Bitumen origin also has an effect
on the precise shape of this curve, hence SP may only be considered a qualitative measure of polymer content in a binder.

![Figure 1 The effect of polymer content on softening point](image)

**European standard methodology for polymer modified bitumen storage stability test**

The European standard method for polymer modified bitumen storage stability testing is EN13399 [2], and is commonly known as the ‘toothpaste tube test’. In this method, binder is poured into an aluminium tube approximately 25mm diameter by 160mm high to about three quarters full. The end of the tube is sealed and the tube stored vertically in a heated oven for 3 days, after which it is removed and allowed to cool. Once cooled the sample is sectioned in three equal parts, with the top and bottom retained for testing and the middle section discarded. In the standard test, the softening point of the top and bottom sections is measured and the difference between the two results indicates the storage stability of the binder. In an unstable binder, the polymer will tend to migrate to the top of the tube and lead to an increase in the SP relative to the bottom. The binders produced in this study were deliberately not stabilised and, as expected, this was reflected in the storage stability testing reported in table 2. Whilst all of the binders are unstable, the Russian bitumen shows a considerably lower difference in SP than the Venezuelan and Middle Eastern bitumens indicating better natural compatibility with the polymer.

<table>
<thead>
<tr>
<th>Bitumen origin</th>
<th>Venezuelan</th>
<th>Venezuelan</th>
<th>Venezuelan</th>
<th>Middle East</th>
<th>Russian</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polymer content</td>
<td>2.5%</td>
<td>5.0%</td>
<td>7.5%</td>
<td>5.0%</td>
<td>5.0%</td>
</tr>
<tr>
<td>Softening Point</td>
<td>Top 83.7°C</td>
<td>102.1°C</td>
<td>106.3°C</td>
<td>114.6°C</td>
<td>96°C</td>
</tr>
<tr>
<td>Bottom 61.5°C</td>
<td>65.8°C</td>
<td>67.4°C</td>
<td>68°C</td>
<td>74.2°C</td>
<td></td>
</tr>
<tr>
<td>Difference 22.2°C</td>
<td>36.3°C</td>
<td>38.9°C</td>
<td>46.6°C</td>
<td>21.8°C</td>
<td></td>
</tr>
</tbody>
</table>

Table 2 Softening Point storage stability results

**Fourier Transform Infra Red (FTIR) background and testing**

In infra red spectroscopy, the absorbance measured for each molecular vibration varies linearly with the path length and the concentration of the species, and is known as Beer’s Law (or sometimes Beer-Lambert’s Law) given in equation 1.

\[
\text{Absorbance (A) = Molar absorbtivity (ε) X Path length (l) x Concentration (c)}
\]  

Equation 1

Bitumen is a complex hydrocarbon and shares many of the same bond structures with SBS. Fortunately there are two comparatively strong absorbing bands in the fingerprint region, which are found in SBS and not bitumen, allowing quantitative measurements of polymer content to be made.
These are highlighted in figure 2 and are at 966 cm\(^{-1}\) C-H out-of-plane bending of trans-alkene (butadiene), and 699 cm\(^{-1}\) C-H out-of-plane bending in monoalkylated aromatics (styrene).

**Figure 2** FTIR Spectra of 5% SBS in Venezuelan Bitumen, with the styrene and butadiene peaks highlighted.

Previous studies [3,4,5] have assessed the polymer content of bitumen in transmission by dissolving samples of the binder in solvent. The difficulties with this approach are that it requires considerable time and care to accurately produce samples, the clean-up time is longer and there are issues regarding solvent disposal.

An alternative method which is particularly suitable for bitumen is the attenuated total reflection (ATR) method [6]. The sample under test is in intimate contact with the surface of the ATR crystal. The IR beam passes into the ATR crystal at an angle exceeding the critical angle and is then reflected back to a detector for collection, as shown schematically in figure 3.

**Figure 3** Schematic of the single bounce diamond ATR and photograph demonstrating the small sample size required.

The ATR method may be used with a variety of crystal materials, such as Zinc Selenide, Germanium, and Silicon. These materials all perform well, but can suffer from being difficult to clean without damaging the crystal. An alternative material is diamond; the price of diamond ATR cells has recently decreased an affordable level for most laboratories. Diamond cells have the advantages of requiring only a small sample size and of being structurally robust and chemically resistant resulting in clean-up and, consequently, sample turnaround time being significantly reduced. Their disadvantage, apart from cost, is the low transmission in the 2300 to 1800 cm\(^{-1}\) range, but for most bitumen analysis this has little consequence. In the ATR method the infra red beam will travel the same distance for each sample under test. The path length \(l\) in equation 1 is therefore constant and by analysing known polymer content samples, which in this case were already prepared, \(\varepsilon\) may be determined and a calibration curve of absorbance against polymer concentration obtained.
Methodology for FTIR analysis of polymer modified bitumen storage stability

As the samples were heated to pour out for softening point testing, a small amount of sample was mounted on the FTIR-ATR diamond cell and its spectrum recorded. This required much less than 0.5g of bitumen and was simple to perform. By using the calibration factors determined earlier, it was then possible to quantitatively measure the polymer content of the sample. Furthermore, from a practical standpoint, the time taken to perform the test and clean the FTIR ready for subsequent tests was around 5 minutes. This should be compared to a softening point test which, particularly with high softening point binders, could take up to an hour to complete.

<table>
<thead>
<tr>
<th>Bitumen origin</th>
<th>Venezuelan</th>
<th>Venezuelan</th>
<th>Venezuelan</th>
<th>Middle East</th>
<th>Russian</th>
</tr>
</thead>
<tbody>
<tr>
<td>Original binder</td>
<td>2.5%</td>
<td>5.0%</td>
<td>7.5%</td>
<td>5.0%</td>
<td>5.0%</td>
</tr>
<tr>
<td>Polymer content</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Top</td>
<td>6.9%</td>
<td>14.5%</td>
<td>19.9%</td>
<td>16.8%</td>
<td>12.7%</td>
</tr>
<tr>
<td>Bottom</td>
<td>1.6%</td>
<td>0.9%</td>
<td>0.6%</td>
<td>0.8%</td>
<td>1.2%</td>
</tr>
<tr>
<td>Difference</td>
<td>5.3%</td>
<td>13.6%</td>
<td>19.3%</td>
<td>16.0%</td>
<td>11.5%</td>
</tr>
</tbody>
</table>

Table 3 Quantitative polymer storage stability results by FTIR

The degree of separation reported in table 3 reflects that observed in the empirical testing earlier. However, by having a quantitative measure of the polymer content, the crude source effect is much more apparent, with the Russian having the best compatibility and Middle Eastern the poorest.

Conclusions

This work has proposed to quantitatively measure the polymer content of polymer modified bitumen using an infrared attenuated total reflection procedure with a diamond cell. Furthermore, the method has been demonstrated as a more rapid quantification alternative to the empirical softening point measurement in storage stability testing.

References

Structural limecrete: an investigation into the potential of hydraulic lime-concrete using pozzolanic and latent hydraulic additions

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Introduction

The cement industry is reported to be responsible for about 5% of the total global anthropogenic carbon emissions. With the demand for cement growing year on year and increasingly efficient manufacturing technologies able to offer less and less savings, the hunt is on for alternative binders, and concretes, that are less carbon intensive [1].

The potential for a structural lime-concrete has been noted, ‘The science of using lime concrete in a similar way to Portland Cement concrete for structural frames has not been developed. There is however, considerable potential for further research and development of lime concrete for its application as an appropriate building technology’[2]. More recently, a review of ‘industrially interesting approaches to “low-CO2” cements’, highlighted the potential for developing clinkers ‘with a lower alite and higher belite content’[3]. Belite, the predominant hydraulic compound in NHL5, is both produced at a lower kiln temperature to alite (the predominate hydraulic compound in Portland cement) and requires less raw material feedstock.

Recent research into the production of hydraulic lime-concretes has reported maximum 28-day cube strengths of 16N/mm² cured at 65% RH[4]. However, the research reported in this paper has demonstrated that it is possible to attain 28-day cube strengths of approximately 50N/mm² at 65% RH, by combining natural hydraulic lime with amorphous aluminosilicate industrial by-products. The feasibility of attaining strengths comparable with Portland cement concretes is thought to be a step-change for research in this area.

This paper presents an overview of the research conducted at the University of Bath, highlighting the key results. Two industry projects are briefly introduced to contextualise the research intent and to qualify the potential impact of the findings.

Context

This research project was initiated by an architect who envisioned a lime-concrete shell roof for a private residence in the Cotswolds. This innovative structural solution was central to a planning application, which demanded a ‘truly outstanding and groundbreaking’ scheme[5]. Demand led, this industry based research programme continues to be shaped by individuals and organisations with an interest in the developing technology.

Most recently a bespoke polished lime-concrete floor screed has been developed for a school in Bath. The material was adapted to incorporate frost-shattered limestone, taken from the school site, as a sustainable aggregate. Trials undertaken, in conjunction with a specialist contractor, demonstrated the feasibility of polishing the lime-concrete to expose the site material as a decorative surface finish.
Such applications have only been rendered conceivable, by the high early strength gain that this research project has demonstrated is achievable in ambient conditions. The slow-strength gain which is so commonly associated with the use of lime in construction, is likely to remain a barrier to the adoption of lime-concretes long after the contrary has been demonstrated in the laboratory.

It should be noted that before the advent of Portland cement in 1824 that lime was the predominant binder for use in construction. The time-honoured practice of ‘gauging’ lime mortars, and concretes, with pozzolanic materials, originated with the Greeks. Many ancient Greek, and Roman structures, most famously the Pantheon, remain today and are a testament to the durability of lime-concretes. However, the research described in this paper does not reflect a return to former technologies, but rather builds upon the substantial developments in concrete technology that have taken place in recent years. Specifically the development and utilisation of highly reactive pozzolans (e.g. silica fume) and the development of polycarboxylate-ether based superplasticizers and other admixtures.

Materials

This research was conducted using naturally hydraulic lime (NHL5) from St. Astier in France. Such eminently hydraulic limes are no longer manufactured in the UK. The pozzolanic and latent hydraulic additions that have been used in this investigation include: silica fume, fly ash, ground-granulated blastfurnace slag, metakaolin and brick dust. The lime-concrete samples were typically prepared using a siliceous sand and a carboniferous limestone aggregate, both of which were dried to a laboratory dry state before use.

Experimental method and key findings

There were a large number of possible pozzolanic and hydraulic additions, and combinations thereof, which were identified as potential options for the partial replacement of the NHL5 (by weight). Testing, of twenty-two lime mortar mixes, was used to identify four promising blends, which yielded the highest compressive strengths. A large range of strengths, tested at 7, 28, 56 and 90 days, demonstrated the relative contribution of the different additions. The majority of the additions were seen to have a beneficial affect on strength development, outperforming mortars prepared with NHL5 alone. A number of the mortars were substantially stronger over a 28 day period.

Four of these promising mixes were then scaled up to investigate the affect of the water-to-binder (w/b) ratio and curing regime on the strength and durability of the resulting lime-concretes. As well as strength development, testing also considered the linear shrinkage, rate of carbonation and elastic modulus of the samples. Considering both the mechanical performance and the commercial viability of the mixes, a single combination of additions was selected for further trials.

Much like concrete the (w/b) ratio has been shown to have a significant affect on the resultant strength, with higher strengths achieved at lower w/b ratios. Achieving a workable fresh material at a low w/b ratio necessitated an additional investigation into the relative performance of a range of superplasticisers. Although the familiar slump test is still thought to be a poor indicator of the workability of fresh lime-concrete, a suitable proprietary polycarboxylate-ether based superplasticiser was identified that produced a workable and compactable material, when prepared at a w/b ratio of 0.35.
Since maximum strength and minimal environmental impact were the overarching goals of the research, the mix design and testing programme benefited from the insight of a range of industry stakeholders and modifications to both were made along the way. The inclusion of the superplasticiser was seen to have a marked improvement of the cube strength of the material, and 28-day strengths in excess of 49N/mm² were achieved. This was a real break through, paving the way for the production and testing of two 3m long reinforced lime-concrete beams.

The two beams were both prepared with the same lime-concrete mix, but one over- and one under-reinforced to assess the structural behaviour of the two beams in bending. The beams were tested 28 days after casting, having been cured in the laboratory at ambient temperature and humidity for this period. The predicted failure load of each beam was calculated using Eurocode 2 [7], substituting in the material properties, ascertained in the lab, and modifying the stress and strain distributions accordingly. Comparing the predicted failure loads with the actual failure loads, demonstrated the suitability of standard concrete design codes for designing lime-concrete elements.

<table>
<thead>
<tr>
<th>Beam Reference</th>
<th>28-day Cube Strength (N/mm²)</th>
<th>Theoretical maximum point load at mid-span (kN)</th>
<th>Failure load, applied at mid-span (kN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Under-reinforced (2no. H12 bars)</td>
<td>40</td>
<td>27</td>
<td>31</td>
</tr>
<tr>
<td>Over-reinforced (4no. H12 bars)</td>
<td>40</td>
<td>44</td>
<td>44</td>
</tr>
</tbody>
</table>

Table 1: Results of reinforced lime-concrete beam testing

This larger-scale structural test, importantly, gave engineer’s in Ramboll, the results needed to confidently design and substantiate the lime-concrete shell roof for the project in the Cotswolds. The results of testing in the laboratory and the calculation package, rather unusually, became a key part of the planning application for this building. At the time of writing the scheme is awaiting planning permission.

**Discussion and conclusions**

Having demonstrated the feasibility of a structural grade lime-concrete, thoughts have turned to it’s potential application, beyond the scope of the two projects for which it has been specifically developed. With the provincial lime industry all but eradicated by the growth of industrial cement manufacture in the 19th century, it is recognised that the production capacity of the lime-industry is dwarfed by that of the cement-industry. For example in 2011 France, a primary lime producer in Europe, produced around 300,000 tonnes of hydraulic lime and around 24 million tonnes of cement (St Astier, pers.comm. April 2012). By that same token the global lime industry is not under pressure, nor incentivised, to invest in new technologies that might improve the efficiency of current manufacturing processes. It is thought that potential energy savings, associated with the production of lime at lower kiln temperatures, are not currently being exploited and that the alleged environmental benefits of lime are in some cases future-orientated. If true, this represents a significant opportunity for growth in this material and industry.

The economies of scale that result in the relatively inexpensive production of cement, are thought will preclude lime based construction materials from competing in a mainstream market for the foreseeable future. However the growing market for ‘green’ materials does create opportunities for bringing lime-based products to market and extending the possible application and demand for these materials.
The lime-concrete mix developed for the shell roof is estimated to have an embodied carbon of 195kg of CO$_2$/m$^3$. The embodied CO$_2$ figures for the constituent materials used in the approximation are as follows: NHL5 (635 kgCO$_2$/tonne), SF (14 kgCO$_2$/tonne), GGBS (52 kgCO$_2$/tonne) as supplied by manufacturers. Despite having a higher overall binder content, the embodied CO$_2$ of this lime-concrete, compares favourably with both CEM1 concrete (285 kgCO$_2$/m$^3$) and typical ‘UK Concrete’ (225 kgCO$_2$/m$^3$)[8]. Further reductions in the embodied carbon of lime-concrete would be realised if the initial free water content could be reduced.

In the context of an industry responsible for the production for around 1.5 billion tonnes of CO$_2$ per year, even modest reductions in the embodied carbon of cementitious materials has the potential to save hundreds of millions of tonnes of carbon emissions every year. In the light of this, the potential savings are thought to derive less from the precise composition of alternatives materials and more from the scale at which alternative technologies are adopted in practise. It is anticipated that the total carbon emissions, associated with lime manufacture could be reduced, and that alternative pozzolanic additions may be found that increase the scope of lime-based concretes to make a significant difference to the global community.

**Further work**

A reduced environmental impact is not thought to be the only potential advantage of the use of lime-concrete in construction. Other benefits are likely to result from other material properties, which differentiate lime-concretes from cement-concretes, including the elastic behaviour, breathability and durability of this material. Further testing is required to qualify these properties and their benefits in specific building applications.

**References**


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* based on the weighted average embodied carbon of all the CEM I, II, III, and IV supplied in the UK, in 2008.
Magnesium phosphate cement: A new material for waste encapsulation

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Introduction

Currently in the UK, encapsulation using composite blends of Portland cement (PC) with supplementary materials such as blast furnace slag (BFS) and pulverised fuel ash (PFA) is the dominant technical solution for the treatment of low and intermediate level nuclear wastes (L/ILW). However, due to the high pH environment (usually around 12.5-13), certain waste metals such as aluminium can corrode, causing the formation of expansive corrosion products and the generation of hydrogen gas. Since aluminium is only passive between pH4 - 8.5, the use of an alternative low-pH cement system could serve to reduce/inhibit the corrosion \cite{1}. In recent years, magnesium phosphate cement (MPC) has generated interest as a potential alternative to PC. Due to an acid-base reaction, MPC offers alternative physical and chemical properties, most notably a near neutral pH \cite{2}.

When MgO is mixed with a suitable phosphate (e.g. KH\textsubscript{2}PO\textsubscript{4}) and water, the following reaction occurs:

\begin{equation}
\text{MgO} + \text{KH}_2\text{PO}_4 + 5\text{H}_2\text{O} \rightarrow \text{MgKPO}_4.6\text{H}_2\text{O} \quad \text{Eq.1}
\end{equation}

The hydration product, generally abbreviated to ‘MKP’, forms rapidly into a densely crystalline microstructure. However, due to this rapid and violent reaction, MPC struggles to fulfil certain requirements of waste encapsulation such as low heat output and acceptable grout working times. In this study, a number of methods were employed to investigate both the early hydration and corrosion performance of aluminium in MPC. To address the issue of heat, mineral additives such as PFA and ground granulated blastfurnace slag (GGBS) were added at 50% replacement levels in an attempt to ‘dilute’ the cement matrix. The effects of these additions on the hydration process and corrosion performance were investigated and some of the results are discussed in this paper.

Materials

The two main reactants- dead burned MgO and KH\textsubscript{2}PO\textsubscript{4} (KDP) were obtained as raw constituents from Richard Baker Harrison and Prayon UK respectively. Dead burned MgO was favoured over light burned in this system in order to reduce the reaction rate. The aluminium metal used in this study was grade 1050 of 99.5% purity. GGBS and PFA were locally sourced.

Experimental methods

Trial mixes using different M/P molar ratios were carried out to achieve specific properties as recommended by the National Nuclear laboratory \cite{3}. These included acceptable workability (mini slump value of 85 ±5mm, initial setting time > 4hrs) and good volume stability when exposed to additional moisture. As was found from literature \cite{4}, the reaction rate increased with higher M/P ratios. Small additions of boric acid were necessary to reduce the reaction rate. It was found that MgO and KDP in a molar ratio of 4.5:1 with boric acid at 4%wt of MgO best suited these initial requirements. Lower M/P ratios produced excessive expansion of the hardened specimens when unreacted KDP and MgO reacted with additional environmental moisture. The mixes chosen for this
study can be seen in Table 1. M1 was used as an MPC reference mix while M4 and M5 were necessary to compare with PC based cements (GGBS/PC 9:1 is one of the standard mixes currently used for waste encapsulation). The w/s of each of the cements was determined using mini slump trials to achieve a minimum acceptable flowability. It was important that a minimum value was used, as the water content must be as low as possible to reduce any free water that could be available for the corrosion reaction. Unlike steel in concrete, the onset of aluminium corrosion is almost immediate. For this reason, a quick DC electrochemical method (potentiodynamic polarisation) was implemented to determine the instantaneous metal corrosion rate from the moment the cement-Al sample was cast. A ‘three electrode’ configuration was adopted consisting of a working electrode (metal under study), counter electrode (required to apply the polarising current) and reference electrode (datum for potential measurements). Aluminium was used for both the working and counter electrodes with high grade stainless steel used as a reference. An embedded capacitance based RH/temperature sensor was also incorporated to provide complementary data to the corrosion rate measurements.

Potentiodynamic scans were carried out using a Gill AC potentiostat (ACM instruments). Careful consideration of the scan rate, scan range and delay time between scans were necessary before conducting the tests. For example, too low a rate could delay the return to steady state after each scan, while too high a rate may cause the charging of double layer capacitance leading to an underestimated corrosion rate [5]. If the scan range is set too high, irreversible polarisation effects may occur causing physical changes on the metal surface [6]. After some trials, a scan rate of 30mv/min through a ± 50mv range of the open circuit potential (reference v.s. working electrode) was found to be most suitable for these tests. The pH of the fresh and hardened cement pastes was measured using a standard glass electrode pH meter. For the fresh pastes, the electrode was protected by a perforated PVC sleeve and wrapped in filter paper (10micron pore size). This allowed measurement of the liquid before it was consumed in the hydration reaction. For the hardened pastes, a solution of 1 part hydrated cement (ground to 63microns) to 10 parts distilled water was prepared and stirred in a rotating stirrer for 24hrs. The solid particles were separated from the solution using a centrifuge and the pH of the liquid containing the dissolved ions was then measured [7]. Initial and final set of the cement pastes were determined using the Vicat apparatus. A multi channel isothermal conduction calorimeter was used to measure the heat of hydration of the cement pastes at 20°C over a 48hr period. The hydration products of each of the pastes were

<table>
<thead>
<tr>
<th>Cement sample</th>
<th>w/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1 100% MPC</td>
<td>0.2</td>
</tr>
<tr>
<td>M2 MPC/PFA 1:1</td>
<td>0.3</td>
</tr>
<tr>
<td>M3 MPC/GGBS 1:1</td>
<td>0.3</td>
</tr>
<tr>
<td>M4 100% PC</td>
<td>0.38</td>
</tr>
<tr>
<td>M5 GGBS/PC 9:1</td>
<td>0.42</td>
</tr>
</tbody>
</table>

**Table 1. Samples tested**

![Schematic of test apparatus](image-url)
characterised using X-ray diffraction (XRD), elemental dispersion spectroscopy (EDS) and secondary electron microscopy (SEM) analysis.

Results and discussion
Compared to conventional PC, it can be seen that MPC reacts much faster, generating most of its total heat output within the first day (Fig. 2). Both mineral additions had caused a reduction in heat of ~30% after 48hrs. It was found that the exotherms correlate well with the results obtained from the Vicat setting time tests. The initial set of M1 was 45mins however with mineral additions this was prolonged to 2hrs 35mins for GGBS and 4hrs 50mins for PFA. It seems that PFA had a considerably higher retardation effect on the initial set (also indicated by the exotherm of M2 in Fig. 2). In previous work, where a lower M/P ratio of 1:1.5 was used, the mineral replacements actually led to an overall increase in total heat output [2]. Unlike the previous study, the higher M/P ratio of 4.5:1 gave rise to better volume stability of the hydrates ensuring that MgO was the only residual reactant. Therefore the dilution effect of the mineral additions proved more effective, serving to cause a significant reduction in heat. Magnesium potassium phosphate hydrate (MKP) was identified by XRD as the main crystalline phase in all of the MPC samples. Peaks of highest intensity were observed in the M2 sample, with slightly lower intensities in M1 and M3. From literature, MPC cements are generally quoted as having a near-neutral pH between 5 and 7. While this was found true for the initial measurement, it did not hold for further measurements of up to 7 days where values in excess of pH 11 were reported from the current study. A pH increase was also observed in the previous study where values of 8-9 were reported after 2 days [2]. In this work, it is thought that the higher amount of Mg$^{2+}$ in solution mainly contributed to this pH increase. Incidentally, a recent study on Al cementation also raised this concern when formulated MPC cements were left in contact with water [8]. Graphs of Al corrosion rate over a 48hr period are shown in Fig. 3. Measured values of polarisation resistance were converted to corrosion rate using the Stern-Geary relationship [9]. These were then converted to metal loss in mm/year using Faraday’s law [5]. Compared to the PC systems, the corrosion was rapidly reduced with little to no corrosion happening within the first few hours. The inset to the right shows the MPC graphs in more detail. It seems that Al corrosion is somewhat inhibited for the first 10hrs in samples with mineral additions. This was thought to be due to a prolonged passive pH in comparison to M1, due to slower consumption of KDP. After 10hrs an acceleration in corrosion rate was observed in M2 and M3, which could be associated with a sudden pH increase. Further pH measurements at early stages may confirm this. The embedded RH sensors gave readings of 100% RH after 48hrs in M2–M5 however the internal RH of M1 was measured as 82% at the end of the corrosion experiment. This may explain why M1 had the lowest corrosion rate at the end of the test,

Table 2. pH evolution of cements

<table>
<thead>
<tr>
<th></th>
<th>Initial</th>
<th>1day</th>
<th>2days</th>
<th>7days</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1</td>
<td>6.1384</td>
<td>11.124</td>
<td>10.988</td>
<td>11.078</td>
</tr>
<tr>
<td>M2</td>
<td>5.74925</td>
<td>11.119</td>
<td>11.102</td>
<td>11.188</td>
</tr>
<tr>
<td>M3</td>
<td>5.82425</td>
<td>11.098</td>
<td>11.193</td>
<td>11.269</td>
</tr>
<tr>
<td>M4</td>
<td>13.167</td>
<td>12.851</td>
<td>12.731</td>
<td>12.751</td>
</tr>
<tr>
<td>M5</td>
<td>12.6505</td>
<td>12.281</td>
<td>12.278</td>
<td>12.284</td>
</tr>
</tbody>
</table>

Fig. 2 Total heat of hydration over 48 hrs
as most of the moisture had been consumed in the hydration reaction leaving little available for aluminium corrosion.

Conclusions
With an M/P molar ratio of 4.5:1 and the addition of small amounts of boric acid (4%wt of MgO), a magnesium phosphate cement with acceptable Al corrosion performance at the early stage was produced. The additions of PFA and GGBS caused a significant reduction in heat, with PFA prolonging the initial set to >4hrs which was desired. Magnesium potassium phosphate hydrate (MKP) was observed as the main crystalline phase in all of the MPC cements with some free MgO as a residual reactant. The mineral additions acted largely as inert fillers, which was confirmed through characterisation. It was found that the pH of the MPC cements increased dramatically after 24hrs hydration, falling outside the safe ‘passive’ range for aluminium. An associated increase in corrosion rate was observed however in comparison to the PC based matrices, the Al corrosion up to 48hrs hours was almost non-existent.

References
Compressive Strength, Hydration Products and Microstructure of Alkali-activated Fly Ash Manufacture with Pulsed Microwave Curing Method

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1 Introduction

The manufacture of Portland cement (PC) exerts significantly negative effects on the environment. Not only is PC production an energy-intensive process, but 0.93 tonne of CO₂ is released into the atmosphere per tonne of PC manufactured. A clinker-free cementitious material, alkali activated fly ash (AAFA), using fly ash as the source material and NaOH as the alkali activator, is regarded as an alternative product to the PC in industrial application [1]. However, one limitation of AAFA is that it cannot harden without oven curing. The prevailing curing temperature for AAFA is 85°C and it usually takes more than 8 hours to achieve an early strength of above 20 MPa [2]. This process is considered not environmentally friendly owing to this consumption of electrical energy and hence the emission of greenhouse gas.

Different from the conventional oven curing which depends on the thermal conductivity, microwave curing relies on the heat generated from the vibration and friction of water molecules under the alternative electromagnetic field, which can achieve volumetric heating. Therefore, microwave curing can potentially reduce the energy consumption. Some attempts have already been made in using microwave as an alternative curing technique in the manufacture of PC-based precast concrete products in order to enhance the hydration process and decrease the curing duration [3]. It has been confirmed that concrete with high early strength (27MPa) could be achieved in less than 6 hours [4]. However, the research on the manufacture of AAFA with microwave curing is scarce. Only one paper reported that microwave curing can potentially be used to manufacture AAFA mortars [5]. The curing duration and energy consumption can be decreased markedly. The results showed that the compressive strength of AAFA with microwave curing for 2 hours was higher than that cured at 75 °C for 48 hours. However, continuous microwave curing along with high power level (e.g. 1200W) led to severe cracking inside the sample within a few minutes due to the thermal gradient formed from uneven heating. Similar phenomena has been observed in the PC-based mortar manufactured with continuous microwave curing as well [6]. To address this issue, the concept of pulsed microwave heating, which has been successfully used in the food industry for resolving the un-uniform temperature distribution [7], was introduced in the current study for manufacturing the AAFA.

This paper is therefore focused on the investigation on using pulsed microwave technique to cure AAFA material. The mechanical performance, hydration products and microstructure of AAFA material were studied and compared with the products manufactured with conventional oven curing.

2 Experimental

2.1 Materials

The chemical composition of the fly ash used in this study is shown in Table 1. Sodium hydroxide of industrial grade with a purity of 95% was supplied by ReAgent.
2.2 Sample preparation
The pastes were made by mixing the fly ash with 8M NaOH solution. The solution/solid ratio was 0.3. The fresh pastes were cast into four steel moulds for conventional oven curing and four plastic moulds for microwave curing respectively. The dimension of the samples was 25×25×25mm.

Prior to the curing in a domestic microwave oven, AAFA samples were firstly pre-cured under the room temperature for 24 hours, which was found can prevent the splashing of fresh pastes during the microwave curing process. Same pre-curing regime was adopted before the conventional oven curing at 85°C in order to compare its efficiency and energy consumption with the microwave curing method.

Pulsed curing regime was used in the microwave heating in this study. The control of the duration for the “on” and “off” was through the pulsing ratio (PR), which is defined as:

\[ \text{PR} = \frac{t_{on}}{t_{on} + t_{off}} \]

Where, \( t_{on} \) and \( t_{off} \) are the duration of microwave power-on and power-off per cycle respectively. The pulsing ratio was 0.05 and each cycle lasted for 2 minutes.

After both the conventional oven curing and microwave curing, the samples were cooled down to room temperature before compressive strength tests. The debris from the compressive strength tests were stored in acetone before any further chemical analysis of the reaction products.

2.3 Analysis Methods
Three 25×25×25mm cubes were crushed, with the average being reported. Reaction products were characterised by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), whilst the microstructure was analysed through scanning electron microscopy (SEM).

3 Results and discussion
3.1 Compressive Strength
The compressive strength results of the AAFA with two different curing regimes are shown in Fig. 1. It was found that prolonged curing time had a positive effect on the mechanical strength of AAFA under 85°C oven curing. With microwave curing, the highest compressive strength of the AAFA was achieved after 30 cycles. It can be seen that the strength of the AAFA with 10 cycles microwave curing (28.16 MPa, with effective curing time, i.e. power-on time, of merely 60 seconds) was comparable to the strength obtained with oven curing at 85°C for 4 hours (28.28 MPa).

3.2 Characterisation of the reaction products
In order to compare the reaction products formed under different curing methods, the samples under oven curing at 85°C for 24hrs (OV85) and microwave curing with 30 cycles (MV30) were characterised by XRD, FTIR and SEM together with a comparison of the energy consumption as described below.

3.2.1 X-ray diffraction
From the XRD patterns in Fig. 2, it can be seen that the main crystalline phases of fly ash are quartz, mullite and hematite along with some amorphous phase (halo part). The diffraction pattern changed appreciably after alkali activation. It has to be highlighted that the area of halo (2θ=17-33°) which is attributed to the amorphous phase in the raw fly ash decreased, indicating the amorphous phase
took part in the alkali activation. The crystalline phases in the raw fly ash remained after the alkali activation, which showed good agreement with previous research. Some new crystalline phases also appeared after alkali activation, which varied depending on the curing method. For OV85, the peak presenting hydroxysodalite appeared at 13.8° and 24° 2θ, along with the present of sodium silicate. Compared with the conventional oven curing, a new crystalline phase assigned to chabazite-Na was identified at 18.0° and 34.2° 2θ in MV30.

3.2.2 FTIR spectroscopy
Figure 3 presents the FTIR spectra of the raw fly ash and the AAFAs cured under the two different methods. The FTIR spectra of the AAFA samples showed some differences from that of the raw fly ash. During the reaction, the band at 1087 cm⁻¹ in the raw fly ash which is assigned to the stretching T-O bond shifted (T: Si/Al atom) to lower frequencies: 1010 cm⁻¹ in OV85 and 1007 cm⁻¹ in MV30. This shift, attributed to the T-O bond in AAFA, has been considered as the fingerprint of alkaline aluminosilicate gel formed. In addition, the band at 1007 cm⁻¹ of MV30 was much sharper than the corresponding one in OV85, implying that the sodium aluminosilicate gel underwent better polymerization and its molecular arrangement became more orderly. Furthermore, the spectrum of OV85 showed the appearance of zeolite species with O-Si-O bond at 666 cm⁻¹, whilst another zeolite species appeared at 737 cm⁻¹ in the spectrums of MV30, which are in good agreement with the hydroxysodalite and chabazite-Na identified in the XRD results respectively.

3.3 Scanning electron microscope
Figure 4 presents the SEM images of OV85 and MV30, which feature heterogeneous microstructures. It can be observed that both of these two samples showed compact textures. Most of the fly ash particles under the conventional oven curing and the microwave curing were covered by the reaction gel. The formation of reaction products, sodium aluminosilicate gel, which can be observed on the shell of fly ash particles and the gap between the particles, would result in strength development. Overall no significant difference can be observed on the microstructure of these two samples.

3.4 Energy calculation
In order to compare the energy consumption between the conventional oven curing and the
microwave curing, the energy consumption of AAFA per unit compressive strength is shown in Fig. 5, which clearly demonstrates that the microwave curing can achieve enormously much lower energy consumption compared with the conventional oven curing.

4 Conclusions
From the above results, it can be seen that the AAFA with pulsed microwave curing can obtain significant reduction in energy consumption. The compressive strength of AAFA sample with microwave curing is comparable with that under conventional oven curing. The XRD results demonstrated that different reaction products were generated in OV85 (hydroxyssodalite) and MV 30 (Chabazite-Na). The FTIR test showed that higher extent polymerization of aluminosilicate product was formed in MV30 compared with that in OV85. It is assumed that a different reaction mechanism is involved in the microwave curing process from the conventional oven curing.

References

Fig. 5 Comparison of energy consumption per compressive strength between conventional oven curing and microwave curing.
Effect of different addition methods of lignosulfonate admixture on the adsorption, zeta potential and fluidity of alkali-activated slag binder

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Introduction
Alkali-activated slag binder (AAS), a novel, non Portland cement (PC) hydraulic material which mainly consists of alkaline activators (such as sodium hydroxide and sodium silicate) and ground granulated blast furnace slag (GGBS), has received increased attention worldwide [1]. It has been identified as a novel, alternative cementitious system with lower CO₂ emission and environmentally friendly nature compared to PC [2]. However, one of the main barriers hindered the industrial application of AAS is its high viscosity, which may reduce the workability and, hence, cause the problem in the placement and compaction of concrete. To solve these issues, superplasticiser (SP) has to be employed to improve its fresh properties. [3].

However, it has been found that the current PC-based SPs do not function properly in AAS. Based on the limited research in literature, the reason for this has been presumably attributed to the competitive adsorption between the activator and the SP, the unique electrostatic properties of slag (which differs from PC) and the chemical instability of SP in alkaline solutions [4]. To avoid the competitive adsorption, separate addition of the activator and the SP could be employed. The aim of this study is therefore to obtain a better understanding of the possible interactions between the slag and the lignosulfonate derivation superplasticiser (LS SP) by adding the LS SP and the activator at different time intervals. The adsorption of LS SP by slag, the effect of LS SP on zeta potential and the rheological properties (in terms of mini slump spread) of AAS pastes were investigated and some preliminary results are reported in this paper.

Experimental
Materials
The slag used in this project was a ground granulated blast furnace slag (GGBS) supplied by Civil and Marine Ltd. UK (Table 1). CEM I from Quinn Manufacturing Group was used to formulate the control mixes. The GGBS were activated by a sodium silicate solution, which was modulated to a modulus of 1.5 by adding sodium hydroxide (NaOH). The raw sodium silicate solution and NaOH were obtained from Charles Tennant & Co Ltd and Tennants Distribution, respectively. Lignosulfonate derivation superplasticiser (LS SP) was supplied by Tianjin Jiangong Special Material Co. Ltd.

<table>
<thead>
<tr>
<th>CaO</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>MgO</th>
<th>Sulphide</th>
<th>TiO₂</th>
<th>Mn₃O₄</th>
<th>Na₂O</th>
<th>Fe₂O₃</th>
<th>K₂O</th>
<th>LOI</th>
</tr>
</thead>
<tbody>
<tr>
<td>39.40%</td>
<td>34.30%</td>
<td>15%</td>
<td>8%</td>
<td>0.80%</td>
<td>0.70%</td>
<td>0.50%</td>
<td>0.45%</td>
<td>0.40%</td>
<td>0.38%</td>
<td>0.70%</td>
</tr>
</tbody>
</table>

Mixing procedures
The water to slag ratio for all the mixes was fixed at 0.45. The dosage of LS SP, the concentration of LS SP was controlled as 0, 0.4, 0.8, 1.2, 1.6, and 2.0% (by the mass of GGBS). Waterglass with a modulus of 1.5 was used as an activator and its content was controlled to be added at 4 % (counted as Na₂O equivalent) content by the mass of GGBS. Both the activator and LS SP were dissolved in
water. Three different addition methods were studied, namely: 1) **simultaneous addition (SA):** adding SP and activator together when mixing with GGBS; 2) **prior addition (PA):** adding SP to GGBS first, then activator at 3 min interval; and 3) **delayed addition (DA):** adding activator to GGBS first and then SP at 3 min interval. To compare the effect of delayed addition on PC, CEM I was mixed with water at w: s =0.45 by using both simultaneous addition and delayed addition (delayed by 3 mins).

**Test Procedures**

Adsorption test was conducted to determine the interaction between the GGBS and the LS SP. The amount of LS SP remained in the AAS was determined by a UV-spectrophotometer (Unicam) at a wave length of 286 nm [5]. The amount of LS SP adsorbed by the GGBS was then calculated from the difference in dosage of LS SP before and after it contacted with the GGBS. The zeta potential of the AAS slurry was determined at 20 °C by using a Malvern Nano ZS90 (Malvern Instruments Ltd., UK). The mini-slump test, carried out with a PVC plate and a cone with a lower inner diameter of 38.1 mm, an upper inner diameter of 19 mm, and a height of 52.7 mm, were used to determine the spread of the AAS paste. The diameters at two perpendicular directions were measured and the average diameter was reported. The initial mini-slump measurements were conducted at 7 minutes after mixing (referred to as zero time hereafter).

**Results and discussion**

**Adsorption Isothermal**

The influence of the different addition methods on the adsorption of LS SP are shown in Fig 1. Palacios et al proposed due to the competitive adsorption between superplasticiser and activator in simultaneous addition method, the adsorption of superplasticiser was highly reduced in both waterglass and NaOH activated systems [4]. From Fig 1, it is clear that the addition of waterglass activator has also reduced the adsorption of LS SP on slags. However, the adsorption of LS SP on GGBS in both prior and delayed additions was increased at all dosages, indicating separate additions can somehow reduce the competitive adsorption between the LS SP and the activator. On the other hand, it is obvious from Fig 1 that the adsorbed amount of LS SP increased rapidly with dosages up to a concentration of around 1.2% due to sufficiently active sites on the GGBS particles [6]. At higher dosages (>1.2%), the adsorption increased slowly and gradually reached a plateau, which follows the trend of Langmuir isothermal adsorption. To determine SP characteristic plateau quantitatively, the data was analysed by using Langmuir adsorption Equation [7] –

\[
\frac{A}{C} = \frac{A_s}{K} + \frac{A_s}{C}
\]

where C is the superplasticiser equilibrium concentration, A is the adsorbed amount of superplasticiser by GGBS, A_s is the saturated adsorbed amount of superplasticiser and K is adsorption constant. The constant K was calculated by —— and the A_s obtained by —— of the regressed straight lines. The results from this analysis were summarized in Table 2 and it can be seen that the characteristic plateaus of both PA and DA were increased. As the characteristic plateau A depends on the electrostatic force between SP and GGBS and the higher the As value the higher the electrostatic force between the LS SP and the GGBS [8]. Therefore, the above results would suggest that when the LS SP and the activator was added separately, there is a higher electrostatic force between the LS SP and the GGBS at the equilibrium condition, which would benefit the fluidity of the AAS paste (as discussed below in the mini-slump results). On the other hand, these results also confirmed the hypothesis that adding the activator and the SP in different mixing stages could reduce the competitive adsorption between activator and SP.

**Zeta potential**

The results of zeta potential test are shown in Fig 2. Compared to the simultaneous addition, the zeta potential of the waterglass activated GGBS was decreased in both separated additions at all LS SP dosages. In general, the zeta potential in PA is higher than that in DA except 1.2% LS SP dosage.
However, there is no so obvious trend among those three addition methods. Lignosulfonate superplasticiser is one type of negatively charged surfactant, which is of the same surface charge as GGBS. When dissolved in water, the LS SP would be hydrolysed into macro molecular anion with sulfonic groups and calcium ions, which can increase the zeta potential of suspended particles [9]. Unlike the adsorption of LS SP on PC particles which have a positive or near neutral electrostatic property, the adsorption of LS SP on the negatively charged GGBS is lower than that on PC due to the reduced or even disappeared electrostatic attraction [10]. However, due to the polarity of the sulfonate group, the LS SP could be adsorbed on the surface of GGBS by Van Der Waals attraction force [11]. Nonetheless, based on the adsorption results, higher zeta potential was expected from the separate addition methods than that of simultaneous addition, as the amount of LS SP adsorbed in the latter is lower [6]. It is still not clear what caused this contrary trend in the current zeta potential results. Although it is considered to be due to the interaction between LS SP and the silica gel in waterglass, further study is still needed to verify this assumption.

Fig 1 Adsorption of LS SP with different addition methods at 3 min interval
Fig 2 Zeta potential with different addition methods at 3 min interval
Table 2 Adsorption characteristics of Lignosulphonate superplasticiser in alkali-activated GGBS for prior, simultaneous and delayed addition at 3 minutes interval

<table>
<thead>
<tr>
<th>Activator</th>
<th>Addition method</th>
<th>R²</th>
<th>Slope</th>
<th>Intercept/ g·L⁻¹</th>
<th>K/ L·g⁻¹</th>
<th>As/ mg·g⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>Waterglass</td>
<td>Prior Addition</td>
<td>0.9927</td>
<td>1955.39</td>
<td>1089.06</td>
<td>1.7954</td>
<td>0.5114</td>
</tr>
<tr>
<td></td>
<td>Simultaneous Addition</td>
<td>0.9835</td>
<td>2339.25</td>
<td>1015.03</td>
<td>2.3046</td>
<td>0.4275</td>
</tr>
<tr>
<td></td>
<td>Delayed Addition</td>
<td>0.9899</td>
<td>2178.85</td>
<td>1072.55</td>
<td>2.0315</td>
<td>0.4590</td>
</tr>
</tbody>
</table>

Mini slump

The initial mini slump test results for the AAS and PC systems in the presence of LS SP can be seen in Fig 3 (a) and Fig 3 (b) respectively. It is obvious that the addition of LS SP could increase the fluidity of both PC and AAS pastes, and the fluidity increased with increasing dosage of SP. In waterglass activated GGBS, the improvement of mini slump by the PA and DA methods was obvious. The spread diameter of mini slump obtained from both prior and delayed addition methods was at least 10 mm higher than that of SA method, which correlates well with the adsorption results, although is not corroborated by the zeta potential results. In PC paste, the delayed addition method significantly improved the fluidity compared with simultaneous addition method, which have been well confirmed and demonstrated by other researchers [12].

(a) Initial mini slump results of waterglass activated slag system at 3 min
(b) Initial mini slump test of delayed addition of PC system
Conclusion

(1) Compared to simultaneous addition method, the adsorption of LS SP on AAS was increased in both prior and delayed addition methods.

(2) Compared to the simultaneous addition, the zeta potential of the waterglass activated GGBS system was decreased in both prior and delayed addition methods.

(3) In waterglass activated GGBS paste, the spread diameter of mini slump obtained from prior and delayed addition methods was higher than that of the simultaneous addition method, with the prior addition better than the delayed addition.

(4) Whilst the observed increase in the workability (in terms of mini slump) is well supported by the adsorption data, it is not corroborated by the zeta potential measurement. Further study is still needed to understand the mechanisms involved.

Reference


