Citation for published version:
Dams, B, Hei, Y, Shepherd, P & Ball, R 2021, 'Novel cementitious materials for extrusion-based 3D printing', Paper presented at 2nd International Conference on Construction Materials for Sustainable Future, Bled, Slovenia, 20/04/21 - 21/04/21 pp. 57.

Publication date:
2021

CoMS_
2020/21

Construction Materials for a Sustainable Future

Proceedings of the 2nd International Conference CoMS 2020/21

Volume 1

Slovenian National
Building and Civil
Engineering Institute,
Ljubljana, 2020

Conference CoMS_2020/21, 2nd International Conference on Construction Materials for a Sustainable Future
Editors Aljoša Šajna, Andraž Legat, Sabina Jordan, Petra Horvat, Ema Kemperle, Sabina Dolenec, Metka Ljubešek, Matej Michelizza
Design Eksit ADV, d.o.o.
Published by Slovenian National Building and Civil Engineering Institute (ZAG), Ljubljana, 2020
Price Free copie

First electronic edition

Available at http://www.zag.si/dl/coms2020-21-proceedings.pdf

http://www.zag.si

ISBN 978-961-94071-8-9 (pdf)

CIP - Kataložni zapis o publikaciji pripravili v Narodni in univerzitetni knjižnici v Ljubljani
COBISS.SI-ID=44042499
ISBN 978-961-94071-8-9 (pdf)

NOVEL CEMENTITIOUS MATERIALS FOR EXTRUSION-BASED 3D PRINTING

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SUMMARY: 3D printing (or 'additive manufacturing' (AM)) systems used to manufacture cementitious structures, either in-situ or off site, utilise specialist formulations. This paper describes a new cementitious formulation which can be extruded from a syringe device without the requirement for the addition of an accelerator at the nozzle. This miniature approach brings advantages in that the system required is smaller, lighter, consumes less power and is suitable for mounting on robots which are not reliant on external power or material supplies. Applications of this smaller scale system include concrete crack repair in hard to access areas and printing of specialist conductive formulations which can be used for sensing. Cementitious pastes were successfully printed using a miniature deposition device which could be carried by a small robotic printing agent. Appropriate workability and buildability following deposition was achieved through the use of cellulose gum additions to the mix formulation. Analysis and characterisation tests carried out on fresh mixes enabled comparison of a 1:1 mix of aluminium lactate and diethanolamine with the commercially available accelerator *Master X-Seed*, and mixes with no accelerating admixture added. When compared to results featuring no accelerating agent, tests demonstrated that *Master X-Seed* was the more effective accelerator, promoting early compressive and flexural strength development, but neither accelerator made a constructive contribution to required rheological properties. *Master X-Seed* was the more effective accelerator, but rheology results suggest the difference occurs logistically too soon for a miniaturised deposition system. The retardation effect of cellulose gum and the potential role of in-situ and off-site miniaturised AM methods are evaluated.

KEY WORDS: 3D printing, cementitious material, accelerators, workability, buildability, rheology.

1 INTRODUCTION

Previous academic and industrial research into the use of cementitious-based materials in additive manufacturing (AM, which is often referred to by the term '3D printing') has evolved at a considerable rate over the previous decade. It is now estimated that there are currently thirty projects worldwide investigating 3D printing with cementitious materials for building and civil use in the construction industry [1]. The vast majority of investigations are based upon the AM principal of fused deposition modelling (FDM), which involves the extrusion of a suitably viscous-like material (which could be cementitious or polymeric) through a nozzle to create an object, which may have been designed as a three-dimensional model within software and then 'sliced' into horizontal layers, one discreet horizontal layer at a time [2].

Deposition in the cementitious-based material projects had predominantly taken place without the use or requirement of a permanent or temporary supporting formwork. This additive approach, which only uses material specifically required for the object, starkly contrasts with traditional subtractive methods [3] which have typically been employed historically in the construction industry – a sector which can be viewed as being conservative and risk-averse [4].

Within the construction industry, AM technology is still in a relative state of infancy [5] and the technology has been relatively slow to make a significant breakthrough in terms of practice and profile, especially when compared to manufacturing-based industries such as aerospace and automotive [6].

AM offers the construction industry several advantages over traditional practise. By building layer by layer, only the material specifically required is deposited, thus significantly reducing material wastage on a construction-scale project.

Less labour is required for an automated AM process, reducing costs, delays and the risk of accidents. When an integrated approach involving services is undertaken, there are potential cost benefits to a project in the reduction of timescales, despite potentially high raw material costs [3]. Additionally, AM also offers bespoke design at little extra cost [3], as a complex, architecturally innovative design typically takes no longer to 3D print than a more straightforward rectilinear design.

The reduction of labour-related accidents, injuries and fatalities is the major justification for increased automation in an inherently dangerous industry which regularly involves working in difficult-to-access locations and at height. Between 2012 and 2017, there were 196 fatalities in the construction sector in the UK, of which 96 were due to falling from high places. Meanwhile, 11,520 injuries owing to falls from height are reported each year. The death toll in the construction sector is four times that of all other industries [7]. Automated construction systems possess the potential to substantially address this situation and promote a safer construction environment.

A cementitious-based viscous material suitable for AM construction must possess an appropriate balance between ‘pumpability’ (the ability of a fresh mix to move through a deposition system), ‘printability’ (the level of ease at which material passes through a nozzle) and ‘buildability’ (the ability of freshly deposited material to retain shape following extrusion and when subjected to load from subsequent layers) [8]. Within this conference paper, ‘pumpability’ and ‘printability’ are encompassed by the general term ‘workability’.

A mix possessing good workability requires liquid-like behaviour and low viscosity, whereas a mix containing good buildability exhibits more solid-like behaviour and high viscosity. There is an inherent trade-off involved in the formulation of a suitable cementitious-based material for AM construction, with the aim being the realisation of a material which possesses an appropriate balance between these two crucial parameters. The formulated cementitious mix must possess sufficient buildability to enable passage through the deposition device used whilst also possessing sufficient buildability to allow the extruded material to retain its shape following deposition and resist excessive deformation which may arise from the subsequent deposition of further layers of material.

There are differing approaches employed for deposition of cementitious-based materials using FDM-based AM extrusion methods. Systems often employ a cementitious mixture containing a retarder which is easily pumped from the mixer. Just prior to the nozzle, an accelerator is then added ensuring a suitably rapid set upon printing to mitigate against sagging of the extruded filament and to allow multiple layers to be printed on top of one another within an acceptable time period.

AM extrusion equipment is often on a large, construction scale with the deposition device being mounted on a frame or gantry. Examples of this approach include concrete printing, developed at Loughborough University, UK [9], [10] and Contour Crafting, developed at the University of Southern California, USA [11]. Alternative methods involve using a large compound robotic arm with multiple degrees of freedom which is either stationary or mobile when situated on a movable platform allowing larger prints. An example of this approach is the digital construction platform project developed by the Massachusetts Institute of Technology, USA [12].

A further option involves the use of smaller, coordinated multiple mobile agents. This approach involves extra elements of complexity, as each mobile unit involved must be aware of the location and activity of the other printing agents in order to avoid collisions in movement and duplication of deposition in identical or similar locations [13]. The approach of using smaller, multiple agents inherently requires the miniaturisation of the deposition equipment involved in relation to larger, construction scale methods. The advantages of using a multiple smaller agent approach is the potential for reducing timescales due to the successful implementation of agents simultaneously printing, the possibility of greater precision due to enhanced mobility for detailed repair work of structures and the printing of conductive specimens for experimental work involving monitoring sensors.

A crucial decision in the formulation of AM cementitious-base mixes is deciding on which approach is used to control the rheology of the fresh mixes and achieve the desired balance between workability and buildability. Two main contrasting options are available. Firstly, the use of accelerating admixtures, the effects of which do not begin immediately while the material is passing through the deposition equipment, but instead take effect once the material has been deposited in order to stiffen the mix to avoid deformation following deposition of subsequent layers. This approach involves high precision with regards to timing; if the accelerator takes effect too soon, the material can stiffen within the deposition device and will not be viable to print. The second approach, as an alternative to accelerating admixtures, is to formulate the mixes so that the fresh rheological properties do not require further acceleration. This can be achieved by adding an agent which possesses shear-thinning properties to the mix. Such materials promote a reduction in viscosity of a fresh cementitious-based material while it is under stress, for example moving through a deposition device, while also promoting an increase of viscosity once the material is deposited and retaining shape while at rest. However, extruded filaments are subsequently stressed by successive layers and need to continually resist deformation.

Here we report both material approaches in the context of a multi-agent deposition system with miniaturised deposition devices. Mixes containing accelerating admixtures and shear-thinning visco-elastic mixes, without accelerating admixtures, were investigated and the results evaluated to determine a suitable approach. A key consideration is timing – if the accelerator is to be effective for a miniaturised AM system, it must not take effect too soon as enough time must be allowed for the deposition of a complete cartridge. The miniaturised deposition device system used by the authors requires approximately 25 minutes to make the mix, load the cartridge, fully extrude the material and withdraw the deposition device. Therefore, the effectiveness of the accelerators will be judged accordingly by performance relative to this timescale.

2 METHODOLOGY

This section outlines the methodology involved in the manufacture of the mixes and the various tests carried out.

2.1 Mix specification and manufacture

Three mixes were manufactured: Mix 1 which featured no accelerating agent, Mix 2 which featured the commercially available product *Master X-Seed* 100 (supplied by BASF, UK) and Mix 3 which employed a laboratory formulated accelerating admixture of 1:1 Aluminium Lactate to Diethanolamine (individual chemicals supplied by Sigma-Aldrich, UK). Previous research demonstrated the effectiveness of aluminium lactate – diethanolamine combinations as an accelerating agent for a simple cement paste featuring CEM I [14], along with just water and plasticiser [15]. Mix proportions were determined by weight and mix constituents by percentage of weight are shown in Table 1.

Dragon Alpha CEM I 42.5R CEM I was the basis of the mixes for this study. The binder was augmented by the addition of type – ‘F’ pulverised fuel ash (PFA) (EN-450, supplied by Cemex). The ratio of CEM I:PFA in all three mixes was 65:35. The polycarboxylate-based superplasticiser Master Glenium ace 499 was added to provide extra workability to the fresh mixes whilst maintaining a water/binder ratio below 0.5 in order to promote strength in the cured material.

Hydroxyethyl methyl cellulose (HEMC) gum (Walocel VP-M-7701, supplied by Dow Chemicals) fulfilled two important functions as an admixture in the mix. Firstly, in preliminary tests it provided a high degree of water retention and ‘thickening’ of the mix which reduced constituent segregation and the potential for dead zones within material cartridges primarily via the mechanism of water sorption [16]. Secondly, it possessed shear-thinning properties, decreasing viscosity when mixes were subjected to shear stresses and increasing viscosity when the mix was at rest. It was utilised in other cementitious-based material investigations for these properties [17].

Mixes were manufactured by firstly hand-mixing the dry constituents, followed by the addition of water, plasticiser and accelerating admixture (where specified). Mechanical mixing was applied using a beater moving in planetary motion for a period of 60 seconds at 60 revolutions per minute (rpm) followed by gathering the mixture together by hand and a further period of mixing at 120 rpm for 60 seconds.

Table 1 - Mix formulation showing constituent quantity as a percentage of weight

Mix	Mix 1	Mix 2	Mix 3
CEM I % Wt.	45.6	46.2	46.2
PFA % Wt.	24.6	24.9	24.9
Hydroxyethyl methyl cellulose, HEMC % Wt.	0.61	0.62	0.62
Plasticiser % Wt.	0.74	0.72	0.72
Water % Wt.	28.5	24.4	24.4
1:1 Aluminium lactate: Diethanolamine % Wt.	-	-	3.25
Master X-Seed % Wt.	-	3.25	-

Total %	100	100	100

Extrusion tests were carried out utilising a miniature deposition device featuring a 60ml capacity syringe as the material cartridge suitable for a small multiple agent printing robot to carry. For a full specification and images of the device, the reader is referred to [15]. The ability to extrude circular beads of material through an 8 mm diameter nozzle was attempted at 25 minutes, 50 minutes and 75 minutes after initial mixing of the constituents.

2.2 Analysis and Characterisation

Rheology of the test mixes was assessed to quantify the effects of the accelerating solutions added. Oscillation tests were carried out on a TA Instruments DHR-2 rheometer to determine the complex modulus *G**, of the fresh mixes. This served to quantify the rigidity of the fresh mixes determined by the extent to which deformation is recoverable (elastic modulus, solid-like behaviour) or non-recoverable (viscous modulus, liquid-like flow) and summing the two components. Disposable aluminium flat plates with a 25 mm diameter upper geometry and 40 mm lower plate were used with a geometry gap of 1 mm, into which material was inserted immediately following the completion of mixing. The displacement-controlled oscillatory tests used a small angular velocity of 5.0×10^{-5} radians per second, ensuring that the material stayed within the linear viscoelastic region. A constant frequency of 1 Hz equating to an angular frequency of 6.28 radians per second and a plate temperature of 25°C was maintained.

A cone penetrometer test was used to evaluate the difference between mixes 1 and 2 while a body of freshly mixed material was at rest and exposed to the environmental temperature (the laboratory temperature was 21.5° ±1° for mix 1 and 17.5° ±1° for mix 2, with relative humidity for both tests at 72% ±3%). 150 g batches of freshly mixed material were transferred to a circular container (as shown in Figure *1*). The cone of the penetrometer device was adjusted to touch the surface of the mortar, with this initial distance reading recorded. At five-minute intervals over a period of 90 minutes, the cone was released and the depth to which the tip penetrated the material was recorded.

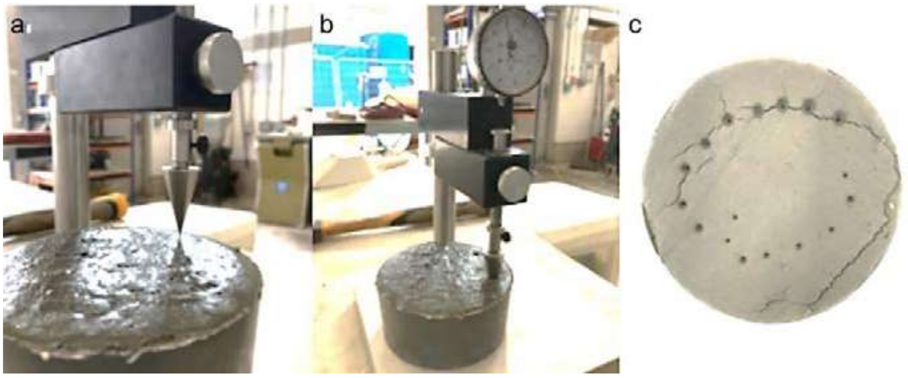


Figure 1 - Cone penetrometer tests showing (a) the lowering of the tip, (b) penetration of the fresh material and (c) the stiffening material at the conclusion of a series of tests conducted at five minute increments.

Compressive and flexural strength tests were carried out 24 hours following the formulation of mixes to ascertain the extent to which the accelerating admixtures impacted upon early strength. A 50 kN Instron Universal 2630-120/305632 testing machine was used for mechanical tests. Prismatic test specimens of the test mixes were manufactured in accordance with British Standards BS EN 12390-5:2009 (BSI, 2009) and tested in four-point bending to determine flexural strength and on 40 mm x 40 mm sections of the prisms to determine compressive strength.

Dry samples of the HEMC powder were coated in a 10 nm layer of gold to prevent charging and increase the signal-to-noise ratio prior to imaging at magnifications of x43 and x500 using a JEOL SEM6480LV Scanning Electron Microscopy (SEM).

3 RESULTS

Mixes can be considered to have an open time in excess of 75 minutes as the deposition device was still able to extrude the material in 8 mm diameter beads at this point (but could not be extruded at 120 minutes). It can be observed in Figure 2, which shows mix 2, that material experienced stiffening, with the 75-minute extrusions showing greater precision and definition in the vertical line printed. This was similarly evident in mix 1, which was also easier to extrude than mix 2. Mix 3, which although extrudable, did not produce defined layers at the 75 minute stage.

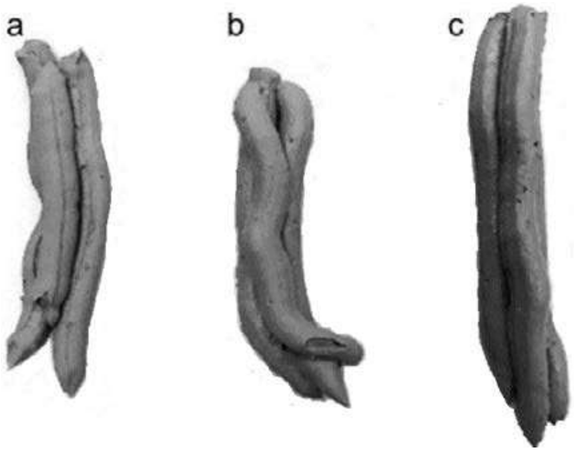


Figure 2 - Beads of mix 2 extruded by the deposition device at (a) 25 minutes, (b) 50 minutes and (c) 75 minutes after mixing.

Figure 3 shows the complex modulus *G** of mixes 1 to 3. It is important for a miniature deposition device that *G** values should be low enough to allow extrusion, but high enough for extruded material to retain shape and resist deformation. A miniature deposition device inherently requires lower *G** value material than large, powerful construction scale deposition equipment (capable of extruding stiffer material, albeit with greater energy requirements). The material of all three mixes exhibited visco-elastic behaviour, with a tendency towards elastic, solid-like properties in fresh material flow; in all mixes *G** component Elastic modulus dominated over the viscous modulus component. It can be seen that *Master X-Seed* exerts the greater influence over *G** (increasing from 3 MPa to 5 MPa at 25 minutes) in the early stages of the test. It can be surmised that given the deposition difficulties at two hours, a *G** value of between 3 – 6 MPa is suitable for a miniature deposition device; less than this value would signify a mix which was too workable for AM.

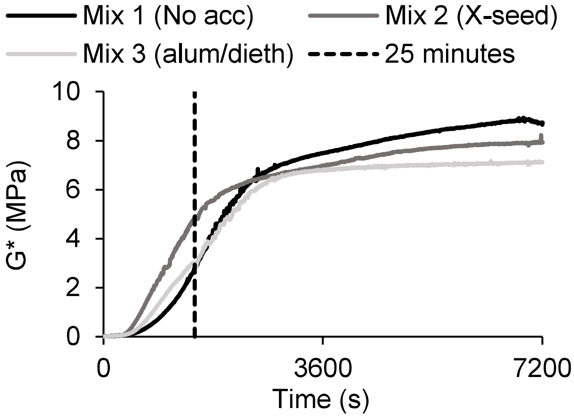
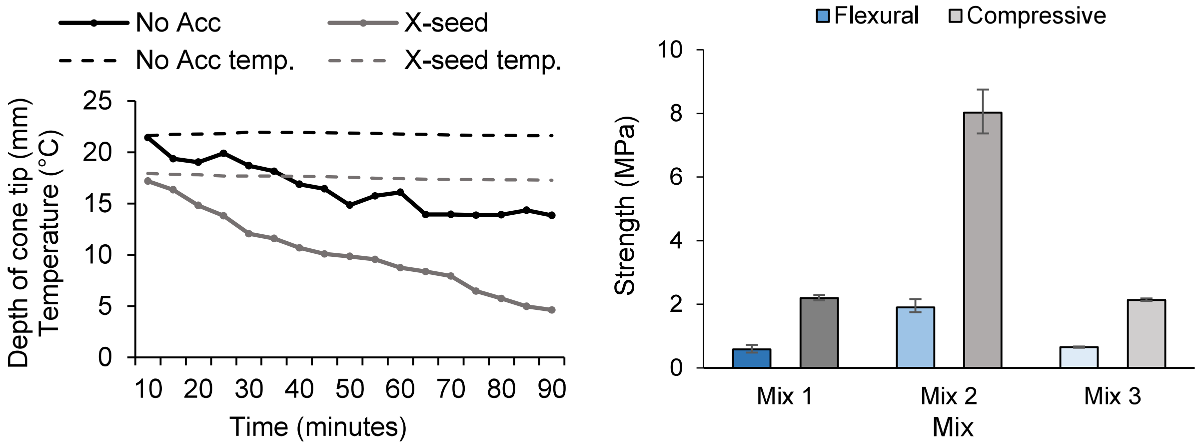


Figure 3 - Complex modulus G* for Mixes 1-3, with the 25-minute mark indicated.

Figure 4 (left) shows the cone penetration results for mixes 1 (no accelerator) and 2 (*Master X-Seed*) with the laboratory temperature indicated. *Master X-Seed* can be observed to have had an impact on material stiffening at 90 minutes, although the cone was still able to penetrate the mix 2 material by 5 mm.

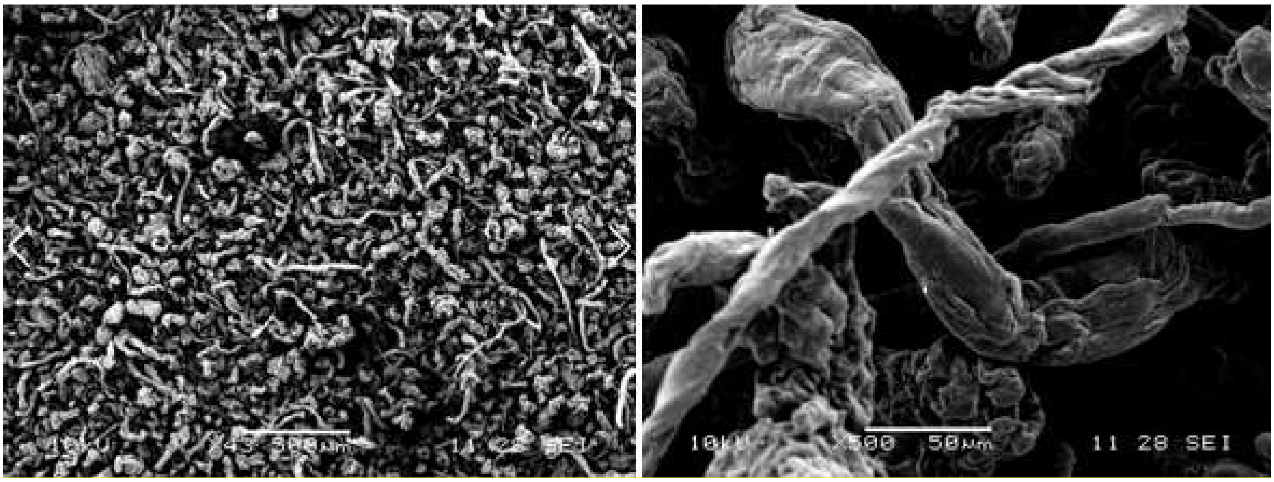
1 day compressive and flexural strength tests are shown in Figure *4* (right). *Master X-Seed* contained in Mix 2 made a significant difference to both flexural and compressive strength, with the aluminium lactate-diethanolamine combination contained in mix 3 having no discernible effect.

SEM results are shown in *Figure 5*. The HEMC microstructural image shows water-absorbing fibres between 10 and 50 μm in diameter. These micro-fibres wrap around water molecules, adsorbing water and expanding, reducing segregation and bleeding in the fresh mix.



(a) (b)

Figure 4 – (a) Cone penetrometer tests showing mix 1 (no accelerator) and mix 2 (*Master X-Seed*). (b) Flexural and compressive strength tested 1 day after mixing for mix 1 (no accelerator), mix 2 (*Master X-Seed*) and mix 3 (aluminium lactate-diethanolamine).



(a) (b)

Figure 5 – Scanning Electron Microscope (SEM) images of Hydroxyethyl methyl cellulose (HEMC) particles at (a) x43 and (b) x500 magnification.

4 DISCUSSION

Master X-Seed was the more effective of the two accelerators, promoting the development of strength and stiffness and the extrusion of defined, straight layers. Aluminium lactate and diethanolamine did not prove to be an appropriate accelerating admixture for a cementitious mix containing cellulose gum.

Water-retaining cellulose ether particles adsorb on to the surface of calcium hydro-aluminates resulting from initial C_3A-water reactivity, delaying the dissolution of C_3A and effectively retarding the fresh mix. With gypsum both present and absent, cellulose ethers have been shown to retard the crystallisation of calcium hydro-aluminates [18]. Through adsorption, cellulose ethers can also reduce the nucleation and growth rate of calcium silicate hydrate (C-S-H) on the surface of C_3S particles [19].

Master X-Seed consists of a suspension of nanosized crystalline C-S-H seeds and is designed to promote the rapid nucleation and growth of C-S-H crystals, primarily targeting the reduction of the dormant period following initial C_3A reactions [20]. When using *Master X-Seed* and considering the demonstration of cellulose ethers adsorbing on to C-S-H particles, it is suggested that mixes developed in this study may have experienced retardation at this later stage, with the effectiveness of *Master X-Seed* diminished by the presence of cellulose ethers. However, in this study it is the early stages of reactivity following mixing which are of prime interest. Although accelerators designed to target silicate hydration and benefit the development of long-term strength are not designed to have radical early effects [21], *Master X-Seed* did demonstrate an early accelerating influence with mixes showing increased G^* and strength.

A solution comprising aluminium salt and diethanolamine is an alkali-free accelerator, which are designed to act upon aluminates, introducing a larger quantity of aluminium ions into the fresh mix to achieve acceleration [22] and promoting the quick formation of needle-like ettringite particles with the intention of stiffening the mix rapidly [21]. The presence of lactic acid in cement has been shown to accelerate aluminate phases rather than silicate phases [23]. Therefore, if cellulose ether inhibits the formation of hydration products arising from initial C_3A reactivity, aluminium lactate – diethanolamine ceases to be an effective accelerating solution and is not appropriate for AM extrusion processes.

HEMC molecules increase the viscosity of the water in the mix by adsorbing on to water molecules, expanding and attracting molecules in adjacent chains. Cellulose ether molecules entangle and intertwine amongst themselves at low shear rates, but at high shear rates disentanglement and subsequent alignment parallel to flow direction occurs [24] - this pseudoplastic behaviour is desirable for a miniaturised AM deposition device suitable for a multiple agent approach. Cellulose ether molecules additionally readily absorb moisture from the air [24].

However, considering the performance of *Master X-Seed* in tests, in practical use, a period of time following the completion of mixing has to be allowed for loading the material into a cartridge, placement of the cartridge into a deposition device, attachment of the deposition device to a printing agent and allowing the printing agent to manoeuvre into position before material can start flowing through the system and the full cartridge extruded (taken as 25 minutes in this study). It can be seen in the G^* tests (Figure 3) that *Master X-Seed* affects the mix prior to this time and suggests that it may be inappropriate for AM due to the risk of excessive stiffening occurring in the material while still in the deposition device prior to extrusion. After this timescale, it is suggested by rheology results that *Master X-Seed* becomes less effective, with the mix with no accelerating agent achieving the highest complex modulus G^* value post-extrusion.

Two possible alternative options are available for miniaturised AM. Firstly, the shear-thinning properties of the fresh mix could be used to provide sufficient buildability following deposition without the need for an explicit acceleration admixture to be applied. Further admixtures known to possess shear thinning properties as an alternative to HEMC, such as Diutan gum or Welan gum [25] could be trialled in miniaturised AM mixes.

A second option would be to investigate the use of calcium aluminate cements (CAC). The addition of CAC to a cementitious binder promotes the formation of C_3A and the rapid hardening or ‘flash setting’ of material [26], [27]. CAC may be added into a Portland cement-rich system, in which CAC would supplement the main binding materials of CEM I and PFA. CAC may additionally be the primary binding constituent in a mix; however, it is suggested that this approach may be challenging for AM, with the risk of a flash-setting occurring while material was still within the deposition system. The aim of this approach would be to control the quantities of CAC added as a minority constituent to a CEM I-rich system to avoid a short-timescale flash-set. Furthermore, adding calcium sulphate (CS) and retarding or accelerating constituents compatible with CAC may also need to be investigated and added as appropriate to achieve this aim and avoid C_3A flash-setting.

Considering the role that multi-agent AM extrusion may take in the construction industry, there are broadly two possible contrasting applications – on-site, in-situ construction or repair of monolithic structures or off-site pre-fabrication of elements and components, which would be transported to site for assembly. Transporting prefabricated materials to site can be costly and challenging and autonomous in-situ construction can improve process control and adaptability to site conditions [12]. However, materials used in monolithic buildings created in-situ can be highly difficult to recycle or reuse in further construction following the initial design life of a building. Typically, for in-situ concrete buildings demolition is the economic and only viable process [28].

In contrast, the manufacture of prefabricated components using indirect mechanical connections to assemble promotes design for disassembly and the potential to reuse or recycle building elements following the completion of first use [29]. In-situ construction typically adheres to linear economy principals of make – use – disposal whereas off-site pre-fabrication is suitable for sustainable construction practices which adhere to circular economy principals, where at the end of design life buildings are disassembled and the elements are either rearranged into a different configuration reflecting a change of use, or recycled for further use in construction without being downgraded (such as crushed aggregate for roads in the case of concrete [28]). Research into reversible design and off-site prefabricated designs appropriate for disassembly is taking place in Europe in research investigations such as the Circular bio-based construction industry (CBCI) [30] and Buildings as material banks (BAMB) projects [31].

5 CONCLUSIONS

These experiments have shown that in a cementitious mix with a viscosity suitable for AM with a miniaturised deposition device, *Master X-Seed* is the more potent accelerating agent. The retardation effects of HEMC within a cementitious paste reduce the effectiveness of aluminium lactate - diethanolamine. However, considering the requirement of fresh material to first pass through a deposition system in the AM process, it can be concluded that *Master X-Seed* may also not be entirely suitable. This is because the impact of the accelerator may occur while fresh material is still passing through a deposition system and high workability and low viscosity are desired at this stage. To allow for variation in the timing of deposition following the mixing of fresh material, this study suggests that using a shear-thinning rheology-modifying agent is a suitable, and time-flexible, approach to provide sufficient buildability with miniaturised AM. Alternatively, the use of CAC as a minority constituent to control the stiffening process may be investigated with a deposition system of known deposition timescales so that workability remains high pre-extrusion with the onset of rapid stiffening of the paste as soon as possible post-extrusion.

ACKNOWLEDGMENTS

The Aerial AM project is funded by the Engineering and Physical Sciences Research Council (EPSRC) [grant number EP/N018494/1]. This study was supported by the EPSRC Centre for Decarbonisation of the Built Environment (dCarb) [grant number EP/L016869/1] and a University of Bath Research Scholarship. The Circular Bio-based Construction Industry (CBCI) project is funded by the European Union Regional Development Fund Interreg 2 Seas Mers Zeeen (2S05-036).The authors express thanks to the following: William Bazeley, Neil Price, David Surgenor, Martin Naidu, (Laboratory Technical personnel, University of Bath, UK), Oscar Cano Iranzo, Robert Baumann and Igor Toutkewicz (The Dow Chemical Company - HEMC supplies).

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