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ABSTRACT

Autonomic self-healing cementitious materials generally rely upon the transport of adhesives via capillary flow in discrete cracks to heal macro-cracks. A series of experimental and numerical studies are presented that simulate the capillary flow of cyanoacrylate in a range of discrete cracks in prismatic cementitious specimens. The numerical procedure developed incorporates corrections to established capillary flow theory to consider stick-slip behaviour of the meniscus and frictional dissipation at the meniscus wall boundary. In addition, two short benchmark studies are reported in order to firstly verify the time–viscosity relationship of the cyanoacrylate in a mortar capillary channel and secondly to examine the capillary flow of the healing agent in small diameter glass capillaries. These studies also provide data to validate the numerical model. The capillary rise response of a healing agent in a self-healing system is predicted using the calibrated model and verified with published experimental data.

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1. Introduction and literature review

In the last decade considerable research effort has been directed towards the development of self-healing materials in order to overcome the construction industry wide problem of civil infrastructure deterioration. These self-healing materials have the ability to adapt and respond to environmental and operational changes to which they may be subjected [1,2]. Such materials are generally classified as 'smart' materials and include self-healing materials as a particular sub-set [3]. In the case of concrete, the presence of pores and cracks in the matrix, the latter caused as a result of plastic shrinkage, mechanical loading and thermal effects amongst other actions, allows the movement of water and gases that contribute to the deterioration of a structure. However, networks of connected pores and cracks may also provide pathways for the flow of healing agents.

Two types of self-healing in cementitious materials are referred to in the literature. The first is autogenic healing, a natural phenomenon which occurs mainly due to the pozzolanic properties of cementitious materials [4–7]. The second type of healing is termed autonomic healing and is a concept originally proposed in 1994 for cementitious materials by Dry [8]. These self-healing cementitious materials normally have tubes or capsules of adhesive agents embedded within the cementitious matrix that, upon damage, break and release the agent into the matrix. The adhesive then flows under capillary action to macro-cracked and micro-cracked regions. The methods of encapsulation have included discrete microcapsules [9], continuous hollow lengths of glass [10,11] and ceramic capillary tubes [12]. Although the concept of autonomic healing has been proven using these techniques at a small scale, there is still significant research required to select an appropriate encapsulation system which can withstand the rigours of reinforcement placement and concrete casting [3].

Alkali–silica and sodium silicate solutions [9,13], one- and twocomponent epoxy resins [9,14], cyanoacrylates [10,11] and most recently a two-component expanding polyurethane foam [12] have been used as healing agents with varied success. The selection of healing agents is usually based on a number of requirements; the agent must have a sufficiently low viscosity to flow into and around the damage region; it must have the ability to bond together two faces and it should have a sufficient shelf life to allow encapsulation for long periods of time without deterioration of the bonding properties [11]. The ability to guarantee the availability and subsequent adequate mixing of both parts of a two-component healing agent at a crack location may be limited [12] and therefore one-component systems may offer significant advantages in this respect.

Cyanoacrylates, more commonly referred to as superglues, typically have a shelf life of one year. When encapsulated within concrete they would be expected to heal damage that occurs within the early life of a structure, due to early thermal effects, shrinkage and construction loading. However, the future development of flow networks may offer the potential to continually deliver superglues and resins to zones of damage throughout the working life of a structure thereby removing the constraint of the healing agent's shelf life.

Previous experiments undertaken on self-healing cementitious materials have established that agents will flow under capillary action

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Table 1

Capillary rise theory.

$2\pi r\gamma \cos(\theta) - \pi r^2 \rho gz \sin(\varphi) - 8\pi \mu z\dot{z} - \rho \pi r^2 \frac{\partial}{\partial t} (z\dot{z}) = 0 (1)$	Full dynamic flow equation for of liquid in a capillary tube (Lucas-Washburn (L-W) equation)
$p_c - \rho gz \sin(\varphi) - \frac{\mu}{k} \dot{z} = 0 $ (2)	L-W equation without inertial and gravitational effects.
$p_c = \frac{2\gamma\cos(\theta)}{r}(3)$	Young-Laplace equation for capillary pressure.
$h_{eq} = \frac{p_c}{\rho g \sin(\varphi)} (4)$	Equilibrium capillary liquid rise height from balance of hydrostatic and capillary pressure.
$\beta_s = 1 - \frac{h_s}{h_{eq}} $ (5)	Correction factor for stick-slip behaviour of the meniscus (unitless) [18].
$\gamma \cos(\theta(t)) = \gamma \cos(\theta_0) - \beta_m \dot{z}(6)$	Correction factor for frictional dissipation at the moving front (units Ns/m ²) after [19,20].
$\dot{z} = \left(\frac{r\beta_{\rm w}}{2} + \frac{r^2}{8\mu}\right) \left(\frac{p_c}{z}\right) (7)$	Velocity of meniscus including the correction factor for wall slip (β_w units m ³ /Ns) [21].
$p_{c0}(1-\beta_{s}) - \frac{2\beta_{n}\dot{z}}{r} - \rho gz \sin(\varphi) - \left(\frac{z}{\frac{r\beta_{w}}{2} + \frac{r^{2}}{8\mu}}\right)\dot{z} = 0 $ (8)	Amended L-W equation proposed by Gardner et al. [16].

([11,12,15]), but to the authors' knowledge, no detailed investigation has been undertaken on the capillary flow of healing agents in cracks in cementitious materials. It is expected that the quantity and rate of flow will be governed by the profile and nature of the crack, the curing rate of the agent as well as by chemical interactions between the host matrix and the healing agent, which may affect this curing rate. The quantification and simulation of this flow is the subject of the present publication. The ability to measure and predict the capillary flow of healing agents through cracks in cementitious materials will allow more efficient self-healing systems to be designed in the future.

This paper presents experimental data on the capillary flow of cyanoacrylate, an all purpose superglue, in discrete cracks in cementitious materials obtained via high speed video measurement. The paper also considers the capillary flow of a cyanoacrylate in a self-healing system and introduces a numerical model to simulate experimentally observed results. The structure of the paper is as follows:

- Section 2 provides an overview of standard Lucas–Washburn capillary flow theory as well as the amendments explored by Gardner et al. [16] and develops this theory further to consider flow in a discrete crack of varying aperture.
- Section 3 presents details of a study performed to provide an independent verification of the viscosity of the cyanoacrylate adhesive and an indication of its time-viscosity behaviour over a 15 minute timescale.
- Section 4 employs the amended theory to simulate the experimentally observed flow of cyanoacrylate in a glass capillary tube.
- Section 5 presents the experimental data on the flow of cyanoacrylate in openings in cementitious materials; including data from experiments with planar, inclined and tapered apertures or 'cracks'. It also provides a comparison between the amended flow theory and the experimental data presented.
- Finally, in Section 6, a study is described which ascertains whether the flow of cyanoacrylate in a natural discrete crack in the self-healing system proposed by Joseph et al. [11] can be predicted using the amended theory.

The present investigation concentrates on the flow of cyanoacrylate in a discrete crack with no detailed consideration being given to flow into or out of the porous media adjacent to the discrete opening. The importance of including both aspects of flow behaviour when simulating these problems with, for example, a finite element model, is however fully acknowledged [17] and this issue is the subject of on-going work.

2. Capillary rise theory

The capillary flow theory previously presented by Gardner et al. [16] is summarised in Table 1. The omission of the inertial term in the consideration of water flow in a capillary tube has been justified

previously by Gardner et al. [16] and it is considered that the relative importance of inertia term will be even less for cyanoacrylate, which has a higher viscosity than water.

Where:

- z = capillary rise height (m); γ = surface tension (N/m); θ = liquid/ solid contact angle (°); φ = capillary inclination angle (°); ρ is the density of liquid (kg/m³); g = gravitational acceleration (m/s²); r = radius of the capillary (m); μ = dynamic viscosity (Ns/m²); t = capillary rise time (s); effective permeability term k = r²/8 [22];
- h_s = the rise height allowing for pinning (modified capillary rise height);
- $p_{c0} = \frac{2\gamma \cos(\theta_0 \alpha)}{r}$ where θ_0 is the static contact angle (°) and α is the inclination of the capillary wall (°).

In an earlier study, Gardner et al. [16] reported no significant influence of wall slip (β_w term) on the capillary rise response of water in glass capillary tubes or discrete cracks in mortar, however, this term is shown here for completeness.

Young [22] accounted for a change in fluid velocity as a result of nonuniform capillaries (Fig. 1) by applying the mass balance equation (9) to the L–W Eq. (1):

$$\dot{z}A(z) = v(x)A(x) \tag{9}$$

Applying the same principles to Eq. (8) leads to the following equation for flow in non-uniform capillaries which allows for stick slip and frictional dissipation.



Fig. 1. Illustration of the parameters used to model water flow in a non-uniform capillary.

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