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Empirical models to predict rheological properties of fiber reinforced cementitious composites for 3D printing

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HIGHLIGHTS

- Factorial design was adopted to evaluate influence of five variables on material rheological properties.
- Empirical models were established to predict rheological properties and were verified by experiment.
- Increased fiber dosage boosts all rheological parameters, while water-to-binder ratio shows opposite trend.
- Torque viscosity increases while flow resistance and thixotropy decrease with fly ash-to-cement ratio.
- Silica fume-to-cement ratio shows an opposite trend as compared to that of fly ash-to-cement ratio.

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ABSTRACT

3D printable construction materials need to be conveyed through a delivery system whilst possess certain flow resistance to ensure materials can sustain the weight of subsequent layers. To meet these requirements, material rheological properties should be optimized. In this study, factorial design was adopted to evaluate the influences of five variables (water-to-binder ratio, sand-to-binder ratio, fly ash-to-cement ratio, silica fume-to-cement ratio, and dosage of fiber) on material rheological properties (flow resistance, torque viscosity and thixotropy). Empirical models were established to predict rheological properties and were verified by experiment. Results imply that the increment of the dosage of fiber boosts all the rheological parameters, which are declined with the increment of water-to-binder ratio. Torque viscosity raises while flow resistance and thixotropy are decreased with the rise of fly ash-to-cement ratio. Conversely, the influence of silica fume-to-cement ratio shows an opposite trend on rheological properties as compared to that of fly ash-to-cement ratio. Flow resistance and torque viscosity are improved whilst thixotropy is declined if sand-to-binder ratio increases. Different formulations were adopted in printing test for verification and demonstration purpose via a robotic arm printing system in the end.

1. Introduction

3D printing technology for cementitious materials builds up objects from successive cross section layers [1]. In a 3D printing process, materials are firstly premixed and conveyed to a nozzle head via a pumping system. A printing system then controls nozzle movement to deposit materials [1–4] based on a CAD model. To ensure a successful printing process, both robust printing system and consistent printable cementitious materials are essential.

Several types of printing systems have been developed in recent years, such as the Contour Crafting system [5], the mortar printer [6] and the robotic concrete printer [7,8]. Besides printing systems, the cementitious materials also plays a crucial role in a successful 3D printing process [9–15], which needs the printable materials to meet pumpability and buildability requirements [16–21]. Material pumpability is directly associated with pumping pressure in the delivering phase of the 3D printing process, and buildability defines the height of the printed structure. In recent studies, researchers have revealed that rheological properties, especially static/dynamic yield stress and plastic viscosity, were critical material characteristics for printable materials to meet such standards. Material constituents serve as vital factors that influence material rheological properties.

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Intensive research has been conducted to investigate the influence of material constituents on rheological properties [22–26]. However, limitations still remain. Firstly, previous works mainly reached qualitative conclusions. Few quantitative result was established to quantify the impact of material constituents on rheological properties. Besides, research needs to be carried out to explore the influence of material constituents on thixotropy, which is an essential time-dependent parameter for the printing process. To overcome these limitations, an efficient approach should be adopted for the experimental design to reduce experimental runs. Meanwhile, quantitative models should be built to provide guidance for the predictions of rheological properties [27]. The influence of material constituents on thixotropy also needs further exploration.

Design of Experiment (DoE) is a scientific experimental methodology for experimental design and data analysis based on fundamental mathematical statistics [28]. As a powerful approach to exploring the correlation between factors and responses, DoE has been successfully used in various research fields [27,29–31]. One of useful methods that adopt DoE is called factorial design, which quantifies the influences of variables on responses through constructing statistical models.

The factorial design is more efficient than traditional one-factor at a time experiment. The method can simplify the experimental process and reduce experimental runs, but enough information still can be extracted for data analysis. Meanwhile, the method can allow the influence of a factor to be estimated at several levels of other factors.

The focus of this study is to construct statistical models to predict how different factors influence rheological properties by adopting the factorial design. The pre-determined factors in this work are sand-to-binder ratio, water-to-binder ratio, fly ash-to-cement ratio, silica fume-to-cement and the dosage of fiber. The responses are flow resistance, torque viscosity and thixotropy of materials. The analysis of variance (ANOVA) was adopted to conduct statistical analysis and create statistical models for predictions. Experimental results indicate that the statistical models can be used to predict material rheological properties with appropriate degree of accuracy. Although the constructed statistical models may not be universally applicable due to change in material chemical composition, different particle size distribution, etc, the validation test indicates that factorial design is efficient to find the desirable formulation within a given boundary.

2. Background and experimental design

2.1. Bingham model

Rheology describes the deformation and flow behavior of materials [32,33]. Bingham model is commonly used for cement pastes due to its simplicity and wide acceptance [34,35]. The correlation between shear stress τ (Pa) and shear rate $\dot{\gamma}$ (1/s) in the Bingham model is described as follows:

$$\tau = \tau_0 + k\dot{\gamma} \tag{1}$$

where τ_0(Pa) and k (Pa·s) are yield stress and plastic viscosity, respectively. Yield stress τ_0 represents minimum shear stress required to initiate or maintain the flow of a material, while plastic viscosity k describes the change of shear stress with altering shear rate. These two rheological parameters are measured by a rotational rheometer test, in which Bingham model can also be expressed as the following formula for convenience of experiment design and data analysis:

$$T = G + hN \tag{2}$$

Eq. (2) describes the correlation between the measured torque T (N·m) and rotational speed N (rpm). The parameter G (N·m) is flow resistance, representing the minimum torque required to initiate or maintain the flow of a material. The parameter h (N·m·min) is torque viscosity. Similar to the k in Eq. (1), the parameter h describes the change of applied torque with altering rotational speed.

Bingham model indicates that if both parameters (yield stress τ_0 and plastic viscosity$k$; or equivalently, flow resistance G and torque viscosity h) are kept at a higher level, the material is difficult to flow at any given speed. As indicated in the study of pumpable concrete, the rise of τ_0 and k yields the increment of pumping pressure [36], which is undesirable to the pumping process and delivering process. In the printing phase, the restraining deformation due to subsequently printed layers requires a high yield stress τ_0[16,37]. Considering the delivering phase and the printing phase as a whole process, yield stress should be enhanced whilst plastic viscosity should be kept at a low level to achieve appropriate rheological control of 3D printable fiber reinforced cementitious composites for a successful printing process.

2.2. Time-dependent effect of rheology

The correlations between buildability, pumpability and rheological properties are expressed as follows [37]:

$$H = \frac{\alpha}{\rho g}\tau(t) \tag{3}$$

$$P = \left[\frac{8\tau(t)}{3R} + \frac{8k(t)}{\pi R^4}Q\right]L \tag{4}$$

where H (mm) and P (Pa) are printed height and pumping pressure, respectively; R (mm) and L (m) are the radius and length of a hose, respectively; Q (m^3/s) is an average flow rate. ρ (g/cm^3) and g (m/s^2) are the density of materials and gravitational constant, respectively.

The rheological properties evolve with time due to the hydration process. Roussel [38] proposed a model to quantify time-dependent effect of yield stress that is shown as follows:

$$\tau_0(t) = \tau_0(0) + A_{thix}t \tag{5}$$

where t is resting time; A_{thix} is thixotropy parameter, which is assumed to be a constant for a given material; *is* is yield stress at resting time zero. Generally a high $A_{\text{thix}}$is required to drive up yield stress to make certain that materials possess enough buildability for a 3D printing process [37].

The time effect on dynamic viscosity is expressed as follows [36]:

$$\mu_0(t) = \mu_0(0) + (1000 - \mu_0(0))(t/t_v)^n \tag{6}$$

where t_v is the moment at which the slurry reaches 1000 Pa•s, *is* is viscosity at resting time zero. However, yield stress is greatly higher than plastic viscosity in general. Compared with the influence of plastic viscosity on the pumping pressure, the influence of yield stress on pumping pressure is the dominant factor.

2.3. 2^k design and 2^{k-p} fractional design

2^k design (factorial design) is a special design method in DoE [39]. In 2^k design, each factor has two levels, i.e. high level and low level. The total number of experiments is 2^k if there are k factors. The 2^k design can effectively reduce a total number of experiments via optimizing experimental runs so that the method saves time and efforts in the material design stage and more factors can be studied in experiments that those studied in conventional experimental design. Additionally, effect values can be calculated