Understanding the structural build-up rate of cementitious materials for 3D-printing

Luiza R. M. Miranda[1] [0000-0001-8481-5255], Karel Lesage[1] and Geert De Schutter[1] [0000-0001-5603-2616]

[1] Magnel-Vandepitte Laboratory, Ghent University, Belgium.
luiza.miranda@ugent.be

Abstract. The concrete 3D-printing technology is highly dependent on the material's rheology and on the deep understanding of how those properties evolve with time. This evolution occurs due to reversible structural build-up and irreversible chemical phenomena, like hydration reactions. To guarantee structural stability of the printing process, the material, as soon as it is deposited, needs to build-up an internal structure to withstand its weight and the weight of the following layers. Therefore, the static yield stress and how it increases over time at rest becomes of first interest. The concrete structural build-up happens due to both CSH bridge formation by nucleation of cement grains at their pseudo-contact points during the dormant period of hydration, and flocculation due to colloidal interactions. The currently accepted knowledge on the subject relates the yield stress growth with the structural build-up rate of the material through different models, as it also uses different measuring protocols and assumptions to its determination. The goal of this research is to summarize the literature on this topic, identifying the similarities and discrepancies in the available data to further propose a more suitable approach to evaluate this parameter. To do so, a systematic review was carried out in the Web of Science database with appropriate keywords. Then the data were organized and analyzed taking into account aspects such as the definition of structural build-up, the model used, simplifications/assumptions of the model, experimental protocol, mixture parameter and model limitations.

Keywords: Structural build-up rate, Thixotropy, 3D-printing..

1 Introduction

The concrete 3D-printing technology is highly dependent on the material's rheology and on the deep understanding of how those properties evolve with time. Cementitious materials have a complex time-dependent rheological behavior and can present shear-thinning viscosity, elastic region, static and dynamic yield stress, and structural build-up at rest [25].

The observed structural changes that the material undergoes after its first contact with water can be of both physical and chemical origins, and an evaluation of each parcel separately is of great difficulty. The term thixotropy is often used to describe the reversible changes occurring when the flow starts from a sample at rest and its subsequent structural recovery when the flow stops. This can only be considered as the single phenomenon happening if one decides to neglect the irreversible chemical phenomenon due to hydration reactions that starts as soon as the binder is mixed with water. The reversible changes were reported to dominate during the first hour, as long as the static yield stress increases linearly during this period [1, 26].

The static yield stress and how it increases over time at rest is currently considered as the main rheological parameter to evaluate in the layered process [27]. The concrete structural build-up happens due to both C-S-H bridge formation by nucleation of cement grains at their pseudo-contact points during the dormant period of hydration, and flocculation due to colloidal interactions [1, 26]. The water to binder ratio, then, exerts an important effect on the structural build-up once it affects both the interparticle forces (by altering the particle distance) and the hydration process (therefore influencing the nucleation and growth of hydration products) [8].

To guarantee structural stability of the printing process, the material, as soon as it is deposited, needs to build-up an internal strength to withstand its weight and the weight of the following layers. The static yield stress is the critical stress necessary to start flow from rest and therefore is key for layer stability. It is important to notice, however, that optimization is required to find a building rate that allows structural stability and also ensures the highest bonding between the deposited fresh layers [3, 27]. The buildability of 3D printed structures is reported to be related to structural build-up, the thickness of the printed layer and the printing rate [16, 17].

The correlation between static yield stress and building time showed that, with a long enough building time, the structure is mostly supported by its yield strength [23]. The currently accepted knowledge on the subject relates the yield stress growth with the structural build-up rate of the material through different models, as it also uses different measuring protocols and assumptions to its determination. Thus, the goal of this research is to summarize the literature on this topic, identifying the similarities and discrepancies in the available data to further propose a more suitable approach to evaluate this parameter.

2 Mathematical models

Roussel [2] presents a linear model for determination of yield stress growth with time, where A_{thix} appears for the first time as a flocculation rate. This model is developed to be simple and with parameters that can be easily measured, even if it is not that accurate in its determinations. It assumes that the increase in the yield stress is linear during the dormand period because the hydration heat release in this period is constant. This assumption makes the model suitable for resting times up to 60 min after the water contact and for mixtures with a clear dormand period, which is not always is the case for mixtures with a printing purpose. When setting accelerators are used and a rapid transition from the initial reactions to the acceleration period can be observed, there is almost no dormand period.

Experimental results showed that after the dormand period a rapid increase in the yield stress is commonly observed, making the behavior highly non-linear. Taking this observed behavior into account, Perrot et al. [3] proposed an exponential model that asymptotically tends to Roussel's model in the first hour and can predict the yield stress increase for the first 2 hours after mixing. This model presents A_{thix} as a structuration rate and has a characteristic time that marks the beginning of the exponential increase of the yield stress, associated with a non-negligible solid volume fraction linear increase. This time, however, is not measured but adjusted to best fit the experimental data, which means that the parameters of the model are not linked to physical properties of the cement paste.

To overcome this limitation, Lecompte and Perrot [4] developed a model that is physically based and uses the definitions of structural build-up proposed by Roussel [2] and the hydration degree of the cement paste. The authors used Roussel's model to incorporate the reversible effect due to nucleation of the cement grains and combined it with the yield stress model "YODEL" that accounts for irreversible solid volume increase due to hydration reactions, linking this solid volume fraction increase to the hydration degree (α). In this case, the parameters related to hydration and coagulation kinetics (A_{thix}) and the hydrate volume fraction within the spherical envelope were adjusted to reach the best fitting.

Differently from those approaches, Muthukrishnan et al. [6] presented what they called a bi-linear yield strength development growth. In the first few minutes after water contact, the material presents a reversible behavior due to flocculation, that is purely due to physical interactions and can be reversed by mixing, and is characterized by the parameter R_{thix} (defined as the rate at which the material yield stress grows from dynamic to static yield stress, after shearing). Then a structuration process starts to occur, as hydration reactions begin to influence the static yield stress growth. The rate at which this structuration process occurs is determined using A_{thix}. The determination of those parameters, though, was made by an average of the peak value in static yield stress tests performed at 0.1, 0.05 and 0.01 s^{-1} shear rates for a given resting time varying from 0 to 60 min.

Pan et al. [5] state that the static yield stress increase with time can be described in two steps: one that happens quickly just after mixing until it slows down and a second where the static yield stress increases slowly with time. To fit the evolution of static yield stress with time they present a thixotropic model that considers these two steps and has five fitted parameters. The authors also considered a short-term structural build-up due to flocculation, where its approximated linear growth rate is R_{thix}; and a long-term structural build-up due to the formation of early hydrates between cement grains, where its rate is A_{thix}. The model works with a turning point (t_{perc}) in which $R_{thix} = A_{thix}$. This time represents the transition from a physical process (flocculation) to a chemical structuration, so that they further defined it as maximum operational time (MOT), that has the same physical meaning as the open time while the reversible process dominates (i.e. when the resting time is less than MOT).

Navarrete et al. [12] also used the bi-linear approach to fit the static yield stress growth results but developed a second-order polynomial regression model to predict the structural build-up as a function of supplementary cementitious materials (SCM) properties (such as particle size, chemical reactivity and surface potential) and mixture parameters (such as w/c ratio, SCM replacement and cement reactivity). They found that the structural build-up is mostly affected by water to cement ratio while it is least affected by the replacement level of SCM.

The stress decay process that a cementitious material undergoes under a constant shear rate is related to structure breakdown and therefore to thixotropy. In this context, Qian et al. [20, 21, 22] defined a thixotropic index I_{thix} as the ratio between a maximum shear stress value (τ_i) needed to start flow-onset and the equilibrium value (τ_e) that define steady-state, for a constant shear rate. The I_{thix} characterizes a relationship between static and dynamic yield stress, and the higher the parameter, the higher the thixotropy of the material. Ouyang et al. [19] state that methods like static yield stress assessment and hysteresis loop, although simple, cannot properly describe structural breakdown and structure build-up of a cementitious material. Therefore, they use the flocculation structure parameter method. This method has a strict mathematical form and defines the structure parameter (λ_0) as the ratio of the difference between the initial and equilibrium shear stress to the equilibrium shear stress. The change of this parameter can be used to describe the structure build-up at rest. Chen et al. [23] also calculated the structure parameter, which they defined as S_{thix}, and stated that high S_{thix} accounts for better thixotropy.

Ivanova and Mechtcherine [10] use both Roussel [2] and Perrot et al. [3] models to fit the experimental data, but propose two different approaches to characterize structural build-up through constant shear rate tests with a single-batch: one based on a proportionality limit and another based on the flow onset points. They determined a breaking criterion in the protocol, suitable for two types of rheometers, to avoid excessive deformation of the samples and ensure similar loading conditions. Also, the proportionality limit was introduced as the point attributed to the second maximum of the apparent viscosity. They recommend calculating A_{thix} from both approaches, being the difference between them an indicator of the capacity of the material to sustain loads before failure (in the region of plastic deformation).

Mostafa and Yahia [13] evaluated the effect of mixture parameters on the physico-chemical kinetics of structural build-up. The authors presented two indices to quantify the structural build-up, one related to the resting time needed to form a structural colloidal network (the percolation time t_{perc}); and another related to the growth of stress-bearing capacity due to chemical hydration (the rigidification rate G_{rigid}). Thus, they proposed a semi-empirical model that can predict the two aforementioned indices by considering the microstructural characteristics of cement pastes, such as inter-particle cohesion (I_C), frequency of Brownian collisions (f_C), and the nucleation rate constant (K_B). The authors found good agreement between model predictions and experimental data for the cases studied.

Zhang et al. [8] present a model that describes the relationship between structural build-up rate and inter-particle forces and nucleation and growth of hydration products. It is worth mention that Zhang et al. [8] considers A_{thix} as a structural build-up parameter and propose a new value called rate of structural build-up (r_{sbu}) that describes the growth rate of static yield stress. The authors make a differentiation from

Perrot et al. [3] exponential growth model, but do not consider the same definition for the A_{thix} parameter as them. They point out that the growth rate of static yield stress does not only depend on A_{thix} and a characteristic time (t_c), but also continuously changes with time, having them found a different growth rate from 30 min to 40 min than the one from 50 min to 60 min.

As sometimes there is a delay during the test duration for the effectively applied shear rate to reach the intended applied shear rate, Narrela et al. [17] proposed a constant-strain method instead of the regular constant shear rate approach to evaluate structural build-up. The authors used both Roussel [2] and Perrot et al. [3] models to fit their data. The hypothesis presented is that as long as the applied strain is constant, measured structural build-up remains the same. This is true even if the shear rate varies, as long as compensation in the test duration is made according to the stiffness of the material. The authors suggest that the applied shear rate and the test duration should vary not only according to the different materials tested but also for a single material based on the changes of stiffness during time. It is worth mentioning that the authors found that Roussel's linear model overestimates the structuration rate before the characteristic time, which is especially problematic for the case of 3D-printing as this can lead to failure of the printed structure.

3 Experimental protocol

A great part of the works that investigate the structural build-up rate of cementitious materials uses the models developed by Roussel [2] and/or Perrot et al. [3] to fit data. Then, another question appears. Not only a model that can properly describe the structural build-up rate of cementitious material, with a simple data acquisition and physically-based assumptions, is needed; but also an experimental protocol for its parameter determination would be of great interest. As there is no standard for the determination of static yield stress, which is a main parameter required in both models, the experimental protocol varies according to the researchers' preferences and expertise.

The structural build-up rate of cement-based materials can be affected by different parameters, such as properties of the constituents materials, mixture design, ambient conditions and the shear stress history of the material (i.e. pre-shear and resting time). This parameter is directly connected with the buildability of layered structures. A feasible construction rate can be determined based on the material initial static yield stress and its structural build-up rate.

The structural build-up rate of cement-based materials is most commonly accessed by the evaluation of the evolution of static yield stress with time at rest through constant shear rate (CSR) tests and small amplitude oscillatory shear (SAOS) tests. In the CSR tests, a low shear is applied at a constant rate and the minimum stress required to initiate flow from rest is recorded. This value is identified as the peak of the shear stress curve [17]. As cementitious materials exhibit both viscous and elastic properties, during SAOS, its elastic and viscous response under continuous sinusoidal shearing within a critical oscillatory strain is recorded as storage modulus (G') and loss modulus (G″), respectively. The values at the point where G′ = G″ are reported to be the yielding point and therefore are used to determine the static yield stress [6, 20].

It is known that to accurately measure A_{thix} of a paste at rest, the tested sample should not be disturbed. According to Zhang et al. [11], it can be considered that structural build-up is only happening during the static yield stress test. That is because the applied shear rate (or stress) is so small that it causes only minimal damages to the structure of the paste. Narella et al. [17] point out that during a static test, the flow occurs at large critical strains, that according to Roussel et al. [1] are related to the network of colloidal interactions between the particles. While during the dynamic test, the flow occurs at lower critical strains, which Roussel et al. [1] related to C-S-H formation at the cement grains contact points.

What can be said, however, is that dynamic and static shear test methods evaluate different responses of the material. According to Moeini et al. [15], if the need is for the characteristic of the sheared material, the evolution of the static yield stress with time should be adopted. If the need is for the characteristic of the structuration behavior of the material, then a SAOS test is more recommended. Zhang et al. [11], on the other hand, states that static shear stress tests were used to evaluate the evolution of structural build-up in the pastes and SAOS test to characterize the instantaneous response of the structural build-up (at flowing and from flowing to standing).

SAOS is considered a non-destructive test and therefore most likely to not disturb the sample. The test requires for the sample to be sheared within the linear viscoelastic region. Yuan et al. [16] remarks on the importance of the selection of the proper strain amplitude and frequency. For cement paste a frequency of 1 Hz and a strain amplitude lower than the critical strain is appropriate. The authors also showed that the addition of superplasticizer increased the critical strain of the paste from the order of 10^{-5} to the order of 10^{-4}.

For the constant shear rate test, there are some divergences in the literature regarding the single-batch and the multi-batch approaches. For the multi-batch approach, every measure is made on a new undisturbed sample in the given age of test. According to Ivanova and Mechtcherine [10], this method has the disadvantage of not being suitable for in-situ situations besides being time, material and labor-consuming. Also, the variability between multiple samples could be larger than the variation a single-batch approach could give.

In the single-batch approach, the same sample is tested throughout all investigated ages and each measurement should stop right after the material reaches the shear stress peak value, to avoid extra deformation of the sample. Ivanova and Mechtcherine [10] state that only this approach is feasible for in-line automated control in the case of concrete 3D printing. However, this method can lead to an underestimation of A_{thix} due to the multiple disturbances of the material.

About the differences in the measurements for single and multi-batch approaches, Ivanova and Mechtcherine [10] mentioned percentage differences of A_{thix} values not greater than 9% between both approaches. Similarly, Yuan et al. [16] state small effects of the previous static yield stress measurement on the subsequent measure for the single-batch approach. Regarding pre-shearing, most of the experimental procedures adopted this step to guarantee the most dispersed state of the material's structure at the beginning of the test. Ivanova and Mechtcherine [9], however, states that pre-shearing leads to a pronounced underestimation of A_{thix}.

4 Definition of A_{thix}

According to Roussel et al. [1], two types of thixotropy can be observed in a cementitious mixture: a short term one, that is related to colloidal flocculation and it has a characteristic time of the order of a few seconds; and a long term one related to the ongoing hydrates nucleation, being the long term thixotropy of practical interest. The authors also mention that the word “structuration” is often used when characterizing consequences of thixotropic behavior, as this word is broader and therefore not associated with a specific physical phenomenon.

Navarrete et al. [12] say that thixotropy is a term usually used to describe the increase of static yield stress of cementitious materials and that one of the main properties of thixotropy is to be reversible. Nonetheless, when cement-based materials are at rest, it is not trivial to separate reversible and irreversible effects that happen due to flocculation and hydration bonding (as CSH bridges), respectively. Therefore, the authors state that “structural build-up (A_{thix})”, which comprises reversible and irreversible processes, is a more accurate term than thixotropy when referring to cement-based materials.

Reiter el at. [7], refers to “structuration” as the time evolution of yield stress for concrete at rest and emphasizes that definitions, as well as quantification techniques, for thixotropy, are especially ambiguous and frequently capture a range of flow rates and a particular shear rate history. Yuan et al. [16] describe that thixotropy has three characteristics: (i) its determination is often based on a viscosity decrease (although the evolution of static yield stress is more used in cement-based materials), (ii) it is a time-dependent property, and (iii) it is reversible. Therefore, the authors strictly state that the term is not applicable for cementitious materials, being structural build-up at rest more suitable. The authors of the paper at hand agree with these remarks and chose to refer to this phenomenon (i.e. the sum of reversible and irreversible effects) as structural build-up and, therefore, consider A_{thix} as a structural build-up rate, which characterizes the increase in static yield stress due to flocculation and early hydration [10]. However, the authors acknowledge that many studies affirm that thixotropy and structural build-up are correlated terminologies at early ages, as the former dominates over the latter during this period [18]. It remains necessary, though, to clearly explain the choice of nomenclature.

It is important to stress then, that a change of nomenclature to designate the same phenomena is not to be ignored. A_{thix} can be addressed as flocculation rate [1, 2], structuration rate [3], thixotropy factor [7], rate of increase of yield stress with time at rest [4], rate of thixotropic build-up [14], structural build-up rate [11], structural build-up parameter [8], long-term structural build-up rate [5], thixotropic index [15]. González-Taboada et al. [24] mention in their work A_{thix} as cement paste yield stress evolution in time, structuration rate of the paste and then cement paste structuration rate at rest. Muthukrishnan et al. [6] state that only after a re-flocculation stage (where the re-building is purely due to physical interactions that could be reversed by shearing), the hydration reaction starts to influence the growth of static yield strength with time, calling this process structuration in fresh concrete and its rate A_{thix}. The term R_{thix} also appears with different definitions, as it is defined by Muthukrishnan et al. [6] as the rate at which the yield strength grows from dynamic yield strength to static yield strength after the applied energy is removed and by Pan et al. [5] as the approximate linear growth rate of short-term structural build-up.

Those variations can be rather confusing for the reader. If the ‘flocculation rate’ term is used, flocculation can be considered as a part of a structuration process due to static yield stress increase at rest, but this increase also happens due to hydration reactions. If the choice is for ‘structural build-up’, an immediate relation can be made with a possible structural breakdown, which occurs in the case when yield stress is reached and the material starts to flow and during dynamic shear tests. ‘Long-term structural build-up rate’ separates the phenomena from the short-term structural build-up rate, which is the one that more directly relates to short-term thixotropy, which therefore is associated with colloidal flocculation. Once more, it is stressed how a more precise choice of terminology would allow the researches made on the topic to be properly compared.

5 Conclusions

This paper summarized the relevant literature on structural build-up rate of cementitious materials for 3D-printing technology. Different mathematical models were discussed and a tendency for the use of Roussel’s linear model and Perrot et al.'s exponential model was found, although it is clear that more research is ongoing on the topic to develop more accurate models. It became also clear that an experimental protocol for the determination of the parameters would be of great interest. As there is no standard, the experimental protocol varies according to the researchers' preferences and expertise, making it difficult to properly compare the contributions.

The differences between thixotropy and structural build-up at rest were also addressed and a more suitable terminology, that comprises both reversible and irreversible parts of the structuration process, was suggested based on definitions available in the literature.

Once more, the need for a more precise choice of terminologies is stressed, as well as a detailed explanation of the physical and/or chemical effect that is being evaluated, so that the researches made on the topic can be properly correlated.

References

1. N. Roussel, G. Ovarlez, S. Garrault, C. Brumaud, The origins of thixotropy of fresh cement pastes. Cem. Concr. Res. 42 (2012) 148–157, https://doi.org/10.1016/j.cemconres.2011.09.004.

2. N. Roussel, A thixotropy model for fresh fluid concretes: Theory, validation and applications. Cem. Concr. Res. 36 (2006) 1797–1806, https://doi.org/10.1016/j.cemconres.2006.05.025
3. A. Perrot, D. Rangeard, A. Pierre, Structural built-up of cement-based materials used for 3D-printing extrusion techniques. Mater Struct 49 (2015) 1213-1220, https://doi.org/10.1617/s11527-015-0571-0
4. T. Lecompte, A. Perrot, Non-linear modeling of yield stress increase due to SCC structural build-up at rest. Cem. Concr. Res. 92 (2017) 92-97, http://dx.doi.org/10.1016/j.cemconres.2016.11.020
5. T. Pan , Y. Jiang, H. He, Y. Wang, K. Yin, Effect of Structural Build-Up on Interlayer Bond Strength of 3D Printed Cement Mortars. Materials 14(2) (2021) 236; https://doi.org/10.3390/ma14020236
6. S. Muthukrishnan, S. Ramakrishnan, J. Sanjayan, Effect of alkali reactions on the rheology of one-part 3D printable geopolymer concrete. Cem. Concr. Comp. 116 (2021) 103899, https://doi.org/10.1016/j.cemconcomp.2020.103899
7. L. Reiter, T. Wangler, N. Roussel, R. J. Flatt, The role of early age structural build-up in digital fabrication with concrete Cem. Concr. Res. 112 (2018) 86–95, https://doi.org/10.1016/j.cemconres.2018.05.011
8. Z. Zhang, J. Xiao, K. Han, J. Wang, X. Hu, Study on the structural build-up of cement-ground limestone pastes and its micro-mechanism. Constr. Build. Mat. 263 (2020) 120656 https://doi.org/10.1016/j.conbuildmat.2020.120656
9. I. Ivanova, V. Mechtcherine, Effects of Volume Fraction and Surface Area of Aggregates on the Static Yield Stress and Structural Build-Up of Fresh Concrete. Materials 13 (2020) 1551; https://doi.org/10.3390/ma13071551
10. I. Ivanova, V. Mechtcherine, Possibilities and challenges of constant shear rate test for evaluation of structural build-up rate of cementitious materials. Cem. Concr. Res. 130 (2020) 105974 https://doi.org/10.1016/j.cemconres.2020.105974
11. Y Zhang, Z. Jiang, Y. Zhu, J. Zhang, Q. Ren, T. Huang, Effects of redispersible polymer powders on the structural build-up of 3D printing cement paste with and without hydroxy-propyl methylcellulose. Constr. Build. Mat. 267 (2021) 120551, https://doi.org/10.1016/j.conbuildmat.2020.120551
12. I. Navarrete, Y. Kurama, N. Escalon, M. Lopez, Impact of physical and physicochemical properties of supplementary cementitious materials on structural build-up of cement-based pastes. Cem. Concr. Res. 130 (2020) 105994, https://doi.org/10.1016/j.cemconres.2020.105994
13. A. M. Mostafa, A. Yahia, Physico-chemical kinetics of structural build-up of neat cement-based suspensions Cem. Concr. Res. 97 (2017) 11–27, http://dx.doi.org/10.1016/j.cemconres.2017.03.003
14. O. A. M. Reales, P. Duda, E. C.C.M. Silva, M. D.M. Paiva, R. D. Toledo Filho, Nanosilica particles as structural buildup agents for 3D printing with Portland cement pastes. Constr. Build. Mat. 219 (2019) 91–100, https://doi.org/10.1016/j.conbuildmat.2019.05.174
15. M. A. Moeini, M. Hosseinpoor, A. Yahia, Effectiveness of the rheometric methods to evaluate the build-up of cementitious mortars used for 3D printing. Const. Build. Mat. 257 (2020) 119551 https://doi.org/10.1016/j.conbuildmat.2020.119551
16. Q. Yuan, D. Zhou, B. Li, H. Huang, C. Shi, Effect of mineral admixtures on the structural build-up of cement paste. Constr. Build. Mat. 160 (2018) 117–126, https://doi.org/10.1016/j.conbuildmat.2017.11.050

17. V.N. Nerella, M.A.B. Beigh, S. Fataei, V. Mechtcherine, Strain-based approach for measuring structural build-up of cement pastes in the context of digital construction. Cem. Concr. Res. 115 (2019) 530–544 https://doi.org/10.1016/j.cemconres.2018.08.003
18. Q. Yuan, X. Lu, K.H. Khayat, et al., Small amplitude oscillatory shear technique to evaluate structural build-up of cement paste. Mater. Struct. 50 (2017) 112, https://doi.org/10.1617/s11527-016-0978-2
19. Ouyang, Y. Tan, D. J. Corr, S. P. Shah, The thixotropic behavior of fresh cement asphalt emulsion paste. Constr. Build. Mat. 114 (2016) 906–912, http://dx.doi.org/10.1016/j.conbuildmat.2016.04.024
20. Y. Qian, K. Lesage, K. El Cheikh, G. De Schutter, Effect of polycarboxylate ether superplasticizer (PCE) on dynamic yield stress, thixotropy and flocculation state of fresh cement pastes in consideration of the Critical Micelle Concentration (CMC). Cem. Concr. Res. 107 (2018) 75–84, https://doi.org/10.1016/j.cemconres.2018.02.019
21. Y. Qian, G. De Schutter, Enhancing thixotropy of fresh cement pastes with nanoclay in presence of polycarboxylate ether superplasticizer (PCE). Cem. Concr. Res. 111 (2018) 15–22 https://doi.org/10.1016/j.cemconres.2018.06.013
22. Y. Qian, G. De Schutter, Different Effects of NSF and PCE Superplasticizer on Adsorption, Dynamic Yield Stress and Thixotropy of Cement Pastes. Materials 2018, 11(5), 695; https://doi.org/10.3390/ma11050695
23. M. Chen, L. Li, J. Wang, Y. Huang, S. Wang, P. Zhao, L. Lu, X. Cheng, Rheological parameters and building time of 3D printing sulphoaluminate cement paste modified by retarder and diatomite. Const. Build. Mat. 234 (2020) 117391, https://doi.org/10.1016/j.conbuildmat.2019.117391
24. I. González-Taboada, B. González-Fonteboa, F. Martínez-Abella, S. Seara-Paz, Thixotropy and interlayer bond strength of self-compacting recycled concrete. Const. Build. Mat. 161 (2018) 479–488, https://doi.org/10.1016/j.conbuildmat.2017.11.157
25. R. P. Ferron, A. Gregori, Z. Sun, S. P. Shah Rheological Method to Evaluate Structural Buildup in Self-Consolidating Concrete Cement Pastes. ACI Materials Journal 104 (3) (2007) 242-250.
26. A. M. Mostafa, A. Yahia, New approach to assess build-up of cement-based suspensions. Cem. Concr. Res. 85 (2016) 174–182, http://dx.doi.org/10.1016/j.cemconres.2016.03.005
27. A. Perrot, T. Lecompte, P. Estelle, S. Amziane, Structural build-up of rigid fiber reinforced cement-based materials. Mat. Struct. 46 (2013) 1561–1568, https://doi.org/10.1617/s11527-012-9997-9