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The Yield Stress of Cement Pastes as Obtained by Different Rheological Approaches

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Abstract:

Different rheological methods for yield stress estimation of cement pastes during initial hydration were used and results were compared. These methods include measuring of the hysteresis loop, flow curves (recalculated to the same time of hydration) and large amplitude oscillating strain (LAOS). Experiments were performed with four Ordinary Portland Cements from one manufacturer, produced at different factories and one polycarboxylate acid based superplasticiser (SP). The yield stress values obtained by constructing flow curves is the only method which gives information about the evolution of the rheological properties, reflecting structure evolution of cements pastes. It was shown that the yield stress values established by the LAOS method and that calculated from the flow curves are similar while the values found from the downward part of the hysteresis loops are much lower. Differences in the yield stress values obtained by various methods are related to the different states of the material corresponding to the kinetics of hydration. The hysteresis loops provide information about thixotropic characteristics of the material including characteristic times of rebuilding and the rate of yield stress evolution of cements. The rheological properties are very sensitive to the chemical and physical differences of the cements and could be used for their characterization.

Key words:

Yield stress, hydration, superplasticiser, rheology, flow curves, hysteresis loops, thixotropy

1 INTRODUCTION

The fresh state of cement paste is becoming increasingly important in advancing new applications of cementitious materials such as self-compacting and high performance concrete in the construction industry. All concrete processes require an understanding at paste level where microstructural changes within the cement paste occurs, influencing both the level of fluidity and rate of solidification. Both of these factors are the subject of the rheological studies and in principle, can be quantified by a set of standard rheological equations and appropriate parameters. Rheology has been the tool used over the years by many researchers to characterize the technological properties of cementitious materials. Cement paste can be treated as a colloidal material with flow properties demonstrating numerous non-Newtonian effects including shear rate dependence of the apparent viscosity and the existence of a yield stress [1–3]. Temperature is also a factor strongly influencing the rheological properties and kinetics of their evolution [4]. Cement pastes are 'living' systems with continuously changing structure and consequently, their rheological properties. The progression of cement paste hydration causes the properties of cement paste to evolve from viscous fluid to solid material [5]. This transformation of cement from fluid to a solid material is referred to by [4] as setting, attributing the yielding of calcium silicate hydrate (C-S-H) as its main contributing factor.

The dependency of cement paste rheological parameter evolution on the hydration kinetics of cement was described in Quanji [6] and its rate is dictated by the main compounds of the cement clinker. One can find many examples of experimental observations of changes in the rheological properties of cement paste during the dormant phase of hydration [5–7]. This process starts a few seconds after the end of mixing and affects the development of the yield stress [7, 8]. Generally speaking, the yield stress is considered as the strength of the interparticle attractive forces responsi-

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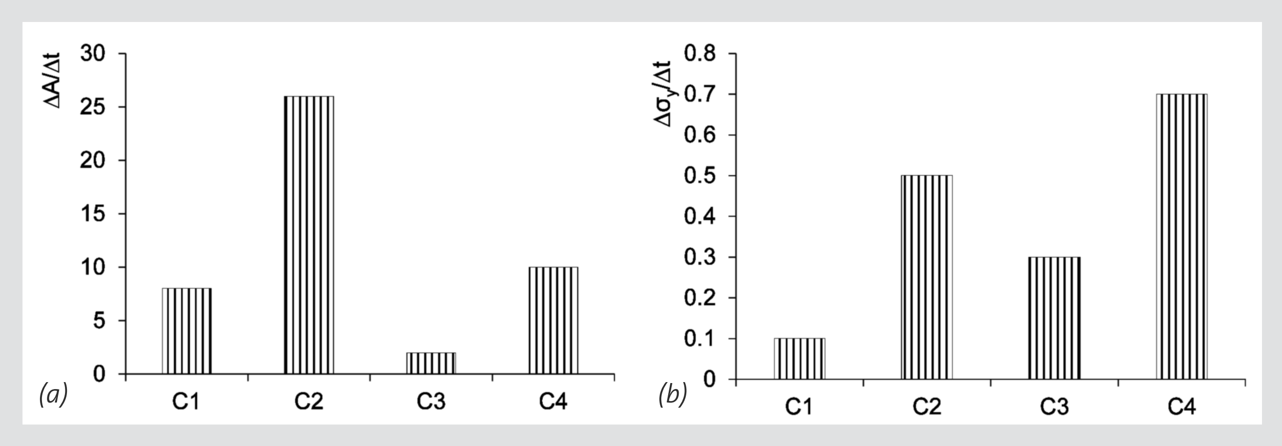

Figure 9: Rate of rebuilding (a) and rate of yield stress changing (b) for all cements without SP.

ent times of hydration, calculating the yield stress from the downward part of the hysteresis loop and attributing the yield stress to the point of deviation from linearity in the method of large amplitude oscillating strain.

Experimental data obtained for four CEM I 52.5N cements with and without polycarboxylate acid based superplasticiser show that the yield stress obtained by constructing the flow curve is the only method which gives information about the evolution of the rheological properties, reflecting structure evolution of cements pastes. The yield stress values of cement paste as determined by amplitude sweep are always higher compared to the two other methods due to the fact that during experiments the hydration is not disturbed and the microstructure is not destroyed. However, the yield stress values obtained from the flow curves are close to those evaluated by the amplitude sweep method.

Yield stress values determined by hysteresis loops appear to be very low compared to the values obtained by the two other techniques. This might be explained by the fact that during the experiments, the structure of the material is continuously destroyed due to shearing. The pre-shearing at 50 1/s for less than 15 s is sufficient to destroy the cement paste structure formed even after 15 minutes. The areas of the thixotropy loops are proportional to the resting time due to the cement paste microstructure development. This area is reduced with the addition of superplasticiser, the magnitude depending on the interaction of SP and cement under investigation. There is a correlation between the rate of rebuilding and the rate of yield stress evolution of cements, such as low rate of structure rebuilding resulted in slow yield stress development within the cement paste.

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