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Rheological Behavior and Workability Box on Foamed Cement Pastes Made with Alternative Reagents

Comportamiento Reológico y “Workability box” de pastas de cemento espumadas fabricadas con reactivos alternativos

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Abstract

Foamed concrete is a type of lightweight concrete. The porous structure is due to foam in addition to the cementitious mixture. The fresh state of foamed mixtures represents a critical point because workability conditions are not typically used for conventional concretes. Also, the porous structure is highly influenced by the fresh state behavior and, therefore, the properties of the hardened state. Rheology is an alternative to determine the flow behavior of the fresh state. This paper presents the results from a rheological characterization of OPC foamed pastes made with alternative reagents. Results show the connection between rheological parameters and fresh-state performance. A workability box with three zones has been proposed to present this connection. Zone I includes foamed pastes with viscosity under 0.08 Pa·s; Zone II represents mixtures with higher viscosity and yield stress, and Zone III, which is the optimal workability zone, is limited by yield stress under 9Pa. This tool is essential to design foamed concretes.

Keywords: Rheology; foamed concrete; workability box; alternative reagents.

Resumen

El concreto celular es un tipo de concreto ligero. Su estructura porosa a la adición de espuma a la mezcla cementicia. El estado fresco de las de mezclas espumadas representa un punto crítico, ya que las condiciones de trabajabilidad de este tipo de concreto no son las mismas que se utilizan habitualmente en las mezclas convencionales. También, la estructura porosa está fuertemente influenciada por el comportamiento en estado fresco, lo que se refleja en las propiedades del estado endurecido. La reología es una alternativa para determinar el comportamiento de flujo en estado fresco. Este artículo presenta los resultados de una caracterización reológica de pastas espumadas de cemento elaboradas con reactivos alternativos. Los resultados muestran la relación entre los parámetros reológicos y el comportamiento en estado fresco. Se propone un workability box con tres zonas para representar esta relación. La Zona I incluye pastas espumadas con viscosidad inferior a 0.08 Pa·s; la Zona II representa mezclas con mayor viscosidad y límite elástico, y la Zona III, que es la zona de trabajabilidad óptima, está limitada por un límite elástico inferior a 9 Pa. Esta herramienta es esencial para el diseño de concretos celulares.

Keywords: Reología; concreto espumado; workability box; reactivos alternativos.

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1. Introduction

Cellular concrete is a type of lightweight concrete. It is created by the uniform distribution of air bubbles throughout the paste mix. The American Institute of Concrete (ACI) in CT-13, Concrete Terminology, defines cellular lightweight concrete as a low-density product consisting of Portland cement, cement-silica, cement-pozzolan, lime-pozzolan, or lime-silica pastes, or pastes containing blends of these ingredients and having a homogeneous void or cell structure (ACI Standars, 2013). The materials used to make it are the same used for normal-use concrete, except for the reagents that produce the air cells.

There are several methods to accomplish the porosity in cellular concretes: chemical agents that include the air in the mortar by chemical reaction, foaming agents that are added to the mixture, or vacuum curing that manages to create pores due to the internal strains generated in the paste. The most used foaming agents are synthetic and protein-based, but detergents, glue resins, and saponins may also be used (L. Chica & Alzate, 2019; Gaviria-Hdz et al., 2019). Use of foaming agent can be done by preformed foam or in mixing methodfoaming (Narayanan & Ramamurthy, 2000). The pre-formed foam method requires the use of compressed air equipment to create foam. The foam is then added to the cement paste. In the in-mixing method, the foaming agent adds cement with hydration water. This method is easy to perform, standardized, and widely used. However, it can produce a big volume of damaged bubbles due to the high rotating speed, which compromises the amount of air included (L. Chica & Alzate, 2019; Beltrán & Chica, 2023).

In the fresh state, the mixture of foamed concrete is fluid and self-compacting. The fresh state properties such as consistency, stability, and workability are strongly influenced by the W/C ratio, the additives, and the foam type and volume (Amran et al., 2016). The consistency and stability of fresh foamed concrete need to be controlled to prevent the separation/breaking of bubbles (Hajimohammadi et al., 2018). Consistency, stability, and workability are related to the fresh mix rheological properties (Lim et al., 2014; Beltrán & Chica, 2023). The rheology of concrete affects mixing, handling, transportation, pumping, consolidation, finishing, surface quality, and mechanical properties after hardening (Zhang et al., 2019). Even more, the rheological behavior of concrete evolves with time because of cement hydration, which is a complicated subject (Jiao et al., 2017).

There have been many attempts to characterize the consistency of fresh concrete by a variety of technological tests, but only a few researchers have applied rheological behavior modeling (de Larrard et al., 1998). In conventional concretes, for high-slump concrete, it has been found that it is best described using the Herschel-Bulkley model, but for low-slump concrete, the Bingham model provides a good description of the flow (Daoud, 2008). The fundamental parameters for describing rheological properties in these models include yield stress. τ_0 and plastic viscosity μ (Banfill, 2018). Some studies (Banfill, 2018; C. F. Ferraris, n.d.; Daoud, 2008) report yield stress and plastic viscosity values for cement–based materials. For cement pastes and grouting, yield stress is reported between 10 and 100Pa, for mortars under 400Pa, and for concretes until 2000Pa. However, foamed pastes or foamed concretes are not included. For conventional concretes, it has been reported that the factors affecting concrete´s rheological behavior are dosage, aggregate quantity and size, and water content. However, the same mixture design can result in different flow properties if other factors are not considered: mixer type, mixing sequence, mixing duration, temperature, transportation, consolidation, and finishing methods (C. Ferraris et al., 2001). Foamed concrete is an extraordinarily complex fluid that not only contains particulate materials but also contains entrapped air. This paper presents the results from a rheological characterization and a workability box for cement foamed pastes made with alternative reagents by in-mixing preparation. Accordingly, the objectives of this research are:

- Rheological modelling for foaming pastes made with alternative foaming reagents
- Yield stress and plastic viscosity are evaluated as function of mix design
- Workability box is proposed to foamed OPC pastes made with alternative reagents

The remainder of this article is structured as follows. Section 2 presents the background of the study, detailing the theoretical. Section 3 describes the research methodology, outlining materials and preparation methods. Section 4 discusses the main results, including a workability box tool. Section 5 presents the conclusions, highlighting practical contributions and its limitations.

2. Background

Foamed concrete shows non-Newtonian behavior. Constitutive equations such as Bingham, Herschel–Bulkley, and Power Law are reported in the literature for these concretes (Ahmed et al., 2009; Beltrán & Chica, 2023). Plastic viscosity is a good indicator of segregation resistance and workability (Feneuil et al., 2019; Feneuil, 2019). A higher w/c ratio would result in a lower relative viscosity but in a weaker bubble-maintaining capacity in cement paste (Liu et al., 2016). The yield stress can be used to characterize the filling ability and stability of fresh concrete (Jiao et al., 2017). For short-time processes such as pumping and stirring, yield stress cannot be relevant, but for longer-term processes such as those impacted by gravity or sedimentation, establishing the presence of a true yield stress can be important (Malvern Instruments, 2012).

The effect of surfactants on the yield stress of foamed cement paste has been studied (Feneuil et al., 2017). For anionic surfactants, yield stress depends strongly on residual concentration (Feneuil et al., 2017). The shear rates in different processes are summarized by different authors, showing that concretes are used in low values of shear rate: mixing truck 10 s-1, pumping 20-40 s-1, casting 10 s-1, mixing 120 to 700 s-1 (Yahia et al., 2015; Yang et al., 2019). Also, there is a strong connection between the rheological model and the fresh state properties.

The rheological behavior of pastes was affected by their shear history (Yang et al., 2019). Changes happened once the preparation mixing speed is altered (Lim et al., 2014; Chiara F. Ferraris, Peter Billberg, Raissa Ferron, Dimitri Feys, Jiong Hu, Shiho Kawashima, Eric Koehler, Mohammed Sonebi, Jussara Tanesi, 2017). It has been shown that increasing plastic viscosity will increase the pumping resistance (Daoud, 2008). Concrete with high viscosity tends to have problems with the finishing (Daoud, 2008).

In conventional concretes and aerated concretes, (Chiara F. Ferraris, et al., 2017) found that segregation can be controlled by increasing the yield stress or the plastic viscosity, and a good surface finish can be achieved by having adequate viscosity. They defined a workability box with three zones for self-compacting concretes. In zone I, there can be an elevated risk for segregation. In zone II, the mixture can stabilize entrapped air bubbles. Mixtures in Zone III will have good surface quality. These results could be extended to foamed concrete.

3. Experimental

An experimental set of tests was performed. All mixtures were made with OPC with different water/binder ratios and reagent dosages. An ordinary OPC cement by Argos Colombia was used to prepare the foamed paste. Cement content was fixed at an equivalent of 500 kg per cubic meter for all tests. Conventional air entraining reagents - at higher doses - were used as an alternative foaming reagent. SikaAer® and AirToc D® were used for this experimental work, and the dosages are set at 3, 4, and 5% with respect to OPC content. Reagent selection corresponds to commercial availability around the world and the previous experimental settings using it, as reported by(L. M. Chica & Alzate, 2022) (L. Chica et al., 2022). An in-mixing method in a Hobart N50A planetary mixer (a low-speed shear mixer under 100 rpm), as (Mukhopadhyay & Jang, 2009) recommended, was used. This equipment has been proven to be suitable for creating foam using the in-mixing method through experimentation. The water-to-cement ratio was set at higher values than normal mixtures (0.6, 0.7, 0.8) because of the production method and the nature of foaming agents. Table 1 shows the experimental set of tests and their conditions. Replication tests were performed to determine the reproducibility of the experimental setup.

Table 1. Experimental test.

Test number	W/B ratio	Foaming agent [%]
1	0.6	3
2	0.6	4
3	0.6	5
4	0.7	3
5	0.7	4
6	0.7	5
7	0.8	3
8	0.8	4
9	0.8	5

Rheological measurements were made in a rotational rheometer Rheotest RN in 25mm concentric cylinders. On stationary measurements, the shear rates employed were set between 0 s-1 and 1200 s-1 from a literature review showing that this is the shear rate range in industrial applications for self-compacting concretes. Concentric cylinders H2 (cup diameter: 38 mm, rotor diameter: 27.5 mm) were used as a measurement system with a 5.25 mm gap. Rheological measurements were made at contrasting times: 0min (fresh state), 30 min, 60 min, and 120 min to evaluate the rheological behavior over time. Dynamic strain sweeps for some samples were made after analyzing the stationary results at 120 min. The frequency was set at 1 Hz as (Feys et al., 2017) recommended.

4. Results

4.1 Rheological characterization results

Rheograms with experimental data were obtained. The effect of reagent dosage on the flow curve is schematically presented in Figure 1. Shaded regions represent the interval for rheological behavior for foamed OPC pastes. For shear rates over 250s-^{1}, bubbles are destroyed, and the flow curve cannot be represented. Differences in the values of the rheograms indicate that reagent type affects rheological behavior even with the same dosage. Superior limit on flow curves (superior line of the shaded region) represents experimental conditions associated with lower reagent dosage and higher water content. Rheological modeling was performed through regression, obtaining the Bingham model $\tau=\mu\gamma+\tau_{\_0}$ It is the most adjusted constitutive model for evaluated conditions, showing an R^2 greater than 0.90. Table 2 shows the results for yield stress τ0 and plastic viscosity μ and their evolution with time. Yield stress and plastic viscosity tend to increase for all dosages used up to 60 minutes after preparation. However, results show that longer times after preparation (120 minutes after hydration begins) yield stress and plastic viscosity decreases, especially when the water/cement ratio and reagent dosage are higher.

For foaming agents, yield stress and plastic viscosity have a strong connection. Figure 2 reveals the relationship between these parameters for each reagent; it considers all experimental results: Figure 2a) related to SikaAer; Figure 2b) associated with AirToc D. Also, time evolution between fresh state - 0 minutes- and 60 minutes after preparation is presented in Figure 2. Dashed lines represent the data trend. For the fresh state, the yield stress rate change (as a function of plastic viscosity) is similar for both reagents (the slope of the grey dashed lines is the same).
Nevertheless, the line that represents 60 min and after is different for both reagents. Thus, for Airtoc-D, significant changes in yield stress do not exist due to changes in plastic viscosity due to OPC hydration.

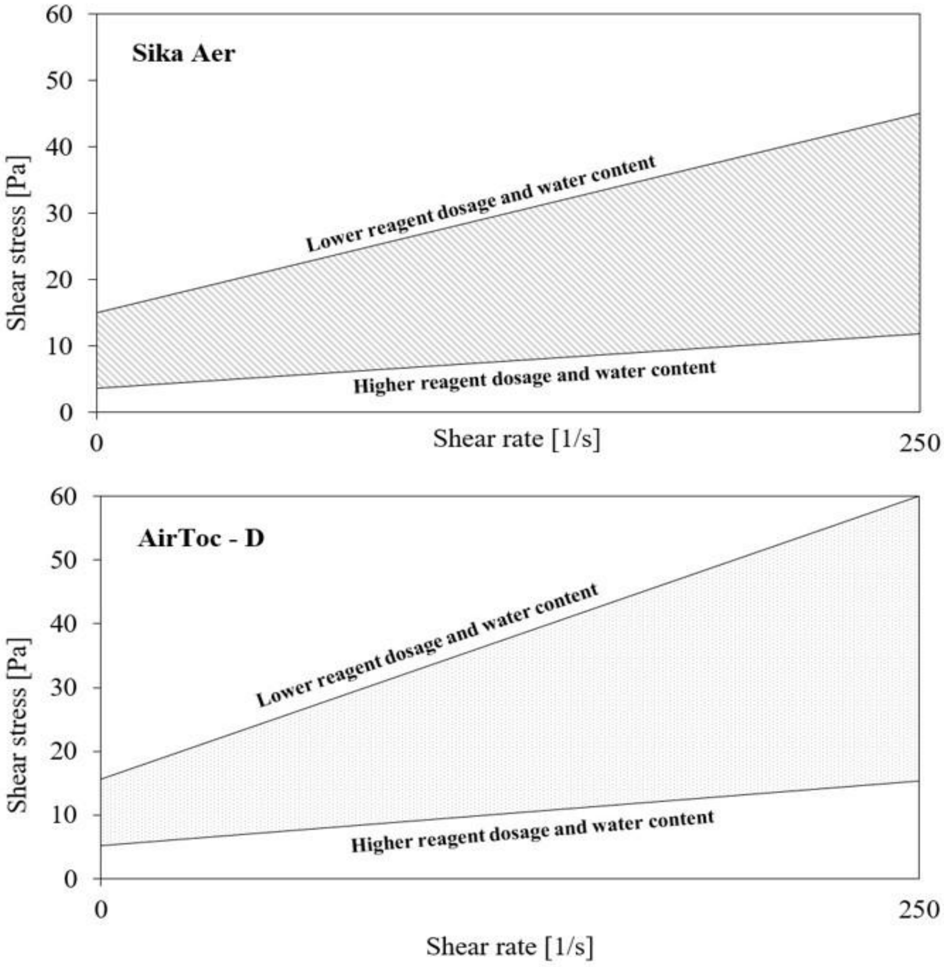
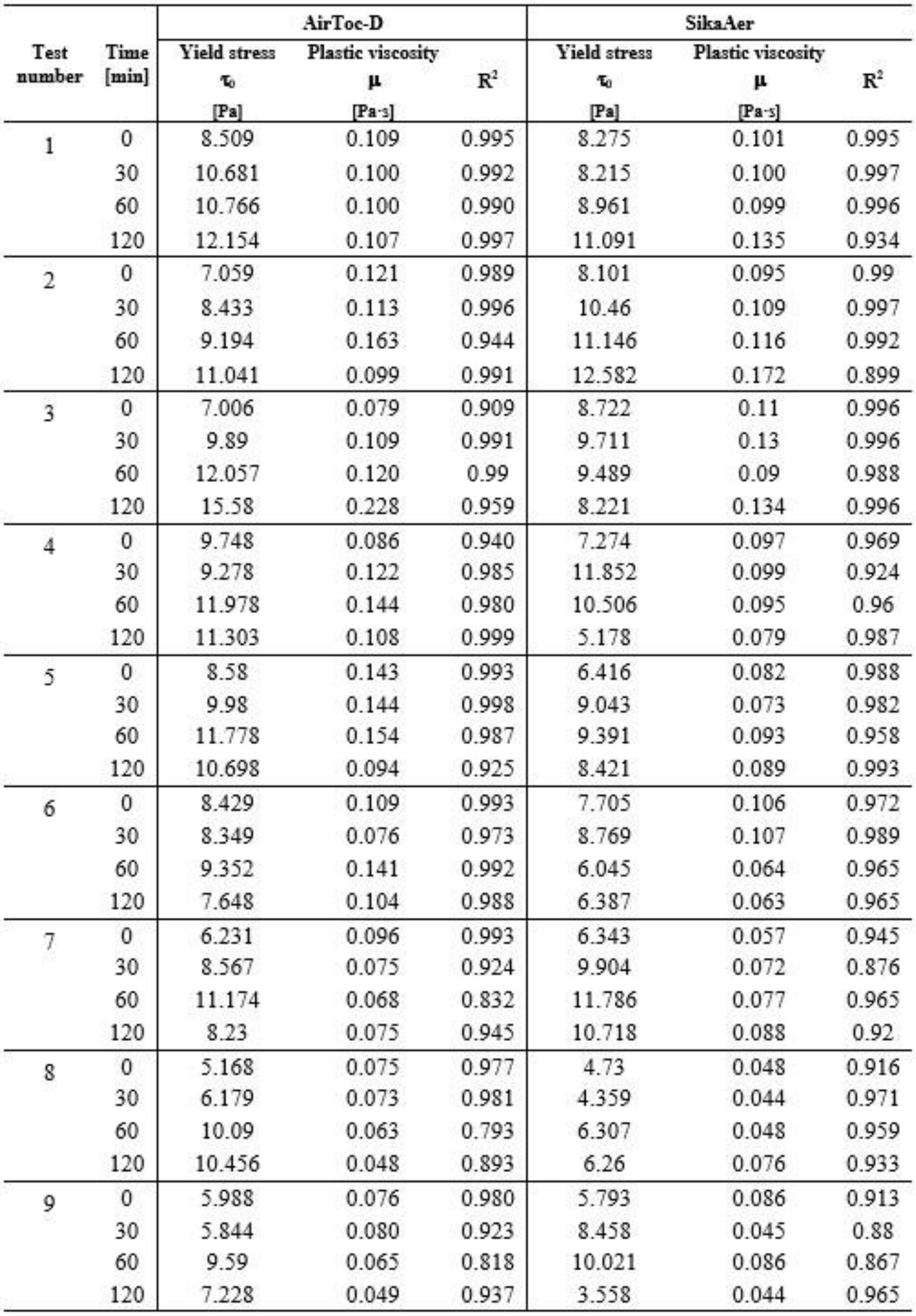


Figure 1. Effect of dosage on rheograms for foamed paste with alternative reagents. a) Plot for Sika Aer reagent. b) Plot for AirToc-D reagent.

Table 2. Bingham model parameters for foaming OPC pastes.



Test number	Time [min]	AirToc-D Yield stress τ_0 [Pa]	AirToc-D Plastic viscosity μ [Pa·s]	AirToc-D R^2	SikaAer Yield stress τ_0 [Pa]	SikaAer Plastic viscosity μ [Pa·s]	SikaAer R^2
1	0	8.509	0.109	0.995	8.275	0.101	0.995
	30	10.681	0.100	0.992	8.215	0.100	0.997
	60	10.766	0.100	0.990	8.961	0.099	0.996
	120	12.154	0.107	0.997	11.091	0.135	0.934
2	0	7.059	0.121	0.989	8.101	0.095	0.99
	30	8.433	0.113	0.996	10.46	0.109	0.997
	60	9.194	0.163	0.944	11.146	0.116	0.992
	120	11.041	0.099	0.991	12.582	0.172	0.899
3	0	7.006	0.079	0.909	8.722	0.11	0.996
	30	9.89	0.109	0.991	9.711	0.13	0.996
	60	12.057	0.120	0.99	9.489	0.09	0.988
	120	15.58	0.228	0.959	8.221	0.134	0.996
4	0	9.748	0.086	0.940	7.274	0.097	0.969
	30	9.278	0.122	0.985	11.852	0.099	0.924
	60	11.978	0.144	0.980	10.506	0.095	0.96
	120	11.303	0.108	0.999	5.178	0.079	0.987
5	0	8.58	0.143	0.993	6.416	0.082	0.988
	30	9.98	0.144	0.998	9.043	0.073	0.982
	60	11.778	0.154	0.987	9.391	0.093	0.958
	120	10.698	0.094	0.925	8.421	0.089	0.993
6	0	8.429	0.109	0.993	7.705	0.106	0.972
	30	8.349	0.076	0.973	8.769	0.107	0.989
	60	9.352	0.141	0.992	6.045	0.064	0.965
	120	7.648	0.104	0.988	6.387	0.063	0.965
7	0	6.231	0.096	0.993	6.343	0.057	0.945
	30	8.567	0.075	0.924	9.904	0.072	0.876
	60	11.174	0.068	0.832	11.786	0.077	0.965
	120	8.23	0.075	0.945	10.718	0.088	0.92
8	0	5.168	0.075	0.977	4.73	0.048	0.916
	30	6.179	0.073	0.981	4.359	0.044	0.971
	60	10.09	0.063	0.793	6.307	0.048	0.959
	120	10.456	0.048	0.893	6.26	0.076	0.933
9	0	5.988	0.076	0.980	5.793	0.086	0.913
	30	5.844	0.080	0.923	8.458	0.045	0.88
	60	9.59	0.065	0.818	10.021	0.086	0.867
	120	7.228	0.049	0.937	3.558	0.044	0.965

The effect of dosage on yield stress and plastic viscosity is represented in Figure 3a. In this Figure, each contour corresponds to the same dosage at a different time. When the W/C ratio increases, curves move downwards and to the left. Also, the values of yield stress and viscosity are lower when Sika Aer is used as a reagent. Arrows in Figure 3b) illustrate the effect of dosage on rheological parameters of foaming agents.

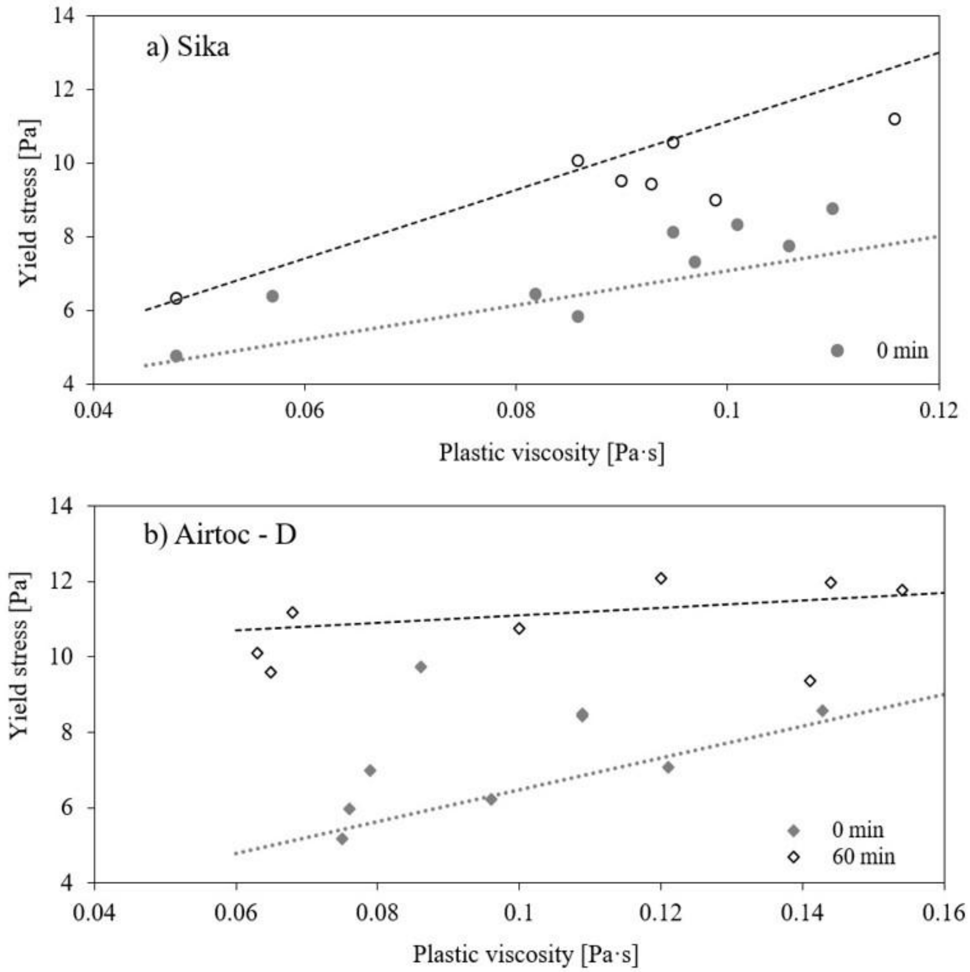


Figure 2. Yield stress as a function of plastic viscosity and its evolution over time. a) Plot for Sika Aer reagent. b) Plot for AirToc-D reagent.

Banfill reports a summary of the effect of mixture parameters on the rheology of fresh mortars (Banfill, 2018). The effect of water content is similar to the results obtained for this research work on foamed pastes. As stated above, experimental data at 120 minutes after preparation are not consistent with expected results. Observations indicate that foamed pastes at that time present regions where OPC is hydrated; thus, the setting process has already advanced. For this reason, when the cylinder of the rheometer rotates, it destroys the OPC hydration crystals and therefore sedimentation of its fragments, leaving in the upper part a greater amount of fluid. This explains why viscosity and yield strength decrease, causing stationary tests to be not accurate. A dynamic characterization was required to establish the proportion of material that has already been set and would be represented by the storage modulus G'. Figure 4 shows a dynamic strain sweep for two mixtures at 120 min. As (Banfill, 2018) presented, strain amplitude sweeps are used to determine the critical strain or stress of cement paste, which corresponds to the LVE or the onset of flow. However, in cement pastes, rheological behavior analysis is affected by the agitation that causes the rupture of the crystalline system. Figure 4a shows the response of the Sika Aer sample to the oscillatory test within the LVR region. At initial deformations and high stresses, the storage modulus is higher than the loss modulus, but in deformations greater than 35%, the behavior tends to be more viscous; thus, it is controlled by the loss modulus. Phase angle moves between 23 and 70°, as an indicator that this sample has viscoelastic behavior as a function of amplitude sweep. In Figure 4b, an example of an AirToc sample is presented. A similar behavior, as it was described before, is presented. However, the regions of elastic and viscous behavior are delimited by 27% of deformation. Experimental results for all the tests conducted establish that at a higher W/C ratio, mixtures tend to be more viscous (loss modulus dominates). Dynamic rheological results are significantly useful to describe the effect of the type of reagent on stress history response on foamed pastes.

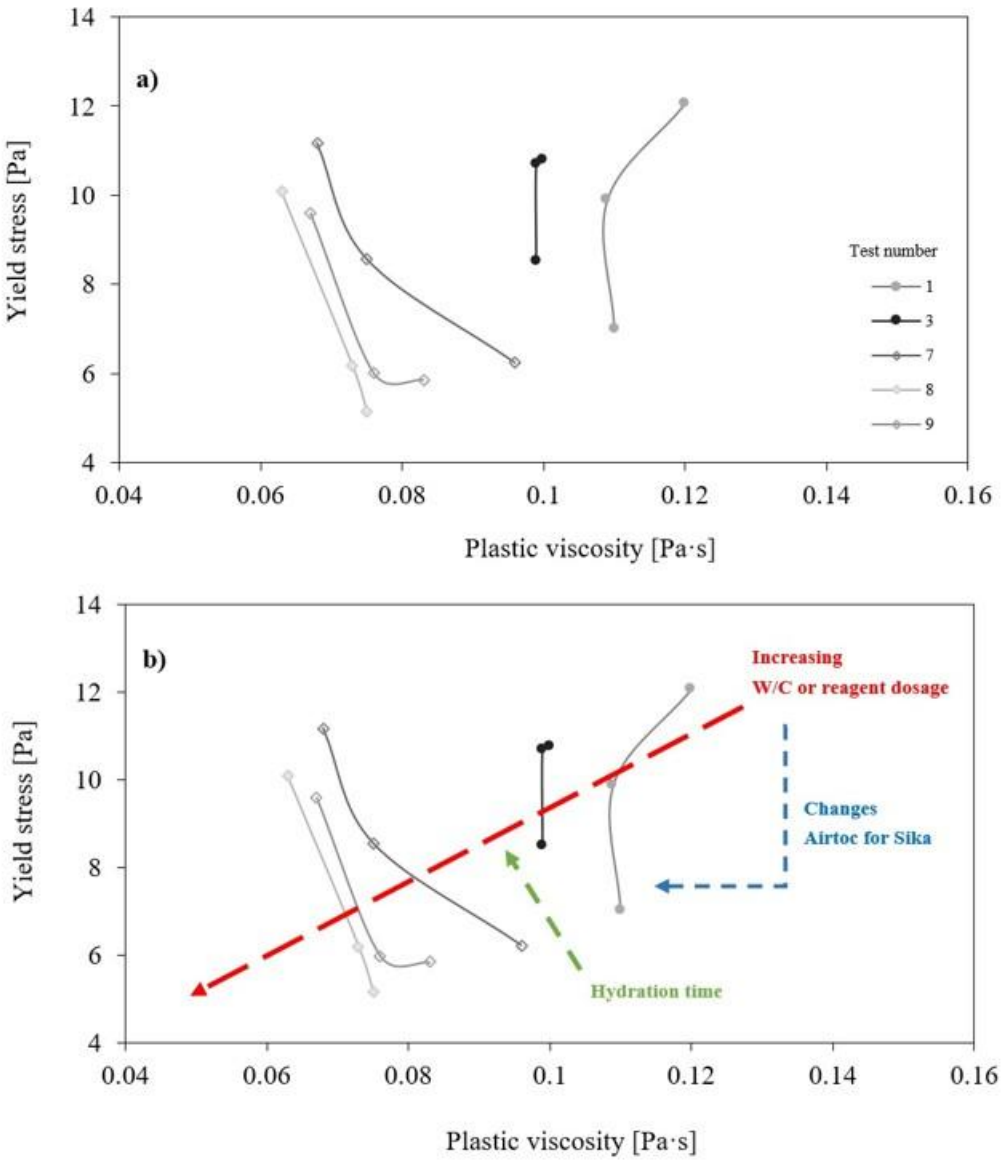


Figure 3. Effect of dosage on rheological parameters for OPC foamed pastes.

4.2 Workability box for high fluidity OPC foamed pastes

Some studies, such as (Wallevik & Wallevik, 2011; Jiao et al., 2017) (Chiara F. Ferraris, et al., 2017) proposed workability boxes to present rheological results, even on self-compacting concretes. However, rheological results for foamed pastes are extremely limited. An analysis of rheological results for this experimental study reveals that foamed pastes are highly fluid. In the boxplot proposed by (Wallevik & Wallevik, 2011) it does not consider the foamed pastes region, thus it is a lower region of yield stress and viscosity. Although (Chiara F. Ferraris, et al., 2017) proposed another box for self-compacting concretes, including the region not previously included, the results always indicate an elevated risk of segregation. Due to the nature of foamed agents and the high-water content in foamed pastes, a workability box for highly fluid foamed pastes is required. Figure 5 presents a workability box for samples made with alternative reagents when the time is 0 hours. All measures – including replication results- are considered.

The criteria defined by (Feneuil et al., 2019) for yield stress were used as a starting point. In this criterion, optimal yield stress was set at 10 Pa. Experimental results for non-conventional reagents show this value set at 9 Pa. When the yield stress value is higher than 9 Pa, cement foams evolved significantly before setting up, leading to uncontrolled morphology, the same as (Feneuil et al., 2019) found. Regarding viscosity, mixtures with viscosities under 0.08 Pa·s have extreme bleeding. Also, foam is highly unstable, and big bubbles tend to suffer coalescence.

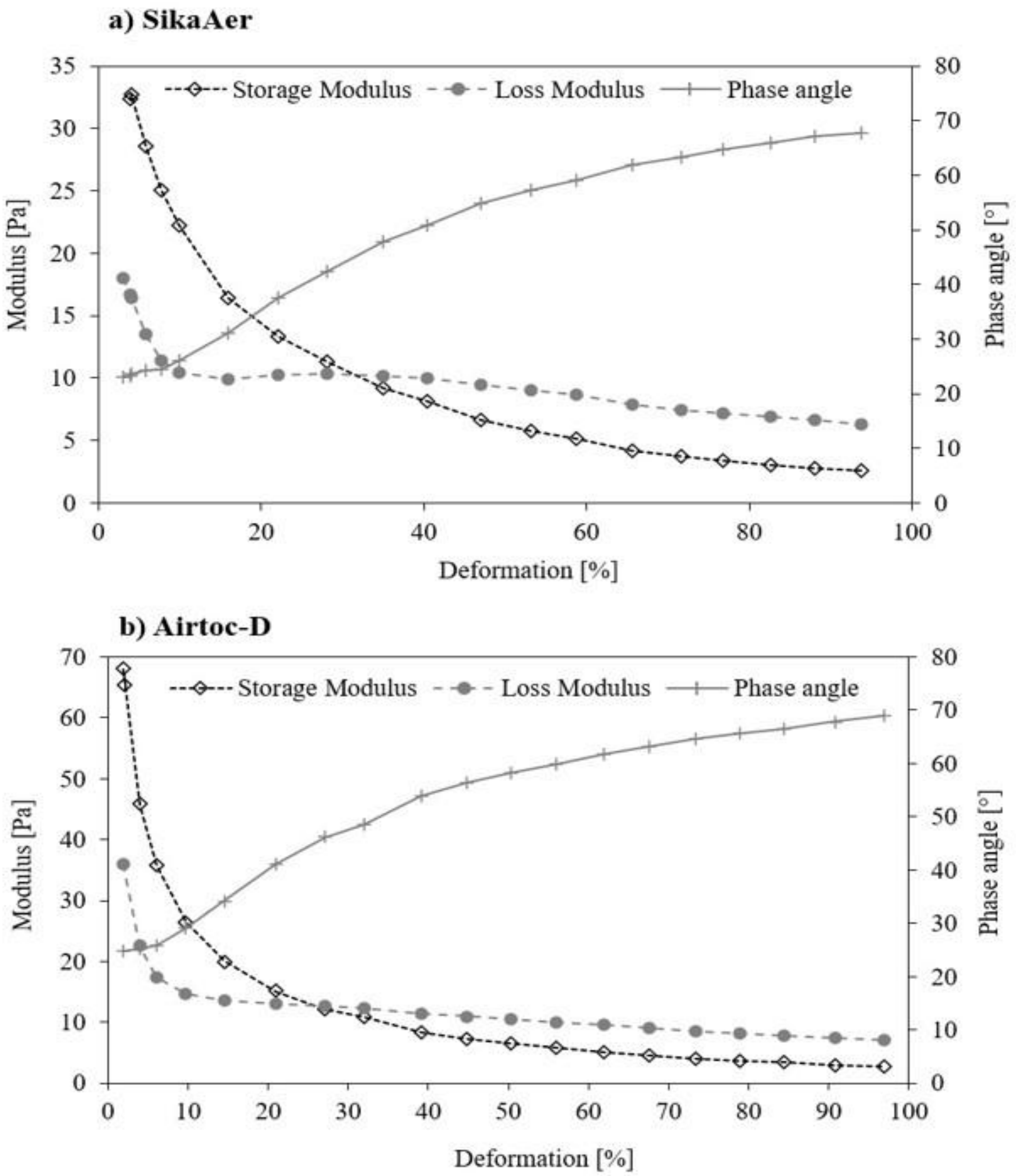


Figure 4. Oscillatory test response of cellular OPC pastes. a) Sika Aer b) Airtoc.

The workability box proposed for foamed pastes (See Figure 5) has three zones, equivalent to the zones presented in (Chiara F. Ferraris, et al., 2017). Each zone represents paste flow conditions and foamed stability. Zone I includes foamed paste with lower yield stress and viscosity. In this zone, foam is extremely unstable, and bubbles collapse. Bleeding and water drainage are present in all mixtures here. Also, foam segregation can be observed. In a hardened state, these pastes show high shrinkage and low compressive strength. Zone II represents mixtures with higher viscosity and yield stress. Mixtures in Zone II present less workability than mixtures in Zone III, but the foam is stable. Bubbles are smaller and isolated, increasing consistency and reducing segregation. However, these mixtures require more energy to flow. In a hardened state, mechanical properties are improved because fine and close pores result in a compacted texture. Zone III is the optimal workability zone. Consistency helps reduce bleeding and segregation. Stable cement foams can be produced when the cement paste yield stress is low. Viscosity is high enough to permit bubbles to stay dispersed, but the paste is fluid and has low yield stress. Depending on the industrial application and placement conditions, users can define their interest in Zone II or III. Figure 6 shows the difference in pore structure in the hardened state. Morphology of the hardened foamed pastes depends on foam stability and bubble size distribution.

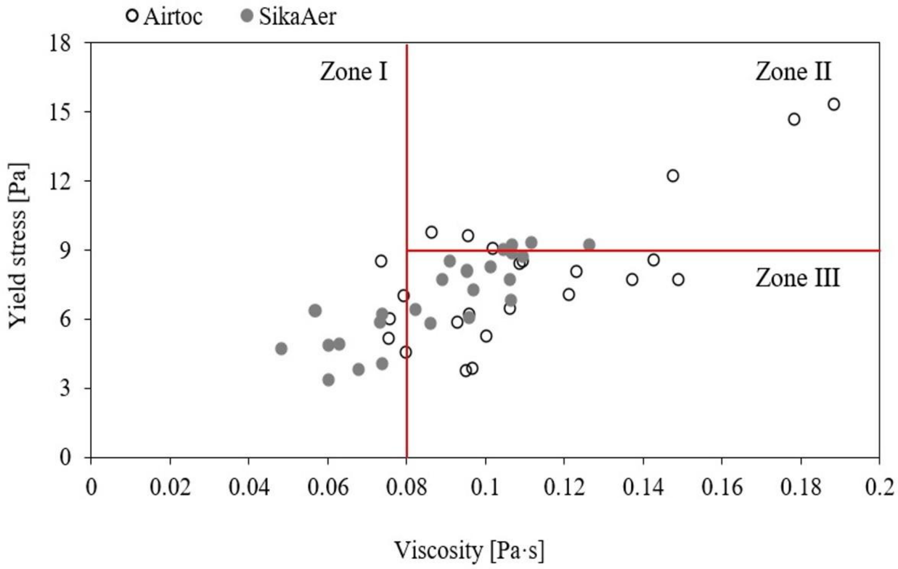


Figure 5. Workability box for OPC foamed pastes made with alternative reagents.

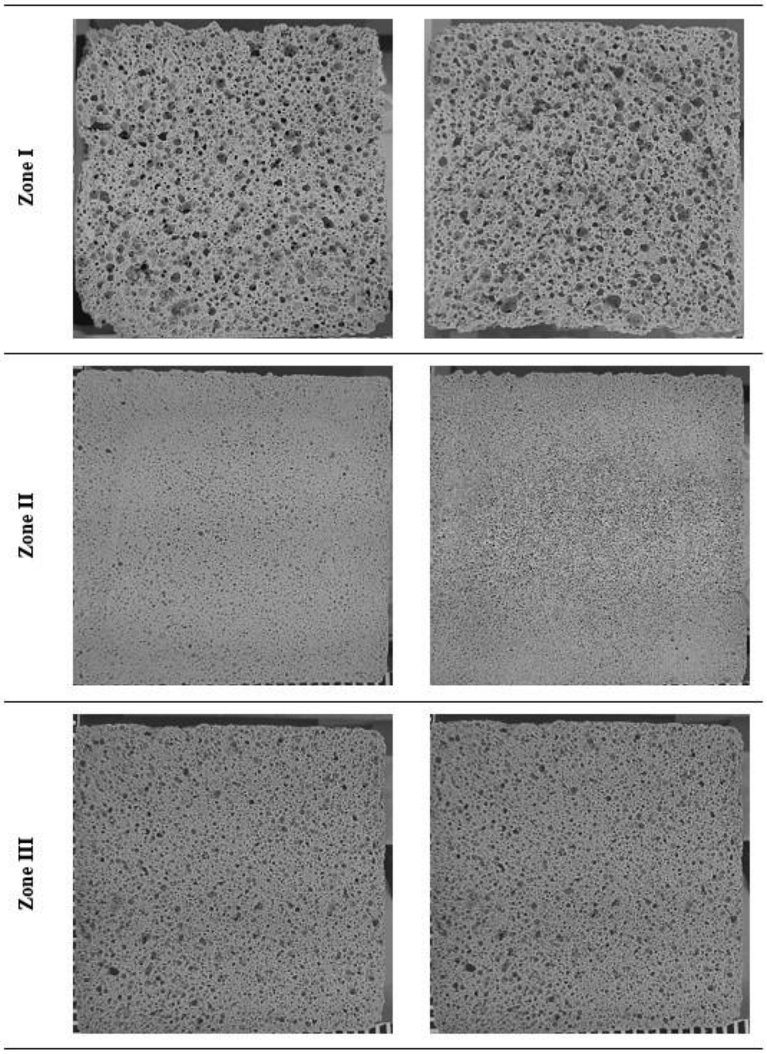


Figure 6. Pore structure related to workability box.

5. Conclusions

Foamed concretes are trending materials. However, information available on the fresh state is limited. Rheological characterization of OPC foamed mixtures made with alternative reagents is presented. A summary of contribution and practical implications:

- Rheological Bingham parameters present the best fit on foamed OPC pastes. Yield stress and plastic viscosity can describe properly fresh state behavior. Oscillation tests show that foamed OPC pastes are viscoelastic materials.
- Time evolution between fresh state - 0 minutes- and 60 minutes after preparation is evaluated. Yield stress significantly increases with time.
- The water-to-cement ratio is the most definitive parameter of rheological behavior. Slight changes in water content cause a significant impact on fresh behavior. Also, the type of reagent can be a critical issue in cellular concrete design when alternative reagents are used.
- For alternative reagents, a workability box was proposed. Three plastic zones are defined by paste consistency. The optimal zone was set in Zone III: Yield stress under 9 Pa and plastic viscosity higher than 0.08 Pa·s. When yield stress values are higher than 9 Pa, cement foams have an uncontrolled morphology. Also, mixtures with viscosities under 0.08 Pa·s have extreme bleeding, and bubbles tend to suffer coalescence. For some industrial applications, Zone II can be used as well.

However, the study faced several important limitations. The first one is the use of an specifical alternative reagents which it is a relevant topic in foamed concrete design.

Future research should expand the understanding of foamed concretes by addressing the limitations identified in this study. In particular, broader investigations involving a wider range of alternative reagents are needed. Additionally, it is required to evaluate the practical applicability of the proposed workability box, especially in real-scale production and placement conditions, where factors such as mixing energy, transport, and environmental exposure may influence foam stability.

6. Statements and declarations

6.1 Competing interests

The authors have no relevant financial or non-financial interests to disclose.

6.2 Acknowledgment

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6.3 Author contributions

All authors contributed to the study conception and design, material preparation, data collection, and analysis. All authors read and approved the final manuscript.

7. Declaration of generative AI and AI-assisted technologies

In preparing this manuscript, AI-assisted technologies such as Grammarly were used to support the structuring of the text and improve clarity, readability, and presentation. All outputs generated with the assistance of these technologies were carefully reviewed and edited by the authors, who take full responsibility for the content of the manuscript.

8. Notes on Contributors

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