

Article

Development and Optimisation of a Standardised Rheological Method for 3D Printing Cementitious Mixtures Using Rotational Rheometry: An Experimental and Statistical Approach

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Abstract

This study presents the development and optimisation of a standardised rheological test method based on rotational rheometry for the characterisation of cementitious mixtures designed for 3D printing. Tests were performed using a Discovery HR-20 rotational rheometer (TA Instruments, New Castle, DE, USA) equipped with a concentric-cylinder cup-and-paddle geometry. A high-early-strength Portland cement (ASTM C1157 Type HE) with a constant water-to-cement ratio (w/c) of 0.35 was employed. The methodological framework comprised five sequential stages: (i) assessment of the pre-conditioning effect; (ii) standardisation of the static shear test; (iii) optimisation of pre-conditioning parameters; (iv) standardisation of the dynamic shear test; and (v) evaluation of the influence of sample volume. Optimal conditions were determined as follows: for pre-conditioning, a shear rate of 50 s^{-1} , holding time (H_t) of 30 s, and rest period of 180 s; for the static shear test, a shear rate range of $0.05\text{--}0.10 \text{ s}^{-1}$ with a H_t of 60 s; for the dynamic shear test, a 30 s ramp up/down, maximum shear rate of 100 s^{-1} , and H_t of 90 s. An optimal sample volume ranging between 150 and 175 mL was established. The proposed method represents a robust and reproducible experimental protocol for evaluating, comparing, and optimising the rheological behaviour of cementitious mixtures using rotational rheometry, providing a reliable tool for the formulation of mixtures tailored to additive manufacturing or 3D printing processes.

Keywords: 3D printing; additive manufacturing; cementitious mixtures; rheological properties; rotational rheometer; rheological test method



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1. Introduction

The study of rheological properties is fundamental to the design and development of cementitious mixtures suitable for 3D printing. Although this construction method is still in its developmental stage, it has achieved remarkable progress since its emergence in the late 20th century [1,2]. Nevertheless, researchers continue to face significant challenges in integrating this technology into the construction industry, due to the stringent rheological and mechanical requirements that printable mixtures must fulfil. These mixtures must exhibit specific flow characteristics at each stage of the printing process. During the initial stages, such as pumping and extrusion, the material must remain sufficiently fluid to

ensure proper transport through the printing system [3,4]. In subsequent stages, such as deposition, the printed layers must develop adequate strength to support both their own weight and that of the successive layers; in other words, the mixture must exhibit sufficient buildability and minimal settlement [5,6]. This behaviour can be enhanced through the use of supplementary cementitious materials (SCMs), chemical admixtures, and rheological modifiers, which optimise the performance of the mixtures at different stages of the process [7,8].

The greatest challenge lies in characterising this behaviour and overcoming the limitations of existing tests or methods used to measure properties in the fresh state, given that traditional procedures applied to pastes, mortars, and concretes, such as the Abrams cone slump test [9], the flow table test [10], or the mini-slump test, are not suitable for characterising cementitious mixtures intended for 3D printing. In this context, rheology has been established as the most appropriate science for characterising the fresh state behaviour of cementitious mixtures designed for 3D printing, as it enables accurate description of the material's responses to external stresses and their evolution over time. Unlike conventional methods that provide qualitative or unrepresentative results under specific conditions, rheology provides fundamental parameters, such as yield stress (static and dynamic), plastic viscosity, and thixotropy, that are decisive in ensuring essential properties such as extrudability, buildability, and dimensional stability during the layer deposition or 3D printing process [11,12].

With regard to the rheological characterisation of mixtures for 3D printing, one of the main challenges is the lack of standardisation in testing methods. This technical limitation has led researchers to adopt different testing parameters and variables, such as shear rates, holding times, rest times, and pre-conditioning conditions, based on their own criteria or experience, resulting in inconsistencies and contradictions among the results reported in the scientific literature. Consequently, it remains difficult to establish standardised rheological properties for cementitious mixtures intended for 3D printing.

Although there are standards such as ASTM C1749 [13], which establish procedures for evaluating the rheological properties of cements and other cementitious materials using rotational rheometry, this standard is not sufficient for assessing the rheological properties of special mixtures, for example, those used in 3D printing. Specifically, it does not define the test variables required for conducting the measurements. Nevertheless, it serves as a valuable starting point for more specialised research, as it provides essential information on equipment specifications and accessories, sample preparation, and testing procedures. It is worth noting that the scientific community has made significant efforts to bridge this gap and achieve a global consensus on the rheological characterisation of cementitious mixtures. In this regard, the work conducted by the RILEM TC 266-MRP Technical Committee is particularly noteworthy. Its report, "Measuring Rheological Properties of Cement-Based Materials" [14], presents a comprehensive and up-to-date guide on the subject. In addition to proposing measurement methods, this report establishes a framework of best practices for test execution, data interpretation, and error minimisation.

The studies and recommendations presented in the report are supported by robust experimental evidence, including inter-laboratory (round-robin) tests reported by Feys et al. (2023) [15]. This research empirically demonstrated the extent of the problem: when the same material was tested in different laboratories, while consistent trends were observed in the measured properties, the absolute values of yield stress and plastic viscosity exhibited considerable dispersion. This confirmed that direct numerical comparison is unreliable and inconclusive without a strictly standardised testing protocol. Similarly, studies by Silvestro et al. [4] and Peng et al. [16] have also compiled and analysed rheological

investigations on cementitious mixtures for 3D printing, identifying key variables and proposing test protocols.

Despite recent advances in the rheological characterisation of cementitious materials for 3D printing, there remains considerable variability in the testing protocols adopted by different research groups. The literature reports numerous experimental configurations including concentric cylinders, parallel plates, and paddle geometries, together with various control modes (stress or strain controlled), as well as significant variations in operating parameters such as shear rate, resting time, shear application time, and loading ramps. This lack of standardisation hinders the comparison of studies, undermines the reproducibility of results, and limits progress toward robust scientific validation of the fresh state behaviour of mixtures for 3D printing. This situation highlights the need to establish a systematic and replicable rheological testing method adapted to the functional requirements of 3D printing with cementitious materials.

In response to this issue, the present study aims to develop and propose a systematic and standardised testing methodology for the rheological characterisation of cementitious mixtures for 3D printing using rotational rheometry. The proposed approach comprises the sequential evaluation of the critical stages of testing: pre-conditioning, static shear, and dynamic shear, together with an analysis of the influence of operational variables such as sample volume. This methodology seeks to optimise measurement parameters, minimise result dispersion, and establish reproducible technical criteria, serving as an experimental guide for researchers and professionals involved in the rheological assessment of such mixtures. Furthermore, the study intends to provide a practical and technically robust framework that enhances the understanding of rheological principles as applied to cementitious mixtures for 3D printing.

2. Reference Framework

2.1. Rheology of Cementitious Pastes

The rheology of cementitious pastes is influenced by several factors, including the mixture composition, testing temperature, relative humidity, testing duration, and, of course, the measurement parameters [17]. Cementitious pastes generally exhibit complex flow behaviour that can be characterised as non-Newtonian, meaning that their viscosity is not constant but varies with the applied shear rate [18,19]. Moreover, they display pseudoplastic behaviour, that is, their viscosity decreases as the shear rate increases [20], which translates into greater ease of pumping or extrusion under shear stress.

This rheological behaviour is particularly advantageous for 3D printing, as the technology requires materials to exhibit viscoelasticity: behaving as elastic solids under low shear stresses and flowing as viscous liquids once the yield stress is exceeded [21,22]. The following section introduces the main parameters that describe the rheological behaviour of cementitious materials used in 3D printing.

- **Yield stress:** Defined as the minimum stress required to initiate or sustain the flow of a material. A distinction is made between the static yield stress (SYS), which corresponds to the stress needed to initiate flow, and the dynamic yield stress (DYS), which is required to maintain continuous flow [23–25].
- **Plastic viscosity:** Represents the resistance of a material to flow once motion has begun and is defined as the additional stress required to increase the shear rate [4,21].
- **Thixotropy:** Refers to a reversible rheological behaviour in which, upon removal of external shear stress, the material ceases to flow, and structural rebuilding occurs, thereby restoring its static yield stress [26,27]. This behaviour is typical of colloidal suspensions such as the water–cement system in its fresh state.

- Shear thickening and thinning: Depending on the shear rate, a material may exhibit shear thickening (an increase in viscosity with increasing shear rate) or shear thinning (a decrease in viscosity, also referred to as pseudoplasticity) [17,28]. In general, the fresh water–cement system exhibits shear thinning, which is known as pseudoplastic behaviour.

The flow behaviour of cementitious materials is commonly described by the Bingham model (Equation (1)), which establishes a linear relationship similar to Newton’s law but incorporates an initial shear stress, known as the static yield stress (τ_0 or SYS).

$$\tau = \tau_0 + \eta \dot{\gamma} \quad (1)$$

where: τ is the shear stress, τ_0 is the static yield stress, η is the plastic viscosity, and $\dot{\gamma}$ is the shear rate.

At present, more advanced constitutive models, such as the Herschel–Bulkley and modified Bingham models, are used to study the rheology of cementitious mixtures [16,29]. These models account for shear-thickening and shear-thinning phenomena and differs from the Bingham model in that, beyond the yield point, the relationship between shear stress and shear rate becomes non-linear (Figure 1) [17]. Furthermore, cementitious mixtures exhibit thixotropic characteristics (Figure 2), meaning that, under shear, the internal structure breaks down and particles become separated; conversely, in the absence of shear, the particles tend to reorganise (flocculation), leading to the recovery of rheological properties [27,30]. The degree of this recovery, or structural rebuilding, indicates the extent of thixotropic in a cementitious mixture. This thixotropic behaviour is crucial, as it directly affects the material’s ability to be extruded and deposited layer by layer during the 3D printing process.

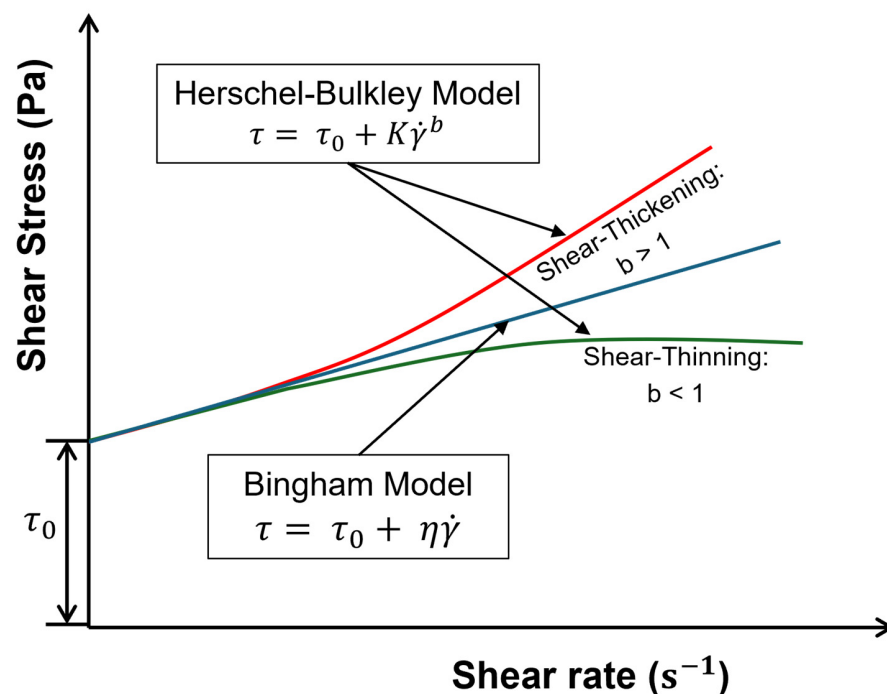


Figure 1. Comparison between the Bingham and Herschel–Bulkley models.

In summary, the rheology of a cementitious mixture plays a decisive role in the printing process, as it governs how the mixture behaves at different stages: before, during, and after printing. Each stage of the process requires specific rheological characteristics, such as those needed for storage, pumping, and extrusion of the printed layers [11,31,32].

The complexity of this behaviour is evident: a mixture with high flowability can facilitate pumping or extrusion, yet excessive fluidity may impair the buildability of the printed structure, creating a conflict between the rheological requirements of each process stage [2]. Conversely, the progressive increase in rheological properties resulting from cement hydration reactions can render the material excessively stiff for extrusion. Nevertheless, this structural build-up can also enhance the strength on the initial layers, enabling them to support the deposition of subsequent layers and thereby improving buildability [30,33].

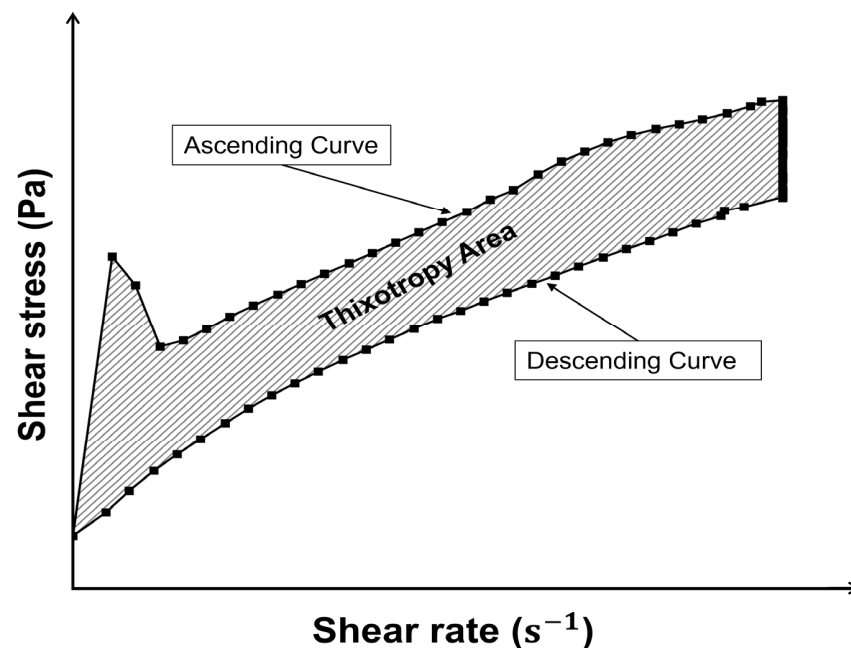


Figure 2. Typical hysteresis curve of a thixotropic material.

A comprehensive understanding of the rheological properties of cementitious mixtures requires not only a conceptual analysis of their behaviour under external stresses but also the application of appropriate measurement techniques capable of quantifying these phenomena with precision and reproducibility. Accordingly, it is essential to review the experimental methods and testing procedures commonly employed for this purpose.

2.2. Measurement and Testing Methods

Among the various techniques employed to determine the rheological properties of cementitious mixtures designed for 3D printing, rotational rheometry is particularly prominent. This method enables the establishment of the relationship between shear stress, shear rate, and viscosity through flow curves [34,35]. These curves are fitted to rheological models such as the Bingham and Herschel–Bulkley models.

Rotational rheometers employ various geometries, such as concentric cylinders, parallel plates, and cone-and-plate systems, to subject the material to controlled stress or deformation while simultaneously measuring its response [34,36,37]. Two operational modes can be distinguished: (i) stress-controlled mode, in which a specific stress is applied and the resulting deformation is measured; (ii) strain-controlled mode, in which a constant shear rate is imposed, and the corresponding stress is recorded [38].

Figure 3 illustrates a general schematic of the rheological testing procedure, in which three stages are defined based on the variation of the applied shear rate as a function of the test time. In stage 1, pre-conditioning is performed to homogenise the mixture and eliminate the air entrapped during its pouring into the test cup. This stage comprises two sub-stages: (i) pre-shearing, during which the material is subjected to the maximum test

shear rate for a specified time; (ii) the rest time, defined as the time interval in which the material remains static prior to measurement; equivalent to the duration required for the mixture to recover its rheological properties following pre-shearing [11,21,39].

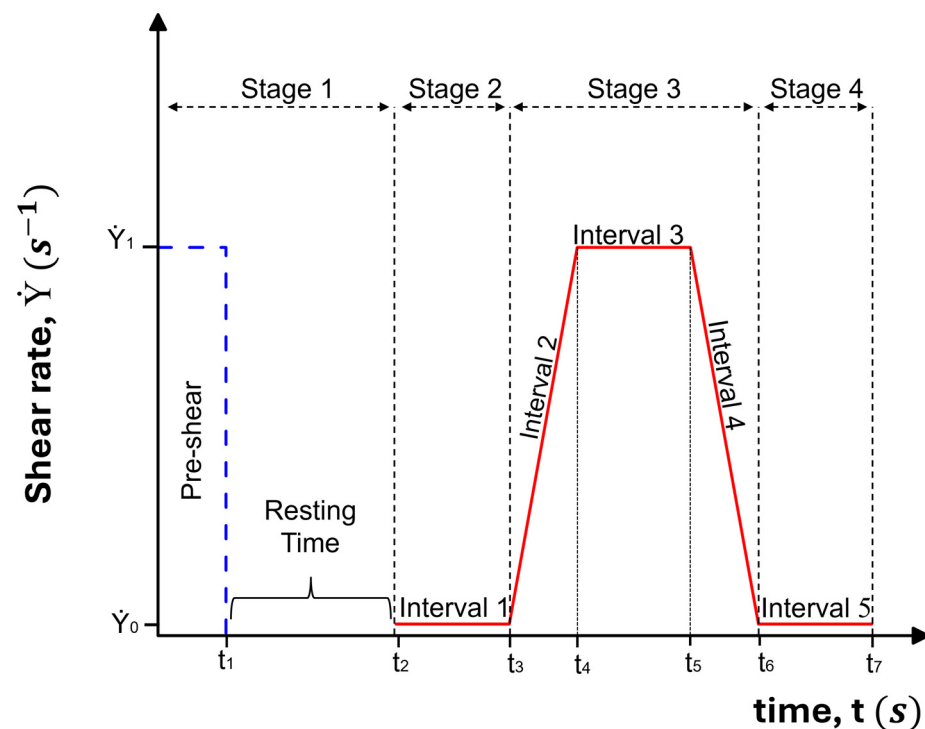


Figure 3. General scheme of the test method with its respective stages and study variables.

Stage 2 corresponds to the static shear test, represented in the diagram as Interval 1. In this interval, the applied shear rate is extremely low (approaching rest) and maintained constant for a defined holding time (H_t) to determine the SYS. Figure 4a illustrates a shear stress vs. time curve obtained during this stage in the experimental test, where the peak shear stress characteristic of the SYS is observed, followed by a subsequent stress reduction indicating the onset of flow [34,40,41]. These curves are similar to those reported by other authors [34]. Occasionally, the material response in this interval deviates from the expected behaviour, making it difficult to identify a distinct flow peak, as shown in Figure 4b. This effect may be associated with an inadequate selection of the shear rate in this interval, an aspect further investigated in the present study.

In stage 3 (Figure 3), dynamic conditions are applied to the mixture, beginning with an ascending [34] shear-rate ramp (Interval 2) that progressively increases until reaching a maximum value. Once this maximum shear rate is attained, it is maintained for a defined H_t (Interval 3). Interval 4 then involves a descending shear-rate ramp under the same conditions as the ascending ramp (Interval 2). The consecutive application of Intervals 2, 3, and 4 results in the formation of the hysteresis loop and the associated thixotropic area illustrated in Figure 2. Intervals 2 and 4 correspond to the ascending and descending flow curves, respectively, to which the rheological models described above can be applied to obtain the hysteresis cycle. Stage 4 corresponds exclusively to Interval 5, in which static conditions similar to those of Interval 1 are reproduced, with the aim of comparing rheological properties, such as SYS and viscosity, before and after the material is subjected to high shear stresses (structural breakdown).

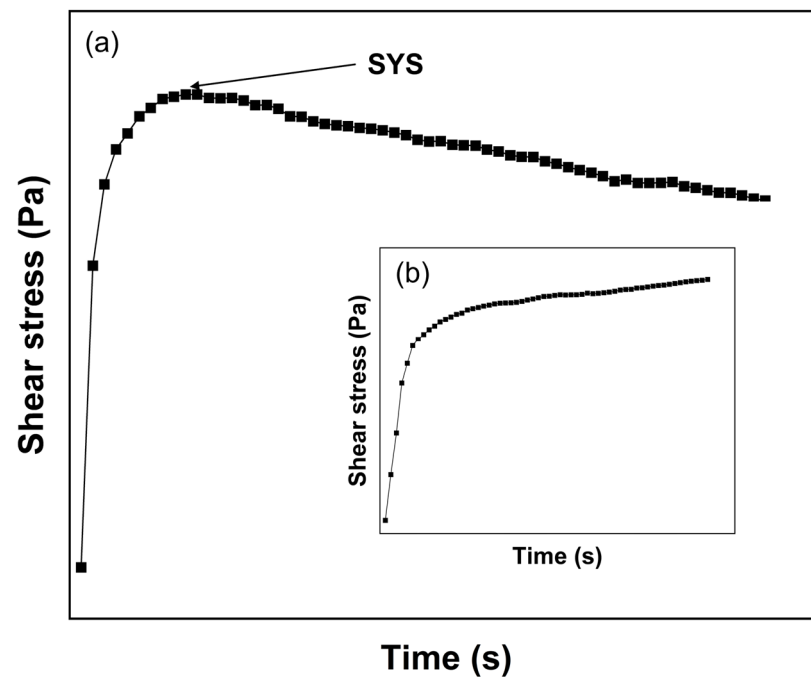


Figure 4. Static shear test (Interval 1): (a) Shear stress–holding time curve showing expected behaviour; (b) Shear stress–holding time curve showing non-ideal behaviour.

It should be noted that the shear history during extrusion 3D printing is highly dependent on the delivery system (pump/screw), nozzle, flow rate, and residence times; therefore, the proposed rheological protocol does not aim to exactly replicate a particular printing setup. In this context, Interval 5 should be interpreted as a controlled analog for evaluating the structural reconstruction of the material after imposed destruction (post-shear), quantifying the change in static yield strength (SYS) and viscosity before and after a high-shear event. This interval is particularly useful for comparing formulations and conditions when the printing process is dominated by a shear stage followed by deposition and rapid stiffness recovery. However, its representativeness may decrease in systems with multiple shear events, recirculation, or extended residence times, where the actual history differs from the simplified scenario; in such cases, Interval 5 should be considered a comparative indicator of reconstruction and not a direct reproduction of the 3D printing process.

3. Materials and Methods

3.1. Raw Materials

A high-early-strength Portland cement (HE), classified according to ASTM C1157 [42], was used in this study. Its particle size distribution was obtained by laser granulometry using a Mastersizer 2000 instrument (Malvern Instruments equipment, Malvern, UK) (Figure 5). The percentiles were: D10: 2.70 μm ; D50: 12.19 μm ; and D90: 32.80 μm . The volume-weighted mean diameter D(4,3) was of 15.32 μm .

The chemical composition, determined by X-ray fluorescence using a MagiX-Pro PW-2440 spectrometer (Phillips PANalytical, Tollerton, UK), is presented in Table 1. The density, measured by helium pycnometry using an Ultrapyc 3000 device, (Anton Paar, Graz, Austria) was 3.07 g/cm^3 .

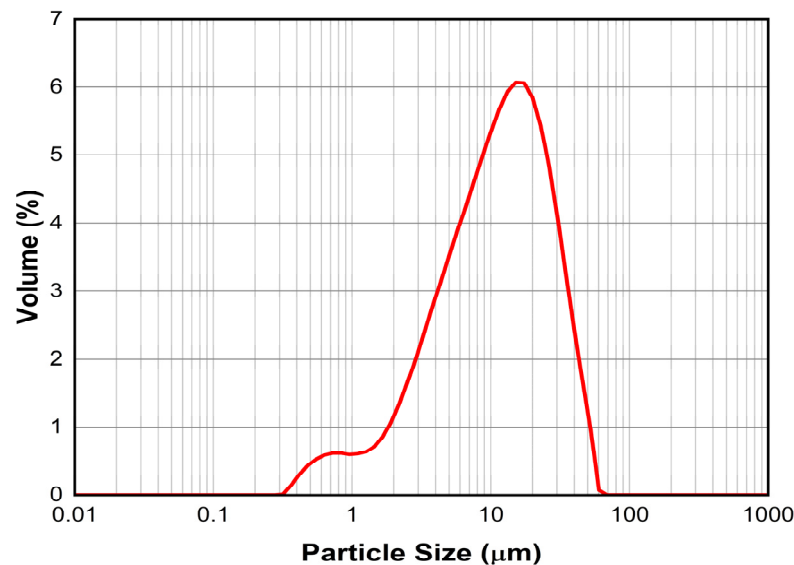


Figure 5. Particle size distribution of Portland cement (HE) used.

Table 1. Chemical composition (XRF) of Portland cement (HE) (%wt).

SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	TiO ₂	SO ₃	LOI
19.13	3.57	3.99	57.92	0.97	0.14	1.11	0.30	5.54	6.67

3.2. Development of the Rheological Method

The reference mixture, consisting of HE cement and water with a water/cement (w/c) ratio of 0.35, was used to develop the rheological testing method. The experiments were conducted using a Discovery HR-20 rotational rheometer (TA Instruments, New Castle, DE, USA) equipped with a construction material testing kit. This kit includes a cup-type fixture with an internal diameter of 57.5 mm and a height of 75.5 mm, together with a paddle-shaped geometry measuring 44.42 mm in maximum width and 30 mm in length (Figure 6). The experimental procedure was developed through the standardisation of test conditions across its different stages, encompassing the variables and parameters listed in Table 2.

Table 2. Rheological parameters and variables studied.

Parameters and Variables	Symbol	Units of Measurement
Shear rate	$\dot{\gamma}$	s ⁻¹
Shear stress	τ	Pa
Rest time	R _t	S
Holding time	H _t	S

To avoid fluctuations in the results and minimise uncertainty in the experiments, the procedures followed were standardised, including the mixing protocol, sample preparation and conditioning, method of depositing the sample into the rheometer cup, and compaction method. In this context, the sources of variability are mainly associated with variations in environmental conditions, especially temperature and relative humidity during the mixing and handling of the sample. However, in general, the ambient temperature in the laboratory was kept within a narrow range (23–25 °C), reducing its influence. By keeping the preparation and testing times constant, the dispersion of the experimental results was reduced.

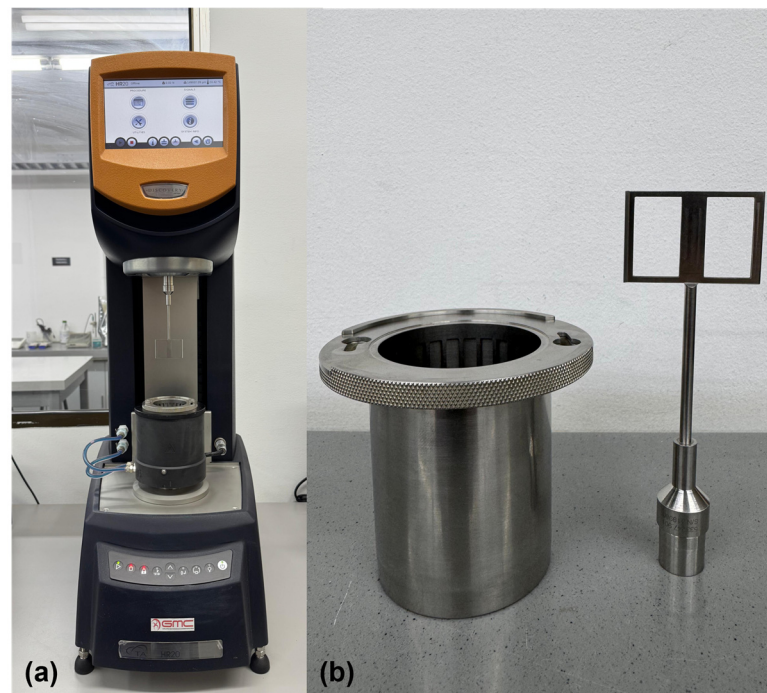


Figure 6. Rheological testing equipment: (a) Discovery HR-20 rotational rheometer (TA Instruments, New Castle, DE, USA); (b) cup-and-paddle geometry based on the concentric cylinder configuration.

3.2.1. Evaluation of the Effect of the Pre-Conditioning Stage

The influence and significance of the pre-conditioning stage, which includes the sub-stages of pre-shearing and resting time, were validated. For this purpose, the test parameters were standardised based on the findings of previous studies and relevant literature [4,7,23,40]. Pre-shearing was conducted at a shear rate of 50 s^{-1} with a pre-shear H_t of 30 s, followed by a R_t of 90 s. The effect of pre-conditioning was assessed through the static shear test, specifically by analysing its influence on the behaviour of the shear stress vs. time curve obtained in Interval 1. To ensure consistency, the test variables corresponding to this interval were also standardised, applying a constant shear rate of 0.05 s^{-1} and H_t of 300 s to provide a broader evaluation range.

3.2.2. Standardisation of the Static Shear Test

This stage involved evaluating the effect of the applied shear rate in Interval 1, corresponding to the static shear test. The shear rate was varied at 0.01, 0.02, 0.03, 0.04, 0.05, 0.1, and 1 s^{-1} , and its influence on the shear stress–time curve and on the SYS values was analysed. The H_t for Interval 1 was kept constant at 180 s, since previous studies have shown that the maximum stress and creep of the material typically occur within the first few seconds of the test [34,40,43].

3.2.3. Optimisation of Pre-Conditioning

As discussed previously, the pre-conditioning stage comprises two sub-stages: (i) pre-shearing and (ii) resting time. After assessing its impact and confirming the necessity of its implementation, it was essential to analyse and standardise the variables and parameters involved in this stage. Accordingly, the study first examined the variables associated with the pre-shearing phase and subsequently evaluated the effect of the resting time, as detailed below:

To evaluate the effect of variations in pre-shearing parameters on the results of the static shear test (Interval 1), a completely randomised experimental design with a 2×4 factorial structure was implemented using Minitab 19 statistical software. Two factors

(shear rate (s^{-1}) and H_t (s)) and one response variable (static shear) were defined (Table 3). Eight experimental treatments were generated from combinations of the factors and their respective levels. Two independent repetitions were performed for each treatment, resulting in a total of 16 experimental units.

Table 3. Factors and levels considered in the evaluation of pre-shearing parameters.

Factors	Symbol	Type	Levels	Values
Shear rate (s^{-1})	$\dot{\gamma}$	Fixed	2	50; 100
Holding time (s)	H_t	Fixed	4	30; 60; 90; 180

To assess the effect of the resting time during the pre-conditioning stage, static shear tests (Interval 1) were conducted after 60, 90, 180, and 300 s. The objective was to determine the influence of the resting time on the SYS and to identify the optimal resting time for the proposed rheological testing method.

Data processing was performed using an analysis of variance (ANOVA) to determine the effect of the main factors and their interactions, considering a significance level of 0.05. The assumptions of normality and homogeneity of variances were assessed using the Shapiro–Wilk and Levene tests, respectively. The inclusion or exclusion of outliers was assessed according to technical criteria.

3.2.4. Standardisation of the Dynamic Shear Test

For the standardisation of the dynamic shear test, given the number of parameters involved, a statistical analysis was again conducted. A completely randomised experimental design with a $2^2 \times 3$ factorial structure was proposed to analyse the rheological variables: viscosity, DYS, thixotropic area (ATIX), and percentage of viscosity recovery (% recovery). The factors and levels evaluated are summarised in Table 4. A total of 12 experimental combinations were generated, and each test was performed in duplicate.

Table 4. Factors, levels, and treatments considered in the dynamic shear test.

Factors	Levels	Treatments
Ramp (s)	30; 60	(R30; S50; H_t 30)–(R30; S50; H_t 60)–(R30; S50; H_t 90)–(R30; S100; H_t 30)–(R30; S100; H_t 60)–(R30; S100; H_t 90)–(R60; S50; H_t 30)–(R60; S50; H_t 60)–(R60; S50; H_t 90)–(R60; S100; H_t 30)–(R60; S100; H_t 60)–(R60; S100; H_t 90)
Shear rate (s^{-1})	50; 100	
Holding time (s)	30; 60; 90	

R: Ramp; S: Shear rate; H_t : Holding time.

The response variables, viscosity and DYS, were obtained by fitting the Bingham and Herschel–Bulkley rheological regression models to the descending flow curve (Interval 4 of the method). In contrast, the ATIX and % viscosity recovery were determined by graphical analysis. The ATIX value was calculated as the area enclosed between the ascending and descending curves of the hysteresis loop and is inherently dependent on the applied shear rate. Considering that two different shear rates (50 and 100 s^{-1}) were analysed during this phase, ATIX was normalised by dividing it by the maximum shear rate used in each case. The % viscosity recovery was calculated using Equation (2).

$$\% \text{ viscosity recovery} = \frac{\text{Final viscosity (Interval 5)}}{\text{Final viscosity (Interval 1)}} * 100 \quad (2)$$

3.2.5. Evaluation of the Effect of Sample Parameters (Mixture Volume)

After establishing all the variables and test parameters of the rheological method, it was necessary to assess the influence of the sample volume (mixture) on the rheology results. To this end, the complete test procedure, including the pre-conditioning, static shear, and dynamic shear stages, was carried out using five different sample volumes: 100, 125, 150, 175, and 200 mL, corresponding to 50, 62.5, 75, 87.5, and 100% of the total capacity of the rheometer cup, respectively. The maximum capacity of the rheometer cup is 200 mL. It should be noted that the height of the paddle (geometry) is 30 mm, with a gap of 1000 μm between its lower edge and the bottom of the cup. In other words, the minimum sample volume required to ensure that the paddle is fully immersed is approximately 100 mL. Figure 7 presents a cross-sectional schematic of the test cup showing the respective volumes evaluated.

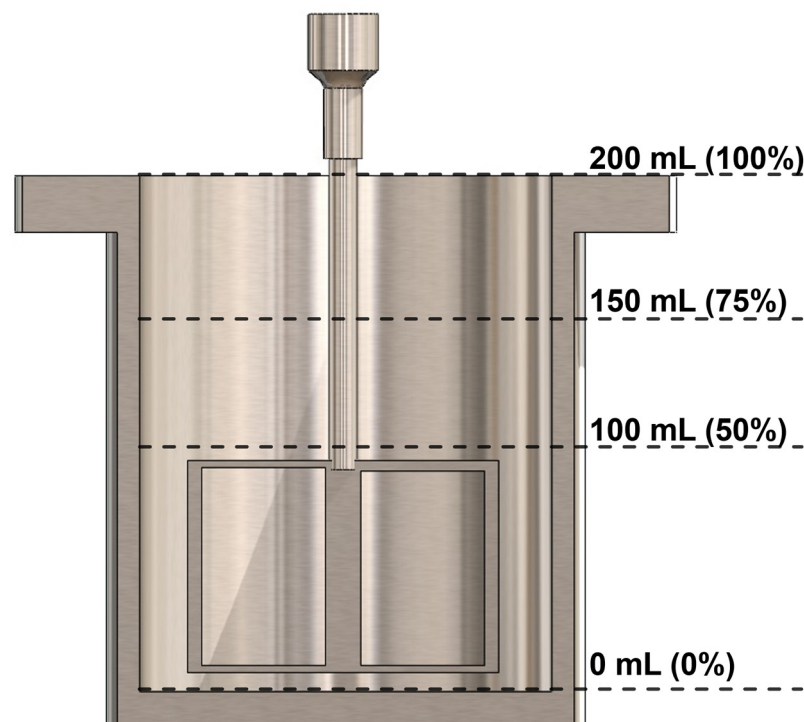


Figure 7. Cross-sectional schematic of the cup-and-paddle geometry showing the sample volumes evaluated.

4. Results and Discussion

4.1. Evaluation of the Effect of Pre-Conditioning

Figure 8 presents the results of the static shear test (Interval 1), performed with and without pre-conditioning. A clear difference can be observed between the curves obtained with pre-conditioning (blue) and without pre-conditioning (red). When this stage is omitted, the stress values recorded throughout the test are higher; the SYS increases from 498.6 Pa to 661.4 Pa, corresponding to an increase of approximately 34%. Conversely, when pre-conditioning is applied, the rate of increase in shear stress over time decreases, and the maximum stress peak (SYS) becomes more clearly defined. This behaviour indicates that the material reaches its maximum stress more rapidly within the elastic region and subsequently behaves as a viscous fluid [21], exhibiting lower stress values.

According to several studies [44,45], the behaviour described above results from the fact that pre-shearing applies sufficient stress to disrupt the initial particle network formed during the early stages of cement hydration. Another relevant phenomenon is flocculation, which occurs within the first seconds or minutes after water comes into contact with cement

and is characteristic of a colloidal system, where particles agglomerate to form flocs. The pre-shearing stage disperses these flocs and temporarily reduces inter-particle interactions, meaning that the material requires a recovery period before regaining a stable state. Similarly, homogenisation helps to prevent adverse effects arising from colloidal interactions between cement particles [46], as well as particle migration, precipitation, and the presence of entrapped air introduced during sample placement [34]. In agreement, Campos et al. [39] reported that pre-shearing prior to rotational rheometry disrupts particle interactions and breaks the internal structure of the cementitious material immediately before measurement, thereby ensuring a consistent shear history. Likewise, Azevedo et al. [47] observed that pre-shearing promotes a uniform state in the samples before performing rheological measurements. These observations, together with the results obtained in this study, highlight the importance and necessity of the pre-conditioning stage, as it enhances the accuracy of static creep stress determination in Interval 1 and yields curve behaviour consistent with theoretical expectations.

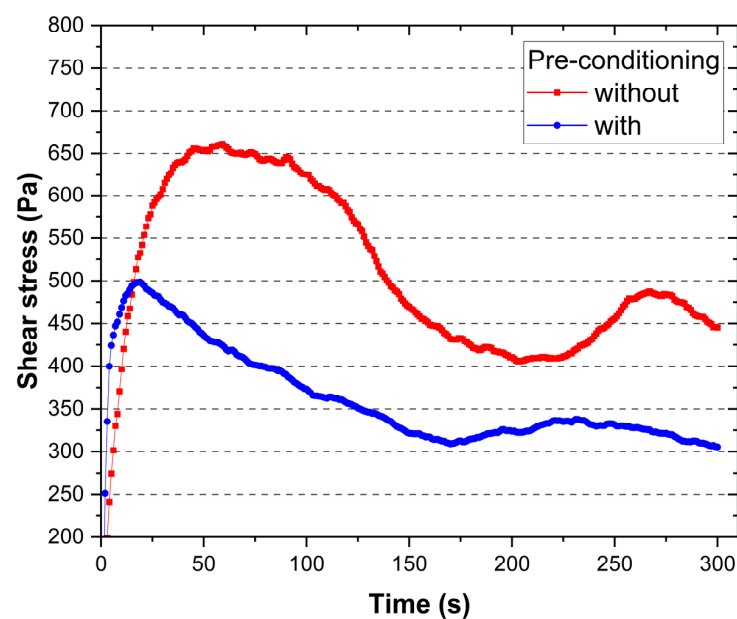


Figure 8. Static shear test: comparison between tests with and without pre-conditioning (Interval 1).

4.2. Static Shear Test

The results of the static shear test (Interval 1) are presented in Figure 9. Figure 9a displays the shear stress–time curves obtained at low shear rates ranging from 0.01 and 1 s^{-1} , while Figure 9b illustrates the variation in SYS and the corresponding time to reach the maximum stress peak at the same range of shear rates.

As shown in Figure 9a, the shear rate strongly influences both the peak stress values and the overall shape of the curves. At very low shear rates, close to 0.01 s^{-1} , i.e., under near-rest conditions, the curves display behaviour that is unsuitable for this test. Although the mixture subjected to 0.01 s^{-1} exhibits a maximum stress peak, the corresponding static yield stress (SYS) is not clearly defined, and the material continues to develop shear stress without an appreciable decrease, suggesting an interval of restructuring of the material and its inability to reach a true flow condition [4]. Many static creep stress procedures recommend using the lowest operational shear rate of the rheometer; in this study, that value corresponds to 0.01 s^{-1} . However, the results reported in Figure 9a indicate that the most appropriate shear rate for a static creep does not necessarily coincide with the minimum instrument capability. Instead, it is advisable to evaluate a range of low shear rates close to the minimum, particularly when the rheometer can operate at values as

low as 0.01 s^{-1} (almost at rest). According to Rouseel et al. [46], the progressive increase in static creep stress can be attributed to the flocculation of cement particles driven by attractive colloidal forces and to the nucleation of hydration products at particle contact points. Nucleation occurs even during the dormant period of hydration, transforming weak colloidal attractions into more rigid particle interactions that cannot be disrupted under low shear stress.

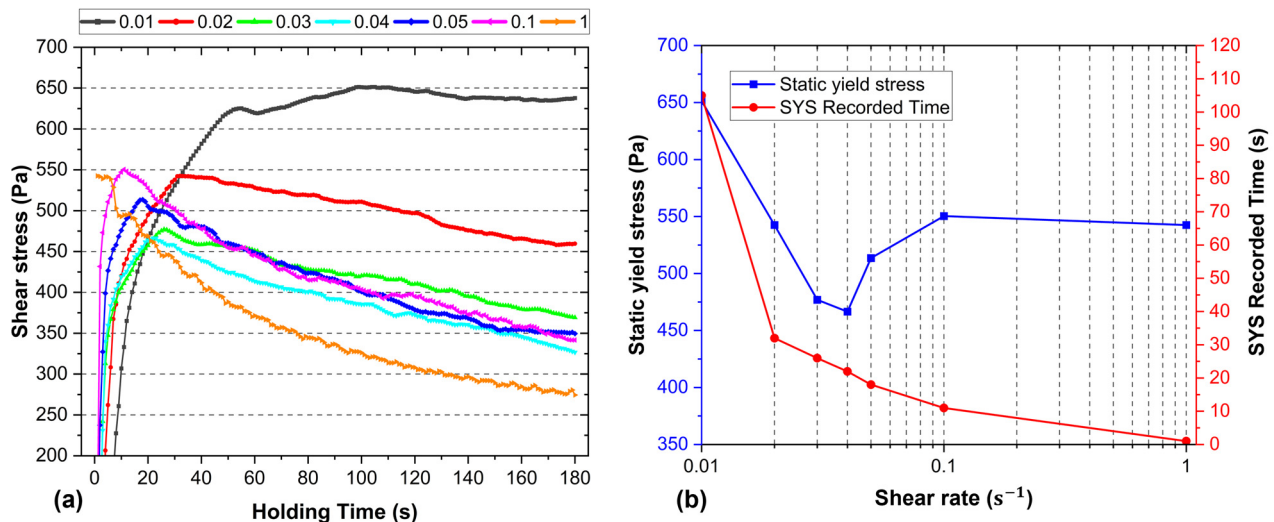


Figure 9. Static shear test (Interval 1): (a) shear stress vs. hold time curves at different shear rates (0.01 to 1 s^{-1}); (b) Relationship between static yield strength (SYS) and the corresponding time to reach SYS within the same shear rate range (0.01 to 1 s^{-1}).

In general, as the shear rate increases, the material exhibits a greater tendency to flow, evidenced by the progressive reduction in shear stress after the SYS is reached. From a shear rate of 0.05 s^{-1} (dark blue line), this behaviour is more evident, with a clearly defined maximum stress peak (SYS) approaching the expected theoretical behaviour. The same trend is observed as the shear rate further increases, up to 0.1 s^{-1} . However, at a very high shear rate (1 s^{-1}), the rheometer fails to capture the initial stress response, preventing identification of a distinct maximum peak; instead, the material begins to flow immediately from the onset of measurement, without exhibiting an elastic region.

Similarly, Figure 9b presents the relationship between static yield stress (SYS) and shear rate; however, the trend is not clearly defined. As the shear rate increases from 0.01 to 0.04 s^{-1} , a decrease in static yield stress is observed, whereas, at higher rates ($\dot{\gamma} \geq 0.05 \text{ s}^{-1}$), the static yield stress values rise again. In contrast, a consistent inverse relationship is evident between the time to reach the peak stress (SYS) and the shear rate: at higher shear rates, the time required to attain the maximum stress peak (SYS) decreases markedly. At very high shear rates (1 s^{-1}), the SYS occurs almost instantaneously, at near-zero time.

These results are consistent with the findings of Nan et al. [38], who reported that high shear rates cause rapid disruption of the mixture structure during static creep stress testing. At elevated shear rates, the material also exerts greater resistance on the rheometer rotor, resulting in higher measured shear stress. Similarly, Yuan et al. [48] demonstrated that, at lower shear rates, a longer time is required to achieve the same level of deformation as that reached under higher shear rates.

Based on these observations, an optimal shear rate range between 0.05 and 0.1 s^{-1} was established for the static shear test. For the subsequent phase, the shear rate was standardised at the lower limit of this range, i.e., 0.05 s^{-1} . This decision was further supported by previous studies [7,34,49], which emphasise the importance of maintaining sufficiently static conditions to accurately determine the elastic limit of cementitious materials, con-

ditions that can only be ensured at very low shear rates. Moreover, the holding time for H_t -Interval 1 was reduced to 60 s, since, as shown in Figure 9b, material creep within the defined range occurs before 30 s. Hence, an H_t of 60 s was deemed adequate to reach the SYS (maximum peak) and to observe the onset of flow (curve descent) once SYS had been attained.

4.3. Pre-Conditioning Optimisation

4.3.1. Effect of Pre-Shearing on the Static Shear Test

Table 5 presents the results of the ANOVA and the corresponding statistical parameters, showing a model fit (R^2) of 92.19%. Both factors evaluated, shear rate and pre-shearing holding time, were found to be significant at the 10% significance level, indicating that each factor exerts a measurable influence on the response variable, namely the SYS. Furthermore, significant differences were identified between the shear-rate levels, while no interaction effects between the two factors were observed. Consequently, a post-ANOVA comparison was carried out to analyse the differences among the levels of the “holding time” factor.

Table 5. Results of ANOVA, analysis of variance. $R^2 = 92.19\%$.

Fuente	Degrees of Freedom (df)	Sum of Squares (SC)	F-Statistic (F)	p-Value (p)	Result
Shear rate	1	3474.2	4.91	0.057	Significant
Holding time	3	62,409.3	29.42	0.000	Significant
Shear rate * Holding time	3	867.3	0.41	0.751	No significant
Error	8	5656.8			
Total	15	72,407.6			

Table 6 presents the results of the post-ANOVA analysis using Fisher’s LSD test. At a 95% confidence level, the H_t factor shows statistically significant differences among the pre-shear durations. Three distinct groups were identified: the 30 s pre-shear time (Group A) differs significantly from all others; 60 s and 90 s form a statistically equivalent group (Group B); and 90 s and 180 s also belong to a common group (Group C). Furthermore, it can be observed that, as both factors, shear rate and pre-shear holding time, increase, the SYS values tend to decrease.

Table 6. Post-ANOVA analysis using Fisher’s LSD test.

Holding Time (s)	Number of Repetitions	Mean (Pa)	Group
30	4	564.7	A
60	4	463.8	B
90	4	431.8	B
180	4	397.9	C

These results are consistent with the experimental findings. Figure 10 shows the shear stress vs. hold time curves (Interval 1) for all treatments, corresponding to the static shear stage. As both the shear rate and pre-shear holding time increase, the stress values recorded in the static shear test decrease, as does the peak shear stress associated with the SYS.

Although the main objective of this phase was to assess the influence of pre-shearing on the static shear response, it is important to note that, in practice, all stages of the rheological method are performed consecutively within a single experiment. Specifically, the following stages are conducted sequentially: (1) pre-conditioning, (2) static shear, and (3) dynamic shear. This sequential procedure enables evaluation of the pre-shearing effect

on subsequent stages of the method. Accordingly, based on previous research [7], the following preliminary parameters were adopted for the dynamic shear stage: (i) ramp-up and ramp-down periods of 30 s, with one measurement per second; (ii) a maximum shear rate of 50 s^{-1} ; and (iii) an H_t -Interval 3 at the maximum shear rate of 60 s.

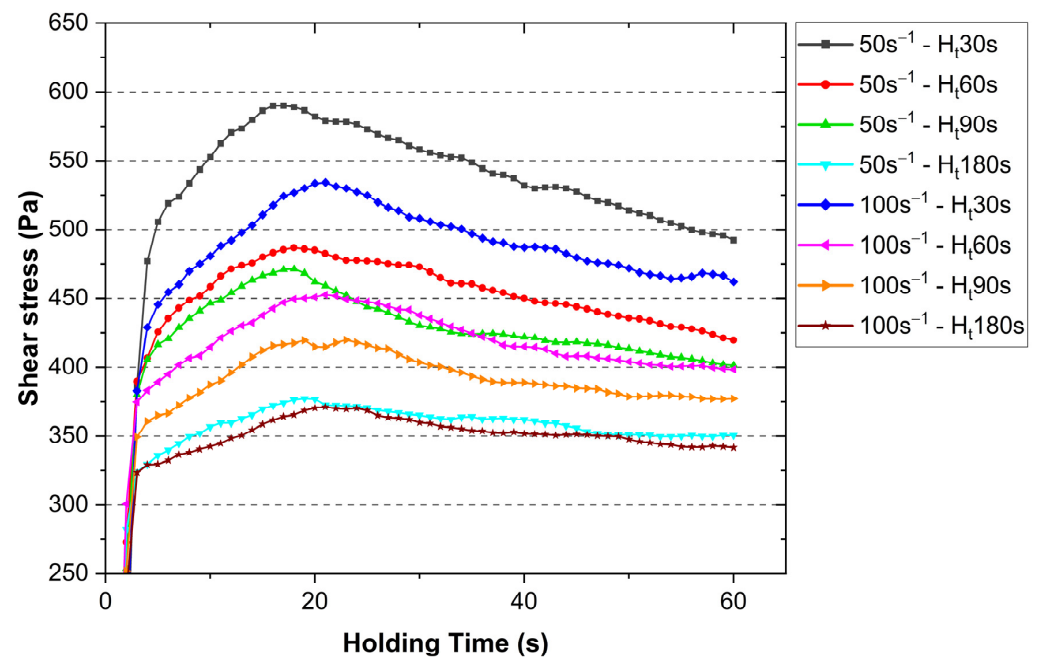


Figure 10. Effect of pre-shearing conditions (shear rate and holding time (H_t)) during the static shear stage.

In this context, the analysis of the flow curves (Figure 11) corresponding to stage 3 of the test method, where the dynamic shear stage is performed, shows that the pre-shearing variables exert a significant influence on this phase. In particular, only two combinations of pre-shearing conditions (50 s^{-1} for 30 s and 100 s^{-1} for 30 s) were found to ensure two fundamental conditions: (i) preservation of thixotropic area and (ii) maintenance of the descending flow curve below the ascending curve.

These conditions are particularly relevant because, according to Bayat and Kashani [11], the thixotropy of a cementitious mixture can be represented by the hysteresis loop obtained from the flow curves, which illustrates how the shear stress varies as a function of shear rate and time variations. Furthermore, Chen et al. [22] noted that, in general, higher shear stresses are recorded on the ascending curve than on the descending one. This occurs because, during the upward phase, it is necessary to break the structure formed during the hydration process, overcoming the colloidal and electrostatic attractive forces associated with cement particle flocculation, thereby increasing the apparent strength of the mixture [50,51]. In addition, the area enclosed by the hysteresis loop represents the total energy dissipated during changes in shear stress and viscosity and is widely used as an indicator of material thixotropy. The larger this area, the greater the thixotropy and the higher the energy required to modify the viscosity [52].

Based on both the statistical and experimental results obtained in this phase of the study, the optimal pre-shearing holding time was determined to be 30 s, as this value was statistically distinct from the others and ensures compliance with the conditions defined for the hysteresis cycle. Regarding the shear rate during pre-shearing, it is recommended to employ the rate that minimises the influence on the SYS while maximising the thixotropic area during the dynamic shear stage, the optimal value was found to be 50 s^{-1} .

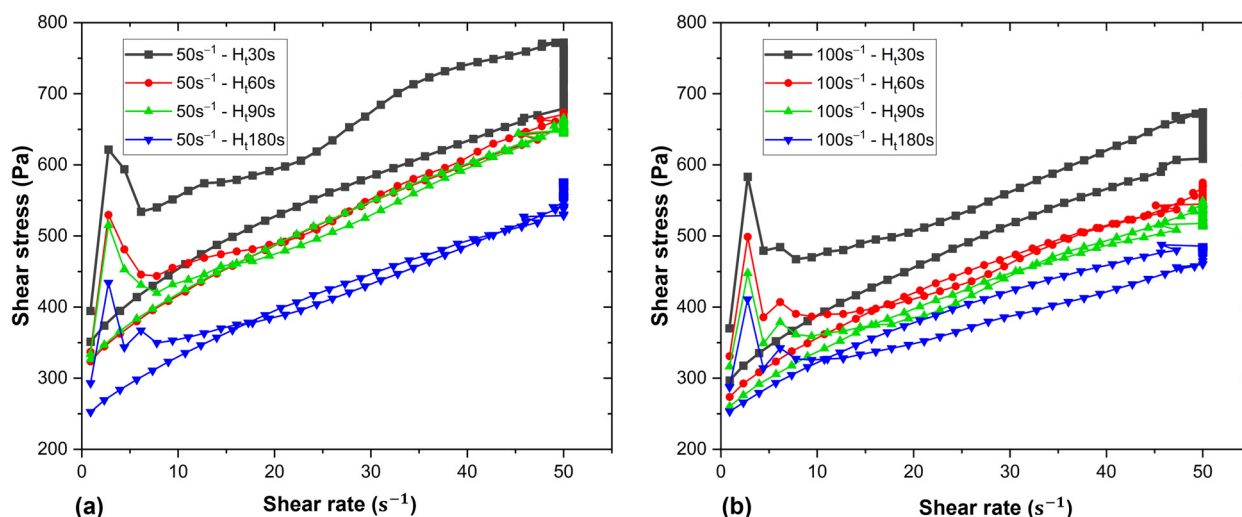


Figure 11. Effect of pre-shearing conditions on the hysteresis curves obtained during the dynamic shear stage. The curves correspond to different combinations of shear rate and holding time (H_t) during the pre-shearing stage: (a) $50 s^{-1}$; (b) $100 s^{-1}$.

4.3.2. Influence of Rest Time (R_t) on the Static Shear Test

Figure 12 shows the results of the static shear test, where R_t was varied at 60, 90, 180, and 300 s. In general, it can be observed that the resting time does not affect the ideal behaviour of the curve, since in all cases a maximum peak is observed before the stress values decrease (ideal behaviour) (Figure 12a). However, the maximum stress value (SYS) increases proportionally to the resting time (Figure 12b). This behaviour is consistent, as several researchers [52–54] have reported that longer rest times promote structural recovery, associated with the flocculation phenomenon of cement particles in suspension that were dispersed during the pre-shearing stage.

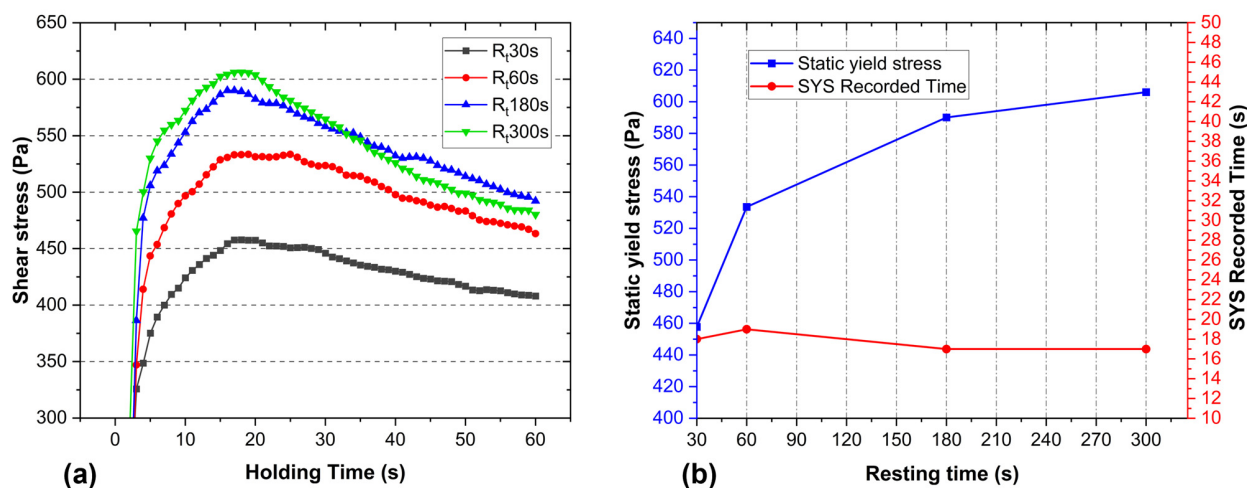


Figure 12. Effect of resting time (R_t) on the static shear test: (a) shear stress vs. holding time curves (Interval 1); (b) static yield stress (SYS) values and the time at which SYS is reached.

According to Lee et al. [52], as the rest time increases, a notable increase in the initial shear stress is observed, whereas the equilibrium state exhibits only minor variations in shear stress. This behaviour occurs because a longer R_t promotes the formation of interparticle “links” between cement grains, thereby increasing the energy stored in the colloidal suspension. Consequently, the initial shear stress tends to increase proportionally with resting time. Likewise, other researchers such as Omran et al. [53] and Lootens et al. [54] have reported

that, after mixing, rapid physical flocculation of cement particles takes place due to attractive interparticle forces. The average number of contacts between cement particles thus increases rapidly from a very low initial value, resulting in a pronounced rise in SYS.

Another noteworthy observation is that the resting time does not significantly affect the time at which the SYS is reached, which remains approximately constant at around 18 s. Based on these results, an optimal resting time of 180 s was established for defining the ideal rheological test method. This choice is further justified by the need to align the rheological test method with the 3D printing process, where the material typically remains under quasi-static conditions for a period of 2 to 3 min before the extrusion process begins. Accordingly, a R_t was selected that minimises its influence on the SYS measurement while remaining consistent with the time intervals characteristic of most 3D printing operations.

4.4. Dynamic Shear Test

Figure 13 shows the flow curves corresponding to the hysteresis cycle obtained during the dynamic shear test (Intervals 2, 3, and 4). Overall, all evaluated combinations or treatments (Table 4) exhibited satisfactory rheological behaviour. When the Bingham and Herschel–Bulkley regression models were applied to the descending branch of the hysteresis curve to determine the viscosity and DYS, coefficients of determination (R^2) exceeding 97% were obtained. This high degree of fit renders the selection of the optimal combination challenging, since, in principle, all treatments conform well to the theoretical models.

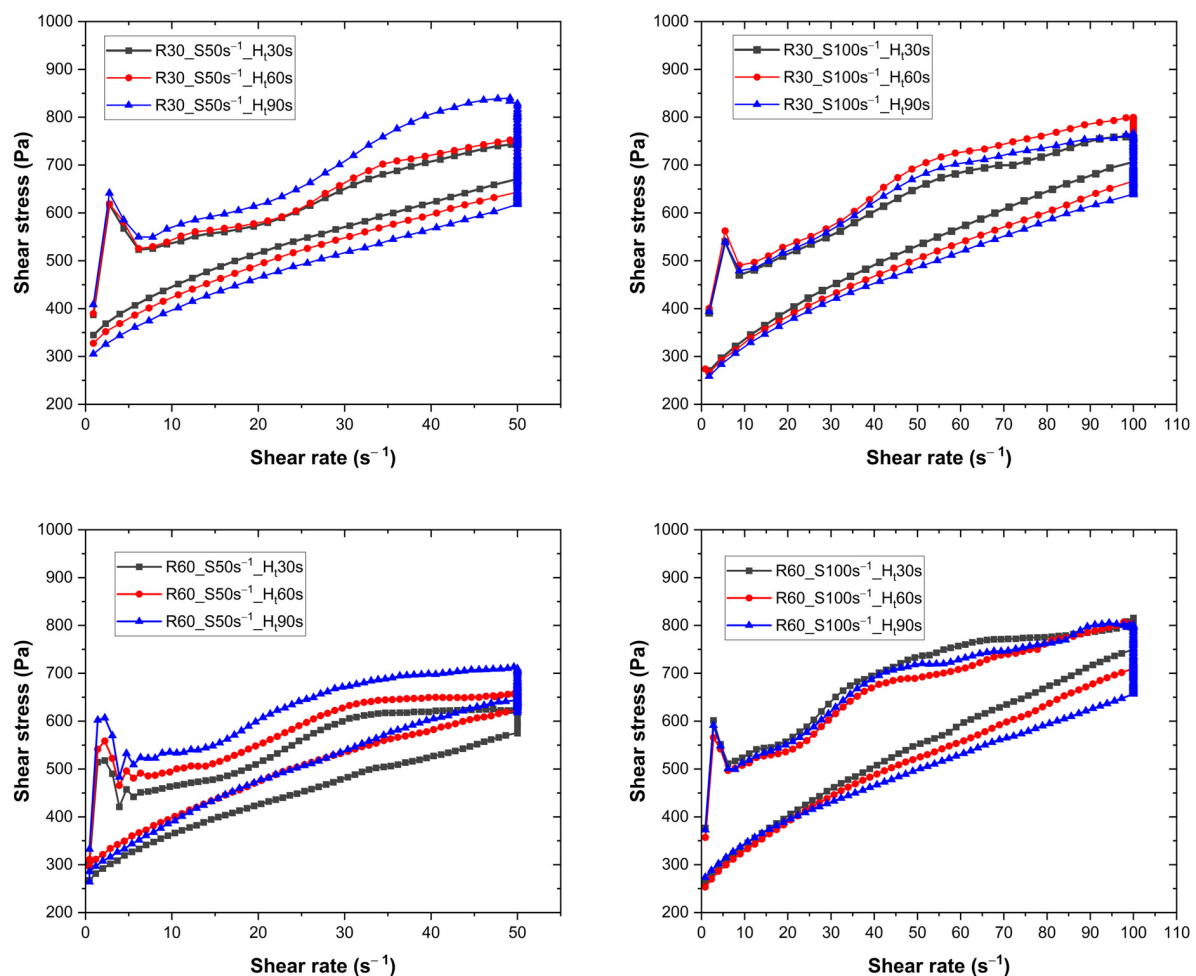


Figure 13. Hysteresis curves obtained during the dynamic shear test, grouped according to the coincidence of the first two factors (R: Ramp, s; S: Shear Stress, s^{-1}), while varying the third factor (H_t : Holding Time, s).

Statistical analysis was therefore performed to identify the most reliable rheological model for the experimental design, comparing the Bingham and Herschel–Bulkley formulations, whose outputs differ under the same test conditions. The objective was to assess the degree of conformity of each model with the experimental data by means of goodness-of-fit indicators, including the standard error (S), the coefficient of determination (R^2), and the adjusted R^2 .

Table 7 presents the goodness-of-fit statistics for each rheological model used to evaluate the effect of the experimental factors. The analysis revealed that the Bingham model provides a superior fit to the experimental data for viscosity and DYS, as evidenced by its higher coefficients of determination (R^2 and adjusted R^2) and lower standard error (S). Consequently, the response variables, namely viscosity and DYS, were analysed under a completely randomised design (CRD) with a factorial structure, using the results obtained from the Bingham model. Within this experimental framework, the statistical assumptions associated with model error, such as normality, homogeneity of variances, and independence, were verified to ensure the validity of the inferences and to support the subsequent analysis of the variance (ANOVA) and post-ANOVA tests employed to identify significant differences among treatments.

Table 7. Goodness-of-fit statistical results for the Bingham and Herschel–Bulkley rheological models.

Statistics	Viscosity		Shear Stress	
	Bingham	Herschel–Bulkley	Bingham	Herschel–Bulkley
S	0.284	3.06	21.05	19.73
R^2	96.64%	86.79%	72.25%	69.34%
Adjusted R^2	93.57%	74.69%	46.82%	41.24%

S: Standard deviation; R^2 : Coefficient of determination; Adjusted R^2 : Coefficient of determination adjusted by the mean.

4.4.1. Descriptive Analysis by Variable: Quantitative Evaluation of Reproducibility

The reproducibility of the rheological protocol (Table 8) was established by means of the coefficient of variation (CV), which was calculated for the response variables (viscosity, DYS, ATIX, and % recovery) in each of the treatments.

Viscosity: The protocol shows high reproducibility, with a mean CV of 5.04%. Of the measured treatments (T7 to T12), 58% had a CV below 5%, indicating low variation in the rheological measurements. Treatments T2, T3, T9, and T12 reported CVs below 2.5%, with T2 standing out at 0.42%. Only treatments T4 and T5 had a CV close to 9%.

Dynamic yield stress (DYS): The reproducibility of DYS showed an average coefficient of variation (CV) of 5.27%. Fifty percent of the treatments had a CV lower than 5%, with T10 showing the lowest value (0.90%), followed by T8 (1.16%) and T5 (1.40%). Treatment T11 had the highest CV (12.62%), although this could be explained by the sensitivity of this parameter to small variations in the internal structure of the paste. In conclusion, all treatments maintained a CV below 13%, demonstrating that it remains within acceptable limits for the reproducibility of this parameter.

ATIX: The average CV for ATIX was 12.98%, a value that could be considered high; however, 42% of the treatments (T5 to T12) achieved CVs lower than 10%. Treatments T1 and T8 are exceptional given their CVs below 1.5%. Treatments T2 and T4 presented CVs of approximately 29%, consistent with their characteristic of being dynamic and time-dependent thixotropic phenomena. From these results, it can be inferred that, considering that thixotropy is an intrinsically variable property, the protocol allows for reproducible measurements under most of the evaluated conditions.

Table 8. Descriptive statistics of the variables Viscosity, DYS, ATIX, and % recovery.

Treatment	n	Viscosity			DYS			ATIX			% Recovery		
		Mean	S	CV	Mean	S	CV	Mean	S	CV	Mean	S	CV
T1 (R60; S50; H _t 30)	2	5.95	0.28	4.76	320.2	26.80	8.36	110.7	1.20	1.08	0.64	0.01	1.08
T2 (R30; S50; H _t 90)	2	6.00	0.03	0.42	337.4	7.07	2.10	176.1	52.80	29.99	0.64	0.05	8.17
T3 (R60; S100; H _t 30)	2	4.74	0.07	1.46	298.3	7.70	2.58	160.0	7.52	4.70	0.59	0.00	0.55
T4 (R30; S100; H _t 90)	2	3.95	0.35	8.83	312.8	29.70	9.49	227.0	64.80	28.55	0.61	0.04	6.30
T5 (R30; S50; H _t 30)	2	5.95	0.54	9.15	380.0	5.33	1.40	107.8	4.73	4.39	0.71	0.02	2.66
T6 (R30; S100; H _t 60)	2	4.02	0.21	5.29	321.6	31.50	9.79	205.7	28.10	13.65	0.64	0.02	3.68
T7 (R60; S100; H _t 90)	2	3.77	0.32	8.55	300.8	18.30	6.08	203.5	8.47	4.16	0.60	0.02	3.34
T8 (R60; S100; H _t 60)	2	4.56	0.39	8.44	295.2	3.41	1.16	167.1	2.03	1.22	0.60	0.00	0.61
T9 (R30; S50; H _t 60)	2	6.07	0.12	2.04	351.9	7.22	2.05	127.9	16.70	13.04	0.68	0.01	1.18
T10 (R60; S50; H _t 60)	2	6.15	0.28	4.53	331.3	2.99	0.90	105.3	11.26	10.69	0.66	0.01	0.80
T11 (R30; S100; H _t 30)	2	4.50	0.24	5.28	332.9	42.00	12.62	157.7	39.00	24.73	0.65	0.01	1.11

Treatment (R: Ramp; S: Shear rate; H_t: Holding time); S: Standard deviation; CV: Coefficient of variation.

Recovery percentage: This variable showed the greatest reproducibility across all treatments, with an average CV of 2.98%, where 58% of treatments reported a CV of less than 3%. Treatments T3, T8, T10, and T11 presented CVs of less than 1.2%, indicating that the protocol is sufficiently robust to determine the structural repair capacity of the pastes. Only T2 presented a slightly higher CV (8.17%), but it was also within acceptable limits (less than 10%).

General comparative analysis: Table 9 presents a comparison of the average CVs obtained in this study and in some previous reports using non-optimised protocols. In general, the results show that the optimised protocol provides highly reproducible rheological measurements, reducing CVs by two to six times compared to conventional protocols. The average CVs obtained (5.04% for viscosity; 5.27% for DYS; 2.98% recovery) are significantly lower than those reported for rheological tests using non-standardised protocols (CV: 10–25%). The overall standard deviation of the experimental design ($S = 0.284$ for viscosity and $S = 21.05$ for DYS; see Table 7) can be considered confirmation of the low level of residual variability. Therefore, these quantitative indices support the fact that the protocol suggested in this study reduces the dispersion of results and increases the reproducibility in the rheological characterisation of cementitious pastes, achieving a level of precision similar to or greater than international standards. It should be noted that ASTM C1749 [13] establishes that the test method for acceptable rheological measurements must report CVs of less than 10%.

Regarding the relative improvement, shown in Table 9, it was calculated from the CVs reported by other authors and the CVs obtained in this study. For viscosity, an improvement of between two and four times was obtained, indicating that the optimised protocol is approximately four times more reproducible, that is, it has four times less variability than

those previously reported. Similarly, relative improvement values are reported for DYS, ATIX, and % recovery (Table 9).

Table 9. Comparative analysis of the CVs obtained in the optimised protocol and previous reports.

Parameter	CV (%) Present Study	CV (%) Previous Reports	Reference	Relative Improvement
Viscosity	5.04	10–20	[55,56]	2–4
DYS	5.27	15–30	[46,57]	2.85–5.69
ATIX	12.98	15–40	[58,59]	1.2–3.08
% Recovery	2.98	8–18	[60,61]	2.68–6.04

4.4.2. Analysis of the Experimental Design Model (ANOVA)

The models developed for the viscosity, DYS, ATIX, and percentage recovery, proposed under the completely randomised design ($2^2 \times 3$ factorial structure), satisfied the assumptions associated with the model error, namely, normality and homogeneity of variances, at significance levels (p -values) greater than 10%. The analysis of variance (Table 10) showed that, for all the variables considered, the contribution of the controlled factors was statistically greater than that of the residual error.

Table 10. ANOVA results for the response variables Viscosity, DYS, ATIX, and percentage recovery during the dynamic shear test.

Variables Source of the Effect	Viscosity R ² : 96.64%		DYS R ² : 72.25%		ATIX R ² : 78.86%		% Recovery R ² : 80.24%	
	SC	P	SC	P	SC	P	SC	P
Ramp	0.6663	0.014	5998.2	0.003	2797.6	0.094	0.009548	0.002
Shear rate	234,888	0.000	4282.3	0.009	22,426.2	0.000	0.010230	0.001
Holding time	0.0310	0.828	1589.8	0.208	9943.2	0.017	0.006195	0.024
Ramp * Shear rate	0.1071	0.272	315.2	0.415	15.6	0.894	0.000035	0.812
Ramp * Holding time	0.2002	0.324	728.5	0.463	1788.3	0.378	0.001515	0.318
Shear rate * Holding time	22,341	0.001	613.4	0.519	435.1	0.777	0.001370	0.352
Ramp * Shear rate * Holding time	11,438	0.009	313.2	0.709	459.2	0.767	0.000344	0.756
Error	0.9679		5315.2		10,150.3		0.007200	
Total	288,392		19,155.8		48,015.5		0.036437	

SC: Sum of squares; P: significance level.

Regarding the interaction hypotheses, the following observations were made:

Viscosity variable: The third-order interaction (ramp * shear rate * holding time) was statistically significant (p -value= 0.009), as was the second-order interaction (shear rate * holding time); therefore, these effects are not independent.

DYS variable: At significance levels above 41.5%, none of the interactions were statistically significant, indicating that the effects of ramp, shear rate, and H_t are independent. No significant differences were observed among the H_t levels (p -value= 0.208), whereas significant differences were found between the ramp (30 and 60 s) and shear rate (50 and 100 s^{-1}) effects (p -value= 0.003 and 0.009, respectively).

ATIX variable: At significance levels above 37.8%, none of the interactions were significant, indicating that the effects of ramp, shear rate, and H_t act independent and statistically significant. Nevertheless, the individual analysis of these factors revealed significant differences between their levels, with p -values below 0.094.

Recovery (%) variable: At significance levels greater than 31.8%, none of the interactions were significant, confirming that the effects of ramp, shear rate, and H_t are

independent and statistically significant. However, significant differences were identified among the levels of these factors, with p -values below 0.024.

Based on the ANOVA results, post-ANOVA tests were performed for the viscosity variable owing to the significant third-order interaction. For the ATIX and percentage recovery variables, post-ANOVA tests focusing on the effect of holding time were also conducted.

4.4.3. Post-ANOVA Analysis

- Viscosity variable. The post-ANOVA analysis using Fisher's LSD test revealed significant differences among the evaluated groups.

Group A: Exhibited the highest viscosity (7.3155 Pa.s), corresponding to the conditions ramp = 60 s, shear rate = 50 s^{-1} , and $H_t = 90 \text{ s}$.

Group B: Displayed intermediate viscosities (5.9490–6.1490 Pa.s) under a shear rate of 50 s^{-1} , with ramp values between 30 and 60 s and H_t of 30, 60, and 90 s, showing no significant differences.

Group C: Presented the lowest viscosity (3.7730–4.7420 Pa.s), characterised by shear rate of 100 s^{-1} and ramp values between 30 and 60 s with H_t of 30, 60, and 90 s, indicating a significant viscosity reduction.

Shear rate proved to be the most decisive factor affecting this variable, with 100 s^{-1} reducing viscosity and 50 s^{-1} maximising it when combined with a ramp time of 60 s and H_t of 90 s. These findings are key to optimising processes that depend on viscosity, such as the 3D printing of cementitious mixtures.

- DYS variable: The ANOVA revealed no significant differences among the levels of the holding time factor (30, 60, and 90 s), with the DYS values averaging between 313.0 (Pa) and 332.8 (Pa). However, significant differences were observed between the levels of the ramp factor (30 and 60 s) and between those of the shear rate factor (50 and 100 s^{-1}).
- ATIX variable: The post-ANOVA analysis using Fisher's LSD test (95% confidence level) identified two groups for the ATIX variable:
 - ✓ Group A: H_t of 90 s with a mean value of 183.205 s (Figure 14), exhibiting a significantly greater influence on ATIX, as evidenced by the increase in the variable compared with H_t of 60 and 30 s.
 - ✓ Group B: H_t of 60 and 30 s, with no significant differences between them.

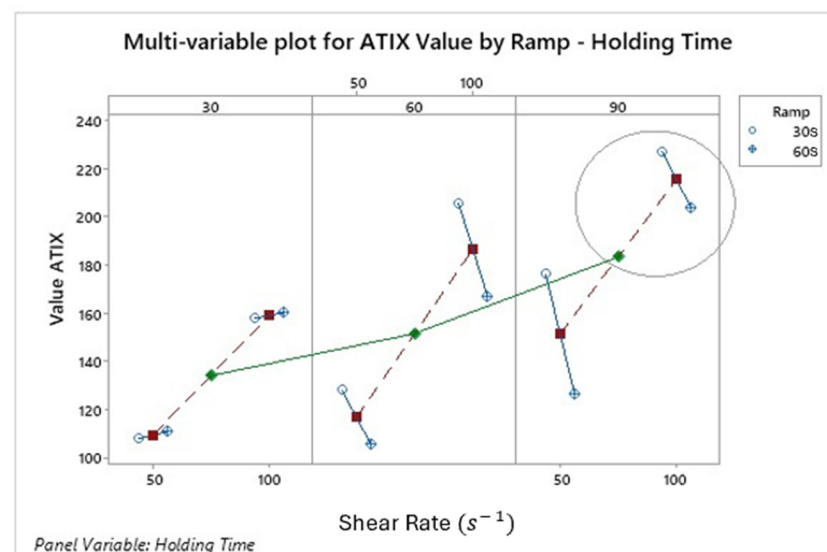


Figure 14. Effect of the factors Ramp, Shear rate, and H_t on the ATIX variable.

In addition, ATIX increased by 14.88% when the ramp duration was raised from 30 to 60 s and by 48.65% when the shear rate was raised from 50 to 100 s⁻¹.

- Percentage recovery variable: Fisher's LSD test (95% confidence level) identified two statistically distinct groups of means.
 - ✓ Group A: H_t of 30 and 60 s, which did not differ significantly from each other, with means values of 65.04 and 64.55%, respectively.
 - ✓ Group B: H_t of 90 s, with a mean value of 61.42%, corresponding to the H_t -Interval 3 that most affects the recovery of the material's viscosity.

Significant differences were also found for the ramp factor (30 and 60 s), with recovery percentages between 65.66 and 61.67%, respectively, and for the shear rate factor (50 and 100 s⁻¹), with values between 65.73 and 61.60%, respectively. Overall, an increase in the factors' levels was observed to negatively affect the percentage recovery of the material.

Once the significance of each factor and its effect on the behaviour of each property had been established, it was necessary to identify the optimal combination of parameters for the rheological method in order to achieve the best performance. Given that all evaluated combinations exhibit a coefficient of determination greater than 98%, regardless of the rheological model applied, they demonstrated consistent behaviour in the flow curves (Figure 13). The ATIX response variable was selected as the optimisation parameter, since higher ATIX values were found to improve the overall quality of the rheological curve, allowing greater sensitivity and interpretability of the method. A higher ATIX is associated with a better-defined hysteresis loop in the dynamic cutoff test, which improves the interpretation of the resulting curves and reduces the risk of ambiguity (e.g., proximity or crossing of branches), also ensuring that, under the evaluated conditions, the ascending curve remains above the descending one. In this sense, ATIX is an operational criterion for the method's clarity and robustness, aimed at broadening its application to other mixtures (e.g., formulations modified with additives, modifiers, and admixtures) while maintaining interpretability and avoiding errors due to hysteresis loop ambiguity.

Accordingly, based on both statistical and experimental analyses, the parameter combination ramp = 30 s, shear rate = 100 s⁻¹, and H_t = 90 s was identified as the optimum configuration, yielding the highest ATIX value (Figure 14). It was also observed that this combination corresponds to the lowest viscosity values, which, although not considered an optimisation parameter, indicates that the viscosity and DYS measurement depend on the rheological model used and are desirable for 3D printing of cementitious materials, as they facilitate the pumping and extrusion processes.

As a summary of this research, Figure 15 presents the proposed standardised rheological test method. The diagram outlines the parameters and time intervals defined for each of the four stages of the test, whose definition and validation constituted the focus of this study.

4.5. Effect of Sample Parameters: Mix Volume

With the test parameters established up to this stage of the research (Figure 15), the effect of the mix volume was evaluated. Figures 16 and 17 present the results of both the static shear test (Interval 1) and the dynamic shear test (Intervals 2, 3, and 4) for the different sample volumes analysed. It can be observed that the sample volume plays a decisive role in the test outcomes. Specifically, when the sample volume is below 150 mL, the behaviour of the curves in both the static and dynamic shear tests tends to deviate from the ideal response.

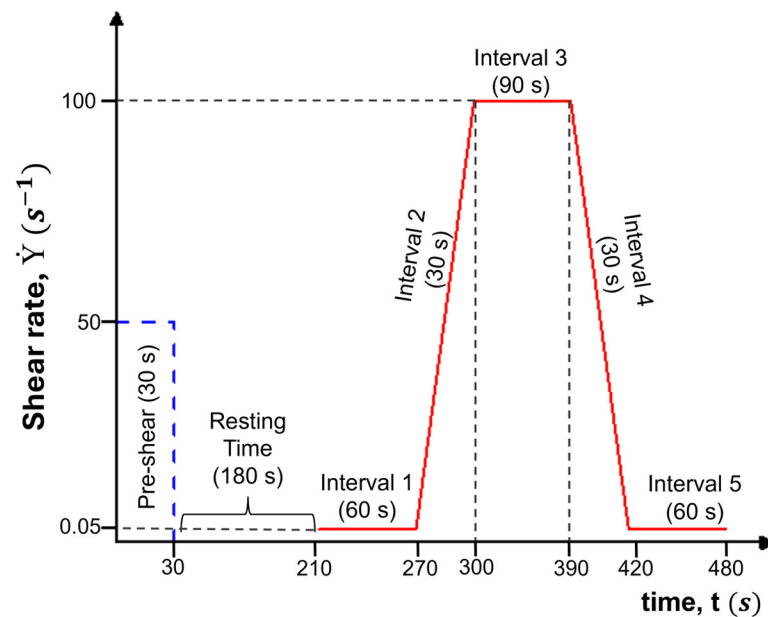


Figure 15. Schematic diagram of the standardised rheological method, for measuring the rheological properties of cementitious mixtures used in 3D printing by means of rotational rheometry.

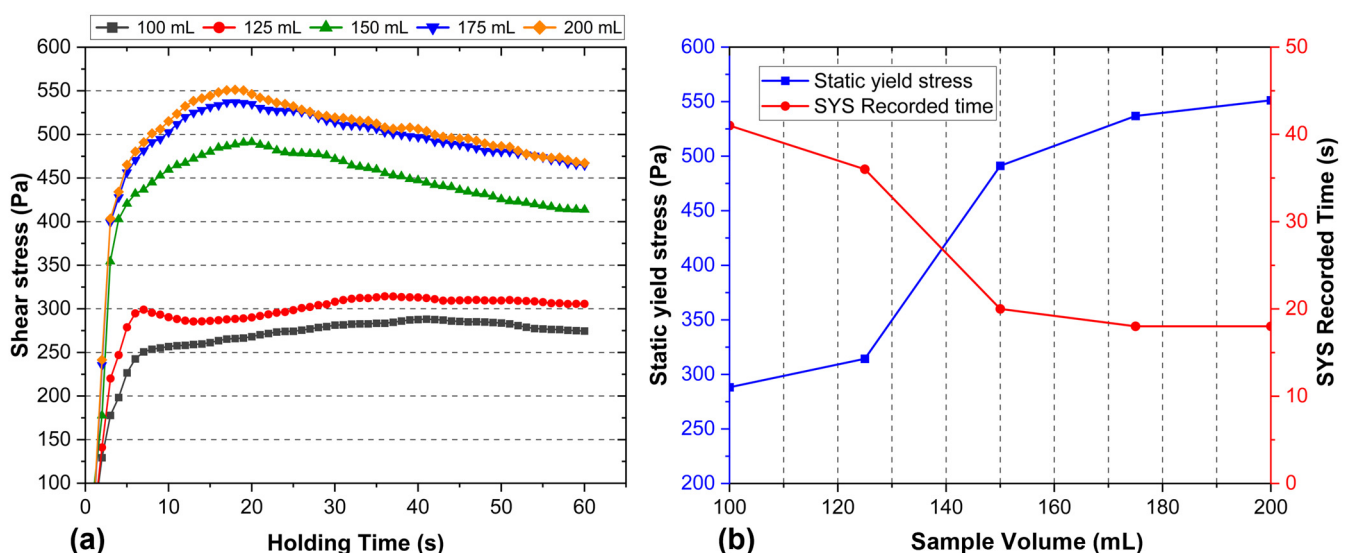


Figure 16. Effect of sample volume on the static shear test: (a) shear stress vs. time for different sample volumes: 100, 125, 150, 175, and 200 mL; (b) Yield strength (SYS) and the time at which SYS is reached.

In the case of the static shear test (Figure 16a), the curves corresponding to 100 and 125 mL reach the maximum stress values and subsequently continue to increase throughout the test period. Similarly, in the dynamic shear test (Figure 17), inconsistencies are recorded, as the ascending and descending flow curves show coefficients of determination (R^2) below 97% in all cases.

In contrast, when the sample volume exceeds 75% of the cup's maximum capacity (vol. ≥ 150 mL), the behaviour of the curves approaches the ideal response. Specifically, in the static shear test, a maximum stress peak (SYS) is recorded, followed by a subsequent decrease in stress values as a function of H_t -Interval 1. Similarly, in the dynamic shear test, the ascending and descending flow curves exhibit improved performance, as evidenced by higher coefficients of determination (R^2).

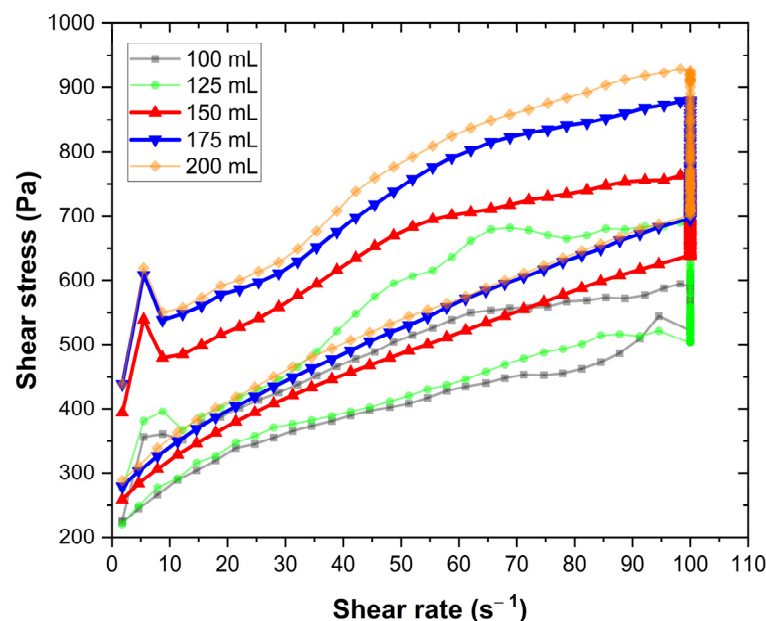


Figure 17. Effect of sample volume on the hysteresis curves obtained during the dynamic shear test (stage 3 of the rheological method). The curves for volumes of 150 and 175 mL are highlighted, as they exhibit the best performance and fit among the volumes analysed.

Additionally, Figure 16b shows that the sample volume has a pronounced effect on the SYS value. For instance, when the volume increases from the minimum (100 mL) to the maximum (200 mL), the SYS value rises by approximately 190%. Moreover, a clear inflection point is observed from 125 mL onwards, corresponding to the range where the most significant increase in SYS occurs.

This behaviour may be attributed to the fact that, when the sample volume is too low, the geometry paddle is not fully submerged, leading to inconsistencies in the measurements. However, it is equally important to avoid excessively high volumes, as these may cause the material to overflow from the cup during testing, resulting in material loss and unreliable measurements. Therefore, an optimal sample volume range of 150–175 mL is proposed, corresponding to 75–87.5% of the total capacity of the test cup. This finding is particularly relevant because, when parameterising of a rotational rheometric test using geometries based on the concentric cylinder's principle, the sample volume must be explicitly defined, yet this parameter is rarely specified in equipment manuals. Furthermore, many studies in the scientific literature omit this variable altogether, creating a gap that hinders the standardisation of rheological methods and, consequently, the comparability of results across different investigations.

5. Conclusions

Through experimental and statistical analysis, a standardised method for the rheological characterisation of cementitious mixtures for 3D printing by means of rotational rheometry was developed and validated. The study demonstrated the strong interdependence between the variables and test parameters, as well as the high sensitivity of the results to factors such as shear rate, resting times, and holding times and the inclusion of ascending and descending ramps in the test method. This complexity highlights the need for meticulous adjustment and control of each parameter, which is essential to ensure the standardisation of the protocol and, consequently, the reliability and reproducibility of the results.

The results confirmed that pre-conditioning plays a fundamental role in optimisation the rheological measurements of cementitious materials. The following parameters were

defined as optimal: a pre-shear stage at 50 s^{-1} for a duration of 30 s, followed by a resting time of 180 s at zero shear rate. The absence of this stage makes it difficult to identify the inflection point associated with static yield stress (SYS), whereas its inclusion allows proper homogenisation of the material and behaviour consistent with theoretical models. Furthermore, pre-conditioning was found to significantly influence both static and dynamic shear tests, reinforcing the need to standardise its parameters to achieve accurate and representative measurements, particularly in the determination of the static yield stress (SYS).

The shear rate was shown to exert a major influence on the results of the static shear test used to determine the static yield stress (SYS). At very low shear rates (approx. 0.01 s^{-1}), the material undergoes structural reorganisation that prevents the establishment of adequate flow conditions. As the shear rate increases, the static yield stress also increases, with a progressive transition to flow being observed and optimal behaviour found in the range of $0.05\text{--}0.1 \text{ s}^{-1}$. However, at shear rates above 0.1 s^{-1} , the static yield stress (SYS) is recorded almost instantaneously, making it difficult to identify the maximum peak on the curve.

It was determined that the Bingham model provides a superior fit for the viscosity and dynamic yield stress (DYS) variables compared with the Herschel–Bulkley model. The ANOVA results showed that viscosity is significantly affected by the interaction between the evaluated factors (ramp, shear rate, and holding time), whereas in the case of DYS these factors act independently. For the ATIX and percentage recovery variables, it was observed that the H_t -Interval 3 of the maximum shear rate has a significant impact, particularly on ATIX, where an H_t of 90 s yielded the highest values. Based on these findings, the optimal combination of parameters to enhance the quality of the rheological method during the dynamic shear stage corresponds to: ramp = 30 s, shear rate = 100 s^{-1} , and H_t = 90 s. This configuration maximises the ATIX value and is associated with lower viscosity values, which are desirable for 3D printing applications, as they facilitate material pumping and extrusion.

The results also demonstrated that the sample volume has a significant influence on the rheological measurements. For this study, the optimal range was established between 150 and 175 mL, corresponding to approximately 75–87.5% of the total cup capacity.

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