# Impact of Using Bauxite Residue in Association with Portland Cement during the Early Age of Suspensions

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**Abstract**

As reported in the roadmap of the International Aluminium Institute the association of bauxite residue (BR) with Portland cement, during the production of clinker or as supplementary cementitious material in compositions of mortar or concrete can be the most impactful applications for this waste. However, while considering the first application it is possible to apply up to 3% of BR replacing the clay in the composition of clinker (without any reduction on the CO2 released during the clinkerization), the use in compositions of mortars or concretes could allow replacing at least 10% of binder. Nevertheless, these values can change a lot in function of the main physical, chemical, and mineralogical properties of BR, and of the level of substitution. In our strategy for the search of a safe large-scale application to BR, we started the research evaluating the impact caused in the hydration reaction of Portland cement, because this is the main indicator of the chemical interaction between them. However, the impact on the workability and hardening of compositions is another aspect to be considered: the increase in the mixing water demand impacts directly on the performance and durability of materials in use. So, the main purpose of this work was to evaluate the impact of using different proportions of BR (from 5 to 40%) during the hardening of the cement compositions, from a combined evaluation of isothermal conduction calorimetry, rotational and oscillatory rheometry. Results indicated that the impact of using the residue collected in Aluminio (state of São Paulo - Brazil), had negligible impact on the chemical reaction, during the flow or even during the hardening of compositions. This information is one of the most important steps to define the BR content that can be applied in compositions with lower impact on the hardened state properties.

**Keywords:** Bauxite residue, Portland cement, chemical reaction, rheology.

## 1. Introduction

Bauxite residue (BR), or as commonly also known, red mud, is an insoluble residue from the Bayer process whose was not yet developed any large-scale application. So, it is disposed into the lakes of mud specially built for this purpose [1,2].
As the production of aluminium and alumina are increasing worldwide, its generation is following the same tendency: it is estimated that for each ton of Al is discarded from 1 to 2 tons of BR depended on the chemical characteristics of ore, process of digestion, and the process of disposal (slurry or made by filter press) [1,3].
It is reported by some different researchers [1,2,4–8], that one of the most impactful potential for a large-scale application for this residue is the association with Portland cement in compositions of cementitious components [1,9,10] or even during the production of clinker Portland [10,11].

Cement sector was responsible for the production of more than 3.6 billion tons of the binder in 2012 [12], representing the industrial material most used worldwide. However, during its production can be released up to 1 ton of $CO_2$ per each 1 ton of cement produced [13]. In this context, BR generated represents around 3-4 % of Portland cement production.

At the same time, cement industry is constantly looking for alternatives to reduce the impact of $CO_2$ release to the atmosphere, and some strategies reported are: improvements on the kiln efficiency, use of better fuels, application of the concepts of carbon capture and storage, and use of supplementary cementitious materials (SCM) [14]. The RoadMap [15] did by the cement association pointed out a challenge to reduce around 33% of $CO_2$ up to 2050, indicating that the most significant strategy is the use of SCMs.

By definition, supplementary cementitious materials comprise those that, in the presence of water, because they have calcium in their composition, or react with the calcium released in Portland cement hydration, set and hardens forming hydraulic products, that is, resistant to the action of water [16,17]. This name was extended to all material that, in some way, interferes with the hydration of cement, as is the case of the limestone filler that interacts with $C_3A$ [13,18–21] or another kind of material that could promote the nucleation effect for the grow of hydrated compounds.

Although there are many types of SCMs (slag, silica fume, metakaolin, fly ash etc.), they are scarce resources to attend the demand of the Portland cement production, due to logistic aspects or availability of each material [22].

Additionally, the replacement of Portland cement by different kinds of SCM is not trivial, because they have different physicochemical and mineralogical characteristics, and its association promotes different microstructural development during the hardening, and performance in use [2,13,23]. Figure 1. , adapted from the work of Lothenbach *et al* [13], illustrates a ternary diagram of calcium oxide, silica, and alumina content for the SCM commonly used in the present days. The ratio between these chemical species in the composition of bauxite residue evaluated worldwide was also illustrated, to indicate that if BR will be used as a SCM, can be a product characteristics different from the others [3,24].

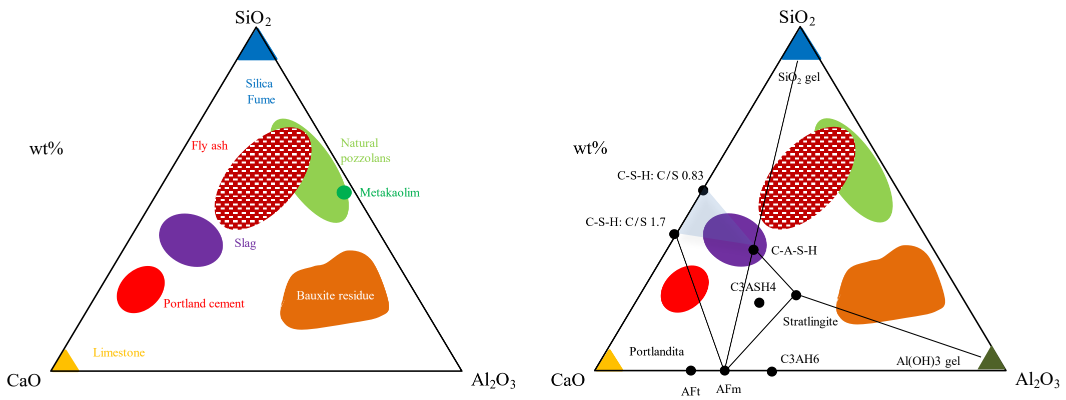


**Figure 1. A) CaO–Al2O3–SiO2 ternary diagram of cementitious materials, B) hydrate phases in the CaO–Al2O3–SiO2 system. Adapted of Lothenbach et al [13], using bauxite residue.**

Association of any mineral addiction with Portland cement is important have a three characteristics chemical: Calcium, aluminates, and silicates; that in association with Portland cement improve the hydrates formation of thugs. In this case, bauxite residue is rich in aluminates, that tend the formation of C-A-S-H, stratlingite and $Al(OH)_3$-gel. It is important to point out that any addition to replace Portland cement, as they have different physical characteristics (Specific surface area, density, particle size distribution), promotes changes in rheological properties, the casting stage or in the consolidation process.

So, this paper was carried out with the main purpose of evaluate the impact of the impact of using different proportions of BR (from 5 to 40%) during the hardening of the cement compositions, from a combined evaluation of isothermal conduction calorimetry, rotational and oscillatory rheometry. This strategy has the objective of evaluate the technical viability of using bauxite residue in association of cement compositions with no losses in the first stage of development of components. i.e.: very early age consolidation.

## 2. Experimental

### 2.1 Materials

Formulations were produced using a Brazilian Portland cement, named as CPV (a binder produced with clinker Portland, up to 15% of limestone and 3% of gypsum), and bauxite residue (BR) from Brazilian Aluminium Company – CBA. BR was dried at 105 ºC and used without any sieving, to reduce one step for its application. This drying was performed only to guarantee the water to solid ratio during the production of suspensions.

The particle size distributions of each raw material are show in Figure 1, quantified using an equipment Helos Sympatec, with range of 0.1-350 µm. In the sequence, Table 1 illustrates the real density, determined using a gas He pycnometer (Multipycnometer – Quantachrome Instruments [25]), and the specific surface area, obtained using the method BET in an equipment Belsorp Max, Bel Japan [19,20]. Table 2, presents the chemical analysis by X-ray fluorescence, in an equipment Axios Advanced – Panalytical [25].

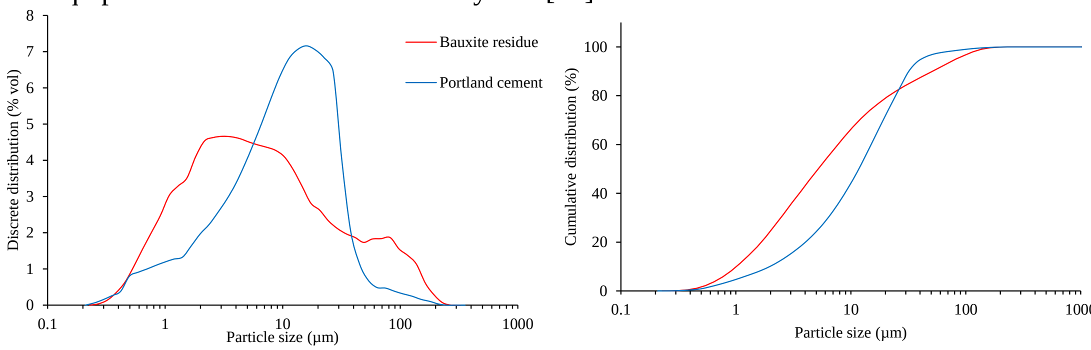


**Figure 2. Particle size distribution of raw materials.**

**Table 1. Physical characteristics of raw materials.**

| Sample | Real density (g/cm³) | Specific surface area (m²/g) | $d_{10}$ (mm) | $d_{50}$ (mm) | $d_{90}$ (mm) |
|---|---|---|---|---|---|
| Portland cement - CPV | 3.14 | 1.15 | 1.39 | 11.8 | 31.9 |
| Bauxite residue | 2.88 | 18.2 | 1.10 | 5.70 | 55.9 |

Portland cement and bauxite residue present similar particle size extension, but 90% of cement particles are lower than 32 µm, while for BR are lower than 56 µm. However, 50% of BR are lower than 5.7 µm, representing that this residue, even without any additional treatment, has finer particles, with potential to be used as a filler in association with the binder. The main challenge is guaranteeing the adequate deagglomeration during the mixing of pastes.

However, the high specific surface area of BR can difficult its high-level association with cement, if was not mixed with any chemical admixture for dispersion or using high shear energy of mixing, because can increase the water demand for cover the particles and disperse them. This is important because the water consumption directly impacts on the performance of cement products in use [14,27].

**Table 2. Chemical characterization of raw materials, in oxide.**

| Chemical elements | Portland cement – CPV | Bauxite residue |
|---|---|---|
| $SiO_2$ | 18.7 | 18.5 |
| $Al_2O_3$ | 4.64 | 21.3 |
| $Fe_2O_3$ | 2.64 | 29.0 |
| CaO | 61.6 | 5.56 |
| MgO | 1.16 | - |
| $SO_3$ | 2.51 | - |
| $Na_2O$ | 0.42 | 6.96 |
| $K_2O$ | 0.85 | 0.21 |
| Alcaline equivalente in $Na_2O$ | 0.98 | 7.10 |
| $TiO_2$ | 0.30 | 3.02 |
| $P_2O_5$ | 0.21 | 0.56 |
| MnO | <0.1 | 0.13 |
| SrO | 0.36 | - |
| Loss on ignition | 5.41 | 14.4 |

The loss on ignition illustrates the decomposition of water and $CO_2$ (if there is a presence of carbonates in the raw material). The percentage quantified for Portland cement illustrates that this cement is in accordance with Brazilian Standard, being detected around 12% of limestone addition. For BR, the loss on ignition of 14.4% indicates the free water, remaining from the drying, and the combined water from zeolite and gibbsite and goethite.

The high level of sodium can also limit the use of BR in association with Portland cement, because considerably affect the alkaline equivalent. This alkali, if not combined during the formation of hydrated compounds, can be easily leachable, causing environmental problems. However, this will not be a problem if the chemical synergism between Portland cement BR is positive. The use of additions like silica fume and slag can contribute to this chemical fixation [3,28].

### 2.2 Methods

Compositions were formulated with partial replacement of Portland cement by bauxite residue, between 10 at the 40% in volume. This dosage in volume was chosen to guarantee the same spatial filling due to the differences of densities.

Suspensions were prepared using a high-shear energy equipment, at 10,000 rpm, with a cawles impeller. The powder was disposed into an aluminium container and the water was added (water-to-solid ratio of 0.5), waiting 30 seconds for the initial wetting. Then, the mixing procedure starts and was performed for 90 seconds. The suspensions obtained were used in the evaluations described below.

- Rotational rheometry: tests were performed in an equipment Mars 60 – Haake, controlling the shear rate from 0.01 to 50 $s^{-1}$ (acceleration) and followed by a deceleration step from 50 to 0.01 $s^{-1}$. It was used a parallel plate geometry with 35 mm, gap of 1000 µm, and temperature control of 23ºC.

- Oscillatory rheometry: performed applying the concept of time sweep test in the sequence of rotational rheometry. Tests were controlled using frequency of 1Hz and strain of $10^{-4}$, for 4 hours. As a result we have the evolution of G' over the time [5]. During the tests, a solvent trap was used to maintain the sample humidity.

- Isothermal calorimetry: heat flow during the hydration reactions was monitored in a TAMAir calorimeter from TA Instruments with precision of ± 20 µW, maintaining the temperature at 23°C for 48 hours.

## 3. Results and Discussion

The first step of rheological evaluation was the rotational rheometry, to understand the rheological properties of the pastes related to the stage of application. The changes in the shear condition allowed us to define, comparatively, the impact of replacement level of Portland cement by BR on the yield stress, viscosity, and rheological behaviour. This is the first stage for a search of any kind of supplementary cementitious material: the impact on rheological properties should be negligible or small to not require any additional material treatment or use of chemical admixtures.

Results obtained were illustrated in Figure 3, correlating shear stress (above) and viscosity (below) with the shear rate. This kind of representation indicated a shear thickening behaviour of pastes for all the compositions. So, the use of BR did not affect the rheological behaviour of pastes.

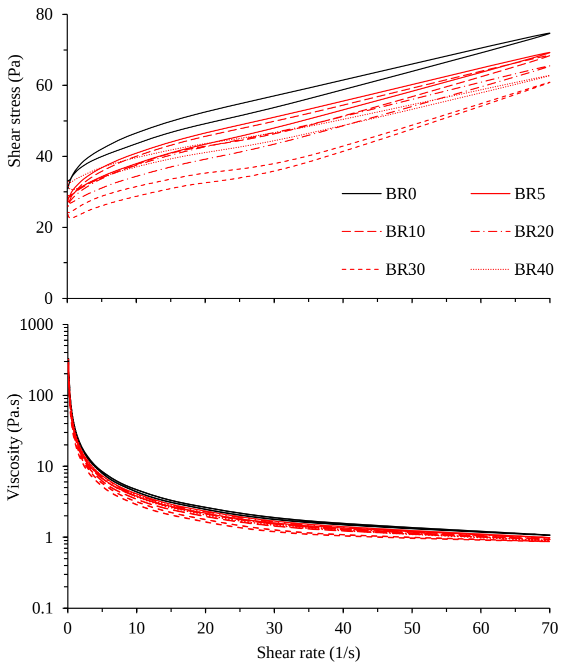


**Figure 3. Shear rate and viscosity in function of shear rate applied.**

The yield stress and plastic viscosity were calculated applying the Herschel-Bulkley model and the results are presented in Figure 4. Yield stress is defined as the minimum value of shear stress that needs to be exceeded to start the flow, i.e., if the shear stress is lower than this value, the paste is not flowing (is at rest). From the moment that the flow starts, it becomes to be governed by viscosity, which is defined as a parameter of energy dissipation that represents the resistance of the material to the flow.

So, according to the rheological parameters obtained applying the model, the use of BR affected the viscosity and yield stress in a different way: while the raise in BR content resulted in a considerable increase in plastic viscosity, the yield stress decreased.

It was observed that the replacements up to 20% of BR did not cause significant changes in the flow, but consumptions higher than 30% caused an exponential increase. On the other hand, it can be said that the yield stress decreased using BR, but there is not a significant change in the function of BR content. So, it is reasonable to say that up to 20% of BR the early rheological parameters were not affected.

It is also important to say that this tendency was not observed in other works using BR [3,29], but this does not mean that it is wrong. This only means that characteristics of BR change from site to site, and the association with Portland cement depends on the physicochemical properties of each material.

Even with the considerable difference between the specific surface area of BR and Portland cement (value around 16 times higher), the raise in BR content can also affect the surface charge of suspension and the pH and ionic forces of the suspensions, variables that affect the rheological properties, according was also demonstrated by *Liberato et al.* [29].

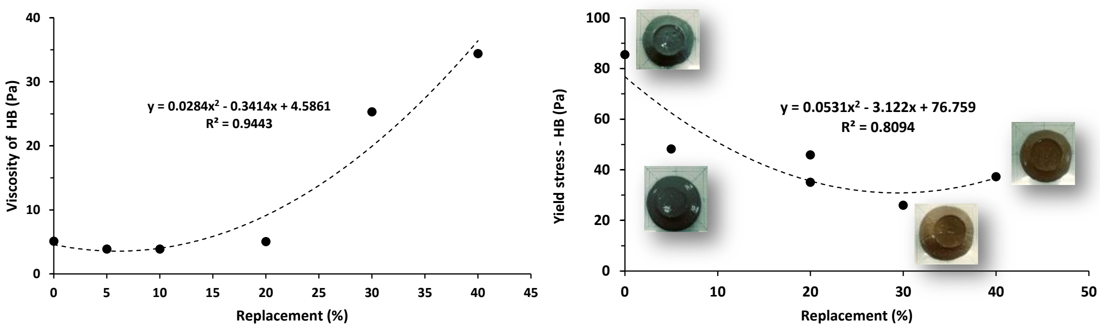


**Figure 4. Herschel Bulkley models for replacement of BR.**

A challenge reported in the literature concerning the works about BR is the need for an increase of water demand to maintain similar rheological properties [30], i.e., to keep constant the workability for application. However, in this work was proved that this is not a tendency for all the contents of the residue from CBA evaluated, and a high level of replacement could be used in cement compositions without impact on the early age flow. Of course, this is a rheological issue, and the level of replacement also depends on the chemical compatibility of BR and Portland cement, and with the aggregates [3,30].

Beyond the very early rheological evaluation, the impact of using BR during the hardening stage is the second step for the search for a large-scale application in association with Portland cement.

After the mixing, transportation, and moulding of the components, the gain on consistency over time, and consequently, the gain of strength at the beginning of hardening are stages that must be adequately monitored.

The cement hydration reaction governs the chemical contribution of hardening and the presence of BR in the compositions affects the nucleation points and formation of new hydrated phases [5,24]. On the other hand, the physical aspects of hardening are as important as the chemical contribution, because impacts the agglomeration and coagulation of particles, creating permanent links between them [24,31].
Of course, both chemical and physical parameters occur concomitantly, but using a combined evaluation of isothermal conduction calorimetry and oscillatory rheometry, it can be possible to understand which is dominant during the beginning of hardening.

Figure  illustrates the evolution of G’ over time for the compositions with different BR content. G’ express the elastic response of material during the transition from fluid to elastic behavior in the consolidation. At the oscillatory test, part of the applied strain (at a controlled frequency) is dissipated by the energy dissipation mechanisms in the bulk of cement paste, and another part is stored in the material: higher G' denotes more solid-like property. So, during the time sweep test, G’ was monitored under a quasi-static condition, indicating the development of the suspension stiffening when at rest and that can be related to the applied or molded material [6].

At the first 2 hours of monitoring, the use of BR promoted an intensification of G’ mainly due to the agglomeration of the particles: during the evaluation of the flow of pastes, the shear rate imposed was responsible for broking down the agglomerates, but when the material is at rest and applying a frequency and strain lower than that need to start the flow (in the linear viscoelastic region – LVR) [31], the development of microstructure occurs without any external interference. So, the gain on consistency at the first 2 hours was more intense using BR (comparing with reference composition).

After this period, compared with the reference, compositions formulated with 10 and 20% of BR presented a clear intensification of G’, while BR30 and BR40 presented lower microstructure rigidification. No difference was observed using 5% of BR.

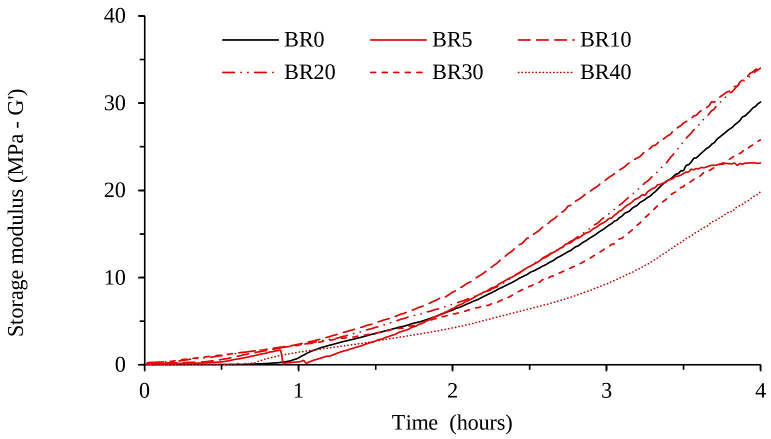


**Figure 5. Evolution of the storage modulus at the first 4 hours of hardening**

On the other hand, the chemical contribution to the hardening is exposed in Figure 6 monitoring the heat flow released (on the left) and the cumulative heat (on the right) during the hydration reaction of cement with different BR content. The use of mineral additions affects the dilution of clinker phases and the nucleation, so changes on the heat released represents the impact of different content of bauxite residue addition.

The use of bauxite residue in association with Portland cement makes the hydration reaction increasingly complex: the presence of soluble aluminates in contact with $Ca^{2+}$-ions from Portland cement, produces calcium aluminates, and it can accentuate or cause a delay in the hydration reaction, depending on the concentration of dissociated aluminates from bauxite residue. However, in this work it was not observed a delay in the induction period. *Scrivener et al.* [27] using the geochemical theory of dissolution, suggest the growing of disorganized C-S-H on the original grains and finishing the induction stage even at $Ca^{2+}$ undersaturation.

The most impactful observation according to the use of this BR occurred after the induction period, according better illustrated in Figure , representing the reaction of alite and or during the reaction of aluminates of clinker. The profile heat released indicates that the raise of BR content resulted in a considerable increase in the reaction rate and intensified the formation of C-S-H, CH, CASH and NASH, respectively, calcium silicoaluminate, portlandite, calcium aluminosilicate hydrated and sodium aluminosilicate hydrated [24], and anticipating the formation of ettringite (Aft) and the conversion in monosulfoaluminate (Afm). This is in accordance with was reported by *Nicoleau et al.* [32] using other materials: the BR use influenced considerably the formation of Si-O-Al bonds.

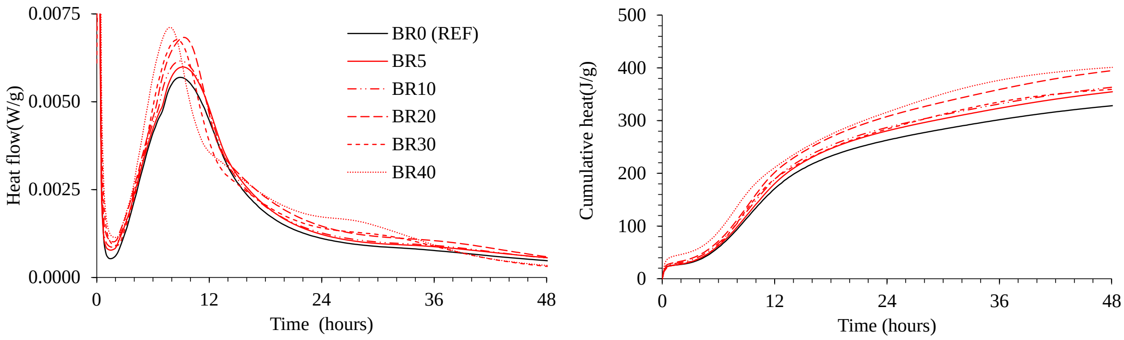


**Figure 6. Isothermal conduction calorimetry up to 48 hours.**

The setting time obtained according to the ASTM – Standard Practice for Measuring Hydration Kinetics of Hydraulic Cementitious Mixtures Using Isothermal Calorimetry is presented in Table , and the results indicated a negligible impact of using BR in the compositions. According to this standard, this is the time to reach 50% of the heat released in the main hydration reaction peak (heat released during the acceleration period of the reactions).

**Table 3. Setting time made from isothermal calorimetry**

| Sample | BR0 | BR5 | BR10 | BR20 | BR30 | BR40 |
|---|---|---|---|---|---|---|
| Setting Time (hour) | 4:35 | 4:35 | 4:35 | 4:45 | 5:00 | 4:35 |

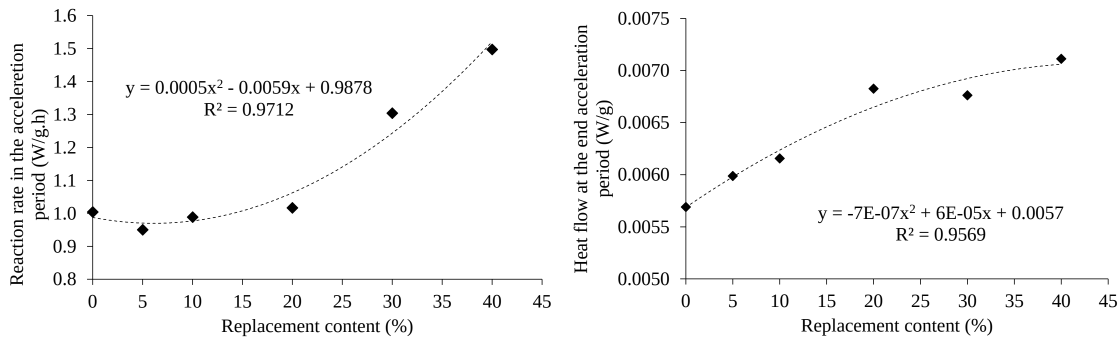


**Figure 7. Main periods of hydration heat.**

So, combining the physical and chemical evaluations, performed using the concepts of oscillatory rheometry and isothermal conduction calorimetry, it was clear that replacements of up to 20% of

Portland cement by BR caused a negligible impact on the hardening at the first 4 hours of consolidation. Higher BR contents intensifies the chemical contribution to hardening while becomes the microstructural less rigid. This does not mean that high level of substitution cannot be used (it the performance and durability parameters on the hardened state were met). This is only the profile of pastes evaluated with these raw materials.

As there are many kinds of cementitious components and products applied in the civil construction sector, each applied differently, the initial properties of the suspensions do not necessarily need to be the same. Therefore, knowledge of the peculiarities of each composition is of great importance for the development of products with better properties.

This joint assessment becomes even more important when it comes to the use of waste from different sources, as there may be considerable physicochemical variations that interfere with hardening.

## 4. Conclusion

This paper evaluated the effect on the fresh state properties of cement compositions made with different levels of bauxite residue replacement.

Our strategy was to show a way of an initial evaluation of the bauxite residue, assuming that the monitoring of the very early rheological properties and the hardening stage are the first steps for this selection. The methods applied are used to select any type of supplementary cementitious material.

Increasing the residue content up to 20% had a negligible impact on the fluidity of the pastes, but higher contents resulted in an exponential increase in viscosity. This indicates that in practice it would be necessary to increase the amount of kneading water to ensure the workability of the product during application.

During the hardening stage, the viscoelasticity of the cement paste was altered by the presence of BR, mainly for levels of up to 20% replacement. Higher contents resulted in a decrease in microstructural stiffness in the first hours of evaluation.

With the increase of the BR content, there was an intensification of the cement hydration reaction after the induction period, illustrating that there is a nucleation effect for the growth of hydrate crystals.

With the combined analysis between the physical and chemical parameters of the hardening stage, it was possible to observe that substitutions of up to 20% of BR in relation to cement cause negligible interference in the development of the hydrated microstructure.

Thus, the monitoring of the transition from fluid to solid behaviour of cementitious pastes from the joint evaluation of the results of calorimetry and oscillatory rheometry is an efficient alternative for understanding the phenomena that govern consolidation.

However, based only on these results, it is not yet possible to conclude that such content of BR is safe for use on a large scale to produce cementitious components. There is a need for a joint assessment of hardened state performance and durability to monitor chemical fixation of potentially leachable or late-reacting chemical species with cement or aggregates.

## 5. Acknowledgements

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