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ORIGINAL RESEARCH ARTICLE

Comparative analysis of mechanical properties of geopolymers incorporating SiC nanowhiskers and TiO_2 nanoparticles

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ABSTRACT

A metakaolin-based geopolymer was fabricated with 5 ratios of two different nanomaterials. On the one hand, silicon carbide nanowhiskers and, on the other hand, titanium dioxide nanoparticles. Both were placed in water and received ultrasonic energy to be dispersed. The effects on mechanical properties and reaction kinetics were analyzed. Compared to the reference matrix, the results showed a tendency to increase the flexural strength. Probably due to the geometry of the SiC nanowhiskers and the pore refinement by the nano-TiO_2 particles. The calorimetry curves showed that incorporating TiO_2 nanoparticles resulted in a 92% reduction in total heat, while SiC nanowhiskers produced a 25% reduction in total heat.

Keywords: Geopolymers; Nanomaterials; Mechanical Strength

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1. Introduction

1.1 Geopolymers

"Geopolymer" can be considered a generic term to define an alternative binder to Portland cement paste. Structured as an inorganic polymer and with similar or higher mechanical strength than a Portland cement cementitious material[1–3], it offers environmental advantages during its manufacture such as: the use of industrial waste[4], reduction of the calcination temperature in clay materials, which positively affects the emission of CO_2 into the atmosphere[1], encapsulation of toxic elements[5–7], resistance to acid attack and other types of attacks[2,3]. However, as a ceramic material, it has certain limitations that continue to be investigated, such as: easy propagation of cracks that can compromise mechanical strength[8,9], flexural strength lower than compressive strength[10,11] and the appearance of efflorescence[12].

Many ceramic and polymeric materials have mechanical limitations that can be reduced by using reinforcement elements in different scales such as steel, metallic fibers, vegetable fibers[13], polypropylene microfibers[14]. In this way, the geopolymers become a matrix and the additive element acts as a reinforcement against the weaknesses of the main matrix.

1.2 Nanomaterials in geopolymeric and cementitious matrices

Nanomaterials, in the form of fibers or particles, can also contribute to the modification of the microstructure of cementitious matrices, attributing their properties and improving their mechanical response. Adding nanomaterials in geopolymeric matrices has been a trend in recent years. The results show: increases in compressive strength when materials such as nano-clay, carbon nanotubes (CNT) and nano-SiO_2 are added[15–18], densification of the microstructure, reduction of the initial setting time, reduction of shrinkage[19,20], increase of ductility with the use of carbon nanofibers (NFCs), alumina nanofibers (NFAs), silicon carbide whiskers (WSC)[10], and even NTC, which are the most commonly used[20–22].

SiC is widely used in mechanical engineering for its high abrasion and wear resistance, high hardness record, thermal stability, flexural strength and others[23]. SiC can be found in the form of fibers, nanoparticles of SiC (NPSC) or nanofibers, called nanowhiskers of SiC (NWSC). These are usually incorporated in epoxy-type resin and alumina matrices[24,25].

For chemistry, TiO_2 is considered the best photocatalyst, chemically stable and low cost[26], which enables the degradation of organic pollutants in aqueous media[27]. It is normally used for water treatment, paint pigmentation and sun protection. Over time, it was considered to introduce this property in ceramic materials with the use of titanium dioxide nanoparticles (NT) and thus, they would be transformed into materials with photocatalytic properties and consequently, more durable materials[28–30].

In several studies[31,32], it was indicated that NT would not be producing relevant changes on geopolymeric matrices. Despite this[33–35], they were able to record increases in compressive strength when NT was added to geopolymers based on blast furnace slag or fly ash. Increases in compressive strength, up to 51% over the reference matrix, when 5% NT was used in the early ages.

In the nanomaterial form[36] incorporated SiC nanowhiskers in Portland cement matrices and obtained relevant changes in compressive and flexural strength when they added 0.25% and 1.00% NWSC in relation to the cement mass, respectively. Similarly[37], added SiC nanowhiskers in a geopolymeric matrix, registering an increase of up to 192% in the flexural strength of the reference matrix when 0.2% was added in relation to the mass of metakaolin.

1.2.1 Implications of the use of nanomaterials

The difficulty of nanomaterials lies in the fact that, due to their small size and high specific surface area, they tend to agglomerate. Van der Waals forces become more intense under these size conditions, making it difficult to disperse them in the dry state and even within water. NTC are an example of this[22]. Therefore, dispersion techniques are commonly used to alter the surface of nanomaterials and/or cause particle separation by using chemical surface treatment with acid, surfactants, applying ultrasonic energy or altering the pH of the medium to obtain homogeneous compounds.

This study focuses on understanding the effects of the incorporation of titanium dioxide and silicon carbide based nanomaterials, individually and together, on a geopolymer matrix. The fraction used for NWSC was 0.10% and 0.20%, while for NT it was 0.50% and 1.50%, on the weight of metakaolin. In view of having favorable results during additions by other authors, the influence of these nanomaterials on the compressive strength, flexural strength and reaction kinetics measured by isothermal conduction calorimetry is analyzed.

2. Materials and methods

The aluminosilicate source, metakaolin (MK), was granted by the company Metacaulim do Brasil, São Paulo, Brazil, its granulometric distribution is shown in **Figure 1**. Its chemical composition is recorded in **Table 1**. The activating solution was constituted by sodium hydroxide (NaOH) in bead format (>98% purity) and a sodium silicate solution (SiO_2/Na_2O = 2.5), both Sigma Aldrich brand. The water used was distilled. The SiC nanowhiskers (NWSC) were obtained from Nanostructured & Amorphous materials Inc., Texas, USA; their main characteristics are shown in **Table 2** and the geometry of one of these is shown in **Figure 2**. The TiO_2 nanoparticles (NT) were obtained from the Aldrich company and their characteristics are recorded in **Table 3**. Similarly, **Figure 3** shows the geometry of the nanoparticles.

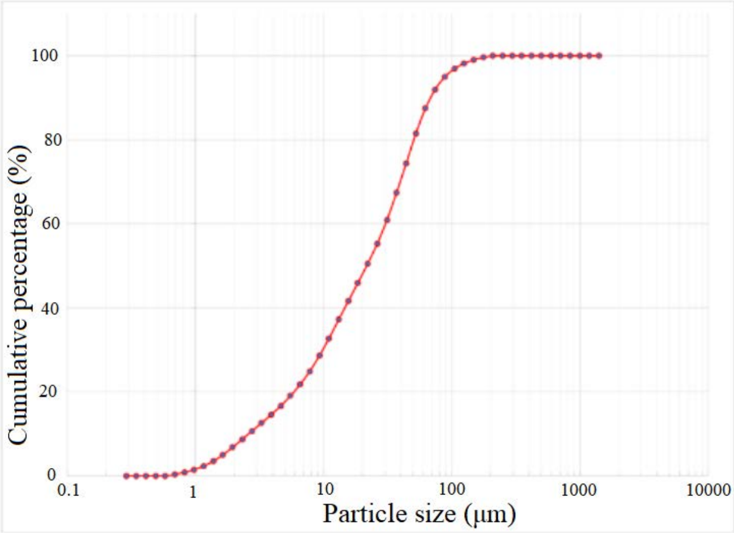

Figure 1. Granulometric distribution of metakaolin.

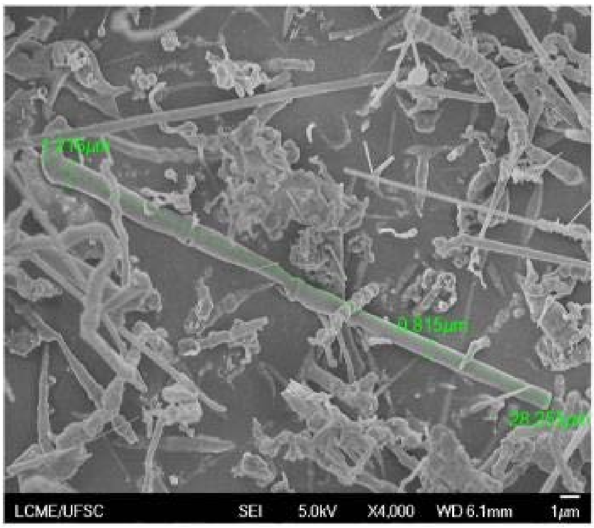

Figure 2. Transmission Electron Microscope (TEM) image of NWSCs.

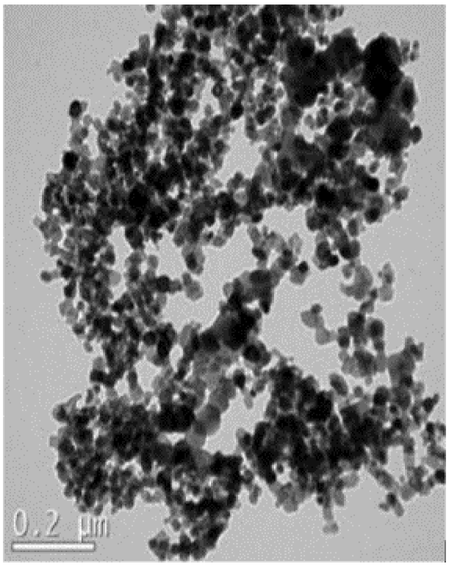

Figure 3. MET image of NT[38].

Table 1. Chemical composition of metakaolin

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	TiO_2
%	57.0	34.0	0.10	0.10	1.50

Source: Metacaulim do Brasil[2].

Table 2. Characteristics of the NWSC

Free carbon	<0.05	Type of glass	Beta
Diameter	0.1–2.5 μm	Length	>2.0–50 μm
Hardness (Mohs)	9.5	Density	3.216 g/cm^3

Table 3. Characteristics of titanium oxide nanoparticles (NT)

Diameter	21 nm	**Surface area**	35–65 m^2/g

The activating solution (12 M NaOH) was prepared by slowly dissolving NaOH in sodium silicate. Since this combination is strongly exothermic, it was necessary to let the solution stand until it reached room temperature 23 ℃ ± 2 ℃. On the other hand, the nanomaterial was added in distilled water, slightly stirred and the solution was separated to receive the ultrasonic energy applied by a Vibra-Cell 750 W sonicator with VCX Series ultrasonic processor—20 kHz frequency. The total duration of the ultrasonic energy cycle was 10 minutes, in times of 20 seconds applied and then 20 seconds stopped (until the end of the 10 minutes, in order to avoid heating the solution), this cycle was chosen based on tests carried out in the laboratory of the work team.

Once the sonication cycle was finished and the activating solution reached stable temperature, a single solution was formed from the two previous solutions (±140 mL), which was manually and controlled and added to the metakaolin (145.3 g). The whole paste was mixed homogeneously in a mechanical stirrer for 5 minutes. Finally, the paste was poured into molds and placed in the oven at 65 °C for 24 hours. The samples were named based on the content of NWSC or NT incorporated. The results were subjected to statistical analysis using Past version 2.17 software.

2.1 Tests

2.1.1 Isothermal conduction calorimetry

The heat flow recording was done by means of the Thermometric AB of TAM Air (TA instruments). The samples were prepared with mixers incorporated to the device, being able to record the heat flow after the contact of the solid material with the activating solution. In this way, the reactions were monitored in four mixtures: the reference (R) (without nanomaterial), with NWSC (R + 0.20% SiC), with NT (R + 0.50% NT) and with joint nanomaterials (R + 0.20% SiC + 0.40% NT), at the same temperature of 65 °C for 24 hours (1,140 minutes). However, after 150 minutes from the start of the test, stability in the heat flux was recorded and based on this time the graph was cut for thermal analysis.

2.1.2 Compressive strength

For the resistance test, an Instron press model 5,569 was used, with a speed rate of 5,000 N/min. For the compression test, the samples had a cylindrical format of 20 × 40 mm. Three samples per age were manufactured.

2.1.3 Bending strength

For the strength test, an Instron press model 5,569 was used, with a speed rate of 5,000 N/min, applied in the center of the specimen, which was supported at two points spaced at 6 cm. For the flexural test the format was prismatic 20 × 20 × 100 mm. Three specimens per age were manufactured.

2.1.4 Density and Young's Modulus

To determine Young's modulus, the impulse natural frequency technique was applied, using the ACTP Sonelastic version 2.8 equipment. Based on the same equipment, the bulk density of the samples could be estimated. The samples were used for flexural testing, 3 samples per age.

3. Results and discussion

3.1 Isothermal conduction calorimetry

The reactions associated with geopolymerization are characterized by being partially exothermic and there are many factors that affect it: concentrations of Si, Al and Na in the precursor materials and in the activating solution; the presence of water; the presence of additives and additions or even the cure temperature, as indicated by Rodriguez *et al.*[3], Abbasi *et al.*[15], Bigno *et al.*[39], and Ma *et al.*[40]. **Figure 4** shows the behavior for all the samples. A

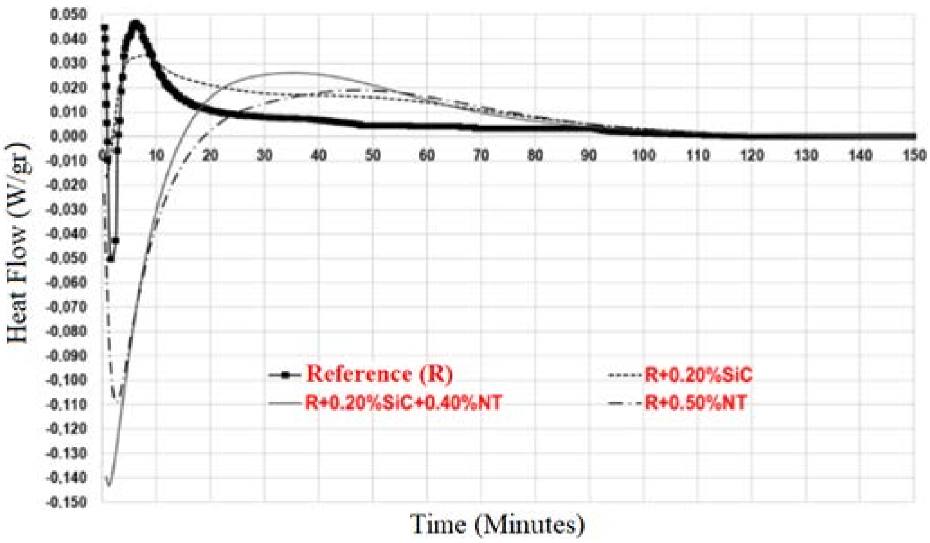

Figure 4. Evolution of the heat flux of the different pastes.

pronounced exothermic peak associated with the initial dissolution of metakaolin (along the vertical axis) was observed, which was not recorded for all samples. A subsequent short endothermic period was associated with the need for the system to enter into equilibrium with the environment created inside the calorimeter. In this way, the sample is forced to absorb the heat until it begins to emit a heat typical of geopolymerization reactions. Subsequently, a third peak with variations in amplitude and length, specific to each sample, but exothermic in nature. Finally, the samples stabilize in an estimated time of 110 minutes.

For the paste containing SiC the highest peak of heat flux occurs at the same time as the reference, however, the form of energy emission is slightly different for these samples. On the other hand, the behavior of the emission rate of this energy is completely different when we refer to the pastes incorporating NT. The behavior of the heat flow modifies their intensities and durations.

This would indicate that the incorporation of NT in geopolymeric matrices, with thermal cure, stimulates the dissipation of the heat generated by the reactions of geopolymerization and the environment, as shown in **Figure 5**.

This phenomenon may enter into discussion with that indicated by Ma and collaborators[41], who with the addition of NT recorded the acceleration of the reaction process of alkaline activated materials, when 1% by mass was added. Further comments on this subject are made below.

Studies analyzing this parameter in geopolymeric matrices are scarce. However, this behavior is also representative in cementitious matrices: the presence of titanium nanoparticles results in the acceleration of hydration reactions and increase of total heat[42–44]. For this study, a reduction of up to 95.87% was obtained in the first 3 hours, when 0.50% of NT is incorporated in the sample.

In the system incorporating NWSC this development is not accentuated, but a reduction of up to 24.24% can be obtained for the first 3 hours, compared to the reference.

Even with the total heat differences in the samples, no hardening process was observed during their preparation.

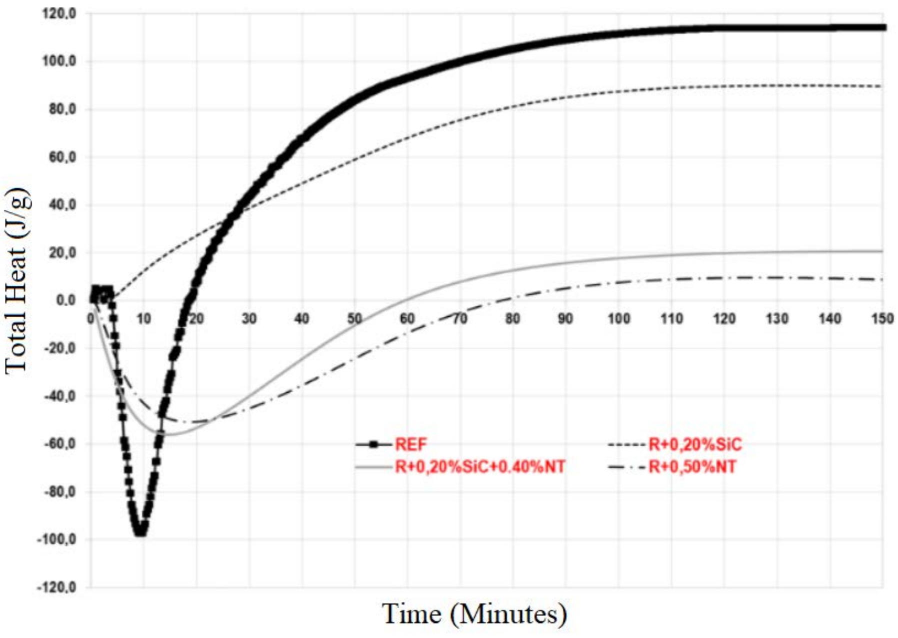

Figure 5. Total heat behavior of the different pastes.

3.2 Mechanical resistors

In general, when SiC nanowhiskers are added, the compressive strength has a tendency to increase, as can be seen in **Figure 6**.

The statistical analysis identified that, after 14 days, only the addition of SiC produces a significant difference in the matrix, generating the greatest increase of 28.80% in resistance when compared to

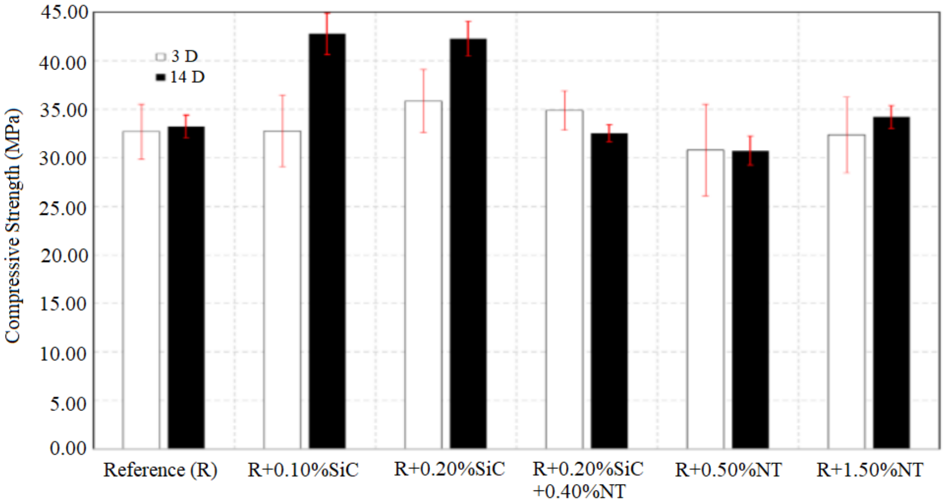

Figure 6. Results of the compressive strength of the different pastes.

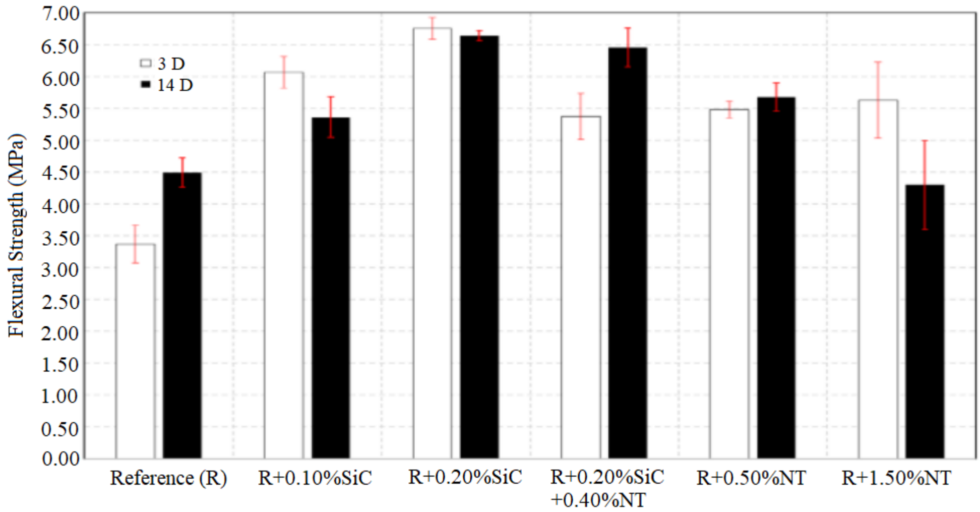

Figure 7. Results of the flexural strength of the different pastes.

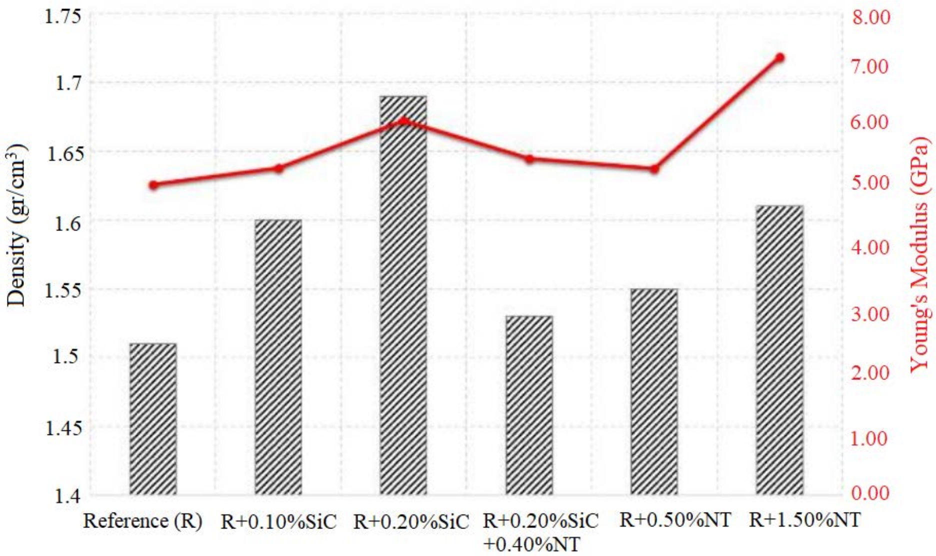

Figure 8. Young's modulus of the samples.

the reference.

As reported by Yuan *et al.*[45], the addition of SiC in the form of fibers up to 2% by volume contributes to a 36.70% increase in compressive strength. However, in the form of whiskers[46] records decrease in strength when they are incorporated more than 5% by mass. Both using metakaolin-based geopolymeric matrix This last author indicates that, in the form of particles, SiC helps in the filling effect, registering better packing than whiskers and, therefore, contribute considerably to the compressive strength, with an increase of up to 102% in the matrix.

In view of this confusion about the effects of incorporating SiC in geopolymeric matrices, it is indicated that, for the case of Portland cement-based matrices[36,47] the presence of SiC in the nanomaterial form contributes positively to the compressive strength. The amount and format will define the contribution to the type of strength.

NT particles, on the contrary, produced a reduction of up to 7.43% in compressive strength or no difference with the reference matrix. The higher percentage of NT in this study fails to produce a significant difference with the reference matrix, however, higher percentages (5%) of NT used by Zhang *et al.*[43] produced relevant increases from early ages (±22%). This probably indicates that the use of higher percentages will contribute to the increase of this property.

In the case of the flexural strength results shown in **Figure 7**, an increase in strength is recorded for any amount of added nanomaterial, except when 1.50% of NT is added. The increases are between 80.02% (with addition of 0.10% SiC) and 100.49% (with addition of 0.20% SiC), in the first 3 days. The highest resistance with the addition of 0.50% NT results in 62.71% increase in the same time. But all of them decrease after 14 days.

From the above, it can be inferred that the addition of SiC nanowhiskers contributes simultaneously to the improvement of compressive and flexural strength. Whereas, the addition of NT does not contribute significantly to the simultaneous improvement of the strengths. The additions cause a large differential at late ages when it comes to compressive strength[48].

Interrelating the calorimetry and resistance profiles, it can be stated that NWSCs would not be modifying the geopolymerization process, by virtue of their shape, they would be acting as nanofibers that allow the transmission of stresses. However, NTs in the alkaline environment and thermal cure conditions, rapidly interact with the OH group that dissolves the precursor material[49], slowing down this polymeric reorganization process. This would lead to the formation of fewer polymeric chains compared to the reference. Considering that the calorimetry was performed during the first 24 hours and the first resistance evaluation took place after 3 days, a stability of chain formation could actually be reached after 24 hours, showing stability or slight reduction.

Figure 8 shows the density and Young's modulus results for the different samples. Statistically, the density values do not represent significant differences for any nanomaterial addition, however, the higher nanomaterial additions cause a significant difference in the Young's modulus of the reference matrix. Confirming the indication of Chen *et al.*[44], NTs eventually produce a filling effect within the cementitious matrix. Such a possibility would induce to use higher proportions to evaluate their incidence on the mechanical strength and still contribute with the photocatalytic effect on cementitious and geopolymeric matrices[33].

4. Conclusions

The influence of adding TiO_2 nanoparticles and SiC nanowhiskers was recorded by different experimental tests and it could be concluded that: These nanomaterials are able to modify the reaction kinetics, the mechanical performance of the reference matrix and some physical properties. Regarding the reaction kinetics, the additions modified the heat emission rate, in what seems to be retarding and dissipative effect specified for NTs. When it comes to mechanical performance, SiC nanowhiskers simultaneously increase the compressive and flexural strength of the geopolymer matrix. However, TiO_2 nanoparticles may be causing a partial increase on the evaluated strengths. In the case of density and modulus of elasticity, with the additions only an increase in modulus was obtained, while density was not altered.

Conflict of interest

The authors declared no conflict of interest.

Acknowledgments

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