Volume 10, Number 6 (December 2017) p. 1174 – 1181 • ISSN 1983-4195
http://dx.doi.org/10.1590/S1983-41952017000600003

REVISTA IBRACON DE ESTRUTURAS E MATERIAIS
IBRACON STRUCTURES AND MATERIALS JOURNAL

Effect of NaOH concentration and curing regime on geopolymer

Efeito da concentração de NaOH e de regimes de cura em geopolímeros

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Abstract

The effect of alkali concentration and curing temperature regime on fly ash-based geopolymer pastes was investigated in this study by using NaOH solutions. Prismatic specimens were molded, cured at 65 °C and 85 °C and submitted to flexural and compressive strength tests. Unreacted fly ash and geopolymers were characterized by X-ray diffraction and thermogravimetric analysis. In general, the mechanical strength was enhanced by increasing the molar concentration and the curing temperature. This trend was confirmed by thermogravimetric data. However, for a lower amount of NaOH there were no significant differences between the strength results. The mixture with the highest strength was obtained with the 16 M NaOH solution and curing temperature of 85 °C, which resulted in flexural strength of 4.20 MPa, compressive strength of 21.35 MPa and also the highest weight loss of 9.89%.

Keywords: alkali-activated binder, strength, x-ray diffraction, thermal analysis.

Resumo

O efeito da concentração alcalina e da temperatura de cura em pastas geopoliméricas a base de cinza volante foi avaliada utilizando soluções de hidróxido de sódio. Corpos de prova prismáticos foram moldados e curados em temperaturas de 65°C e 85°C, sendo posteriormente submetidos aos ensaios de resistência à flexão e compressão. Foi realizada a caracterização microestrutural da cinza volante utilizada e das pastas produzidas, a partir de análises de difratometria de raios X e termogravimetria. Em geral, a resistência à compressão foi favorecida pelo aumento da concentração molar e da temperatura de cura. Esta tendência foi confirmada nos dados termogravimétricos. No entanto, para um pequeno teor de NaOH, não foram obtidas diferenças significativas entre os resultados de resistência. A mistura com maior resistência foi produzida com uma solução de NaOH com concentração de 16 M e temperatura de cura de 85°C, resultando em resistência à flexão de 4,20 MPa, resistência à compressão de 21,35 MPa e perda de peso máxima de 9,89%.

Palavras-chave: ligante álcali-ativado; resistência; difração de raios X; análise térmica.

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Received: 21 Mar 2016 • Accepted: 26 Apr 2017 • Available Online: 11 Dec 2017

1. Introduction

Portland cement is the traditional binder used in the production of concretes and mortars. However, during production a large amount of carbon dioxide (CO_2) is released resulting in significant environmental impact. Besides the CO_2 emissions, cement production is an energy-intensive process. Thus, while some research has been focused on the improvement of Portland cement concrete technology, other studies are needed to find new alternative binders, which require less energy and have a lower environmental impact [1].

Considering the importance of developing sustainable technologies, one notable option being studied is the geopolymer obtained from the polycondensation of aluminosilicate minerals in a highly alkaline environment. It is well known that using this process it is possible to transform vitreous structures into compact materials with binder properties [1,2].

In general, industrial wastes are used as sources of aluminosilicate minerals, particularly class F fly ash from thermoelectric plants, which has been used in the production of Portland cement since it is able to reduce the consumption of clinker and fossil fuels and hence the emissions of CO_2. This is an environmentally friendly solution that contributes to the reduction of the carbon footprint of concrete [3].

The class F fly ash is chemically composed of acids oxides, such as silica (SiO_2), aluminum oxide (Al_2O_3) and hematite (Fe_2O_3), able to react with alkalis from activator. Especially in the case of silicate-based activator, the sum of reactive oxides above 93% is recommended to high strength under lower curing regime [4].

A SiO_2 content between 40% to 50% is suggested by [5], since silica is the main component in the geopolymeric precursor formation. Under highly alkaline conditions, this oxide yields polymeric Si-O-Al bonds, forming a zeolitic-type gel that contributes to the mechanical properties [5].

With respect to lime content, it is still inconclusive the true contribution on strength [5]. It is known that this oxide can react with silicates and aluminates, resulting in hydrated products found in Portland cement mixes [6]. However, percentage above 5% [5,7] may interferes negatively in polymerization [8].

The alkaline activators are sodium or potassium hydroxide solutions [8-14], stand-alone or combined with the respective silicate [2, 6, 8,15-19]. In the latter case, the reactions tend to be faster and benefit the strength development. Its dosage is associated to the atomic ratio Si:Al from precursor, so that a mass ratio of alkali metal to silicate in the range of 1.7 to 2.0 is recommended for fly ash [7].

Besides the nature of the raw material, several factors influence the development of geopolymers, for example, the curing temperature regime [2, 8, 20], the alkaline solution-to-fly ash ratio [2,11, 21] and the solution concentration in terms of molarity [8,11, 22].

As examples, the following studies carried out on fly ash geopolymer based and NaOH alkaline solution can be mentioned. Geopolymers cured during 24h in oven at 65°C, with 12 M molar concentration, presented initial compressive strength of 21.2 MPa and 17.3 MPa, to an alkaline solution-fly ash ratio of 0.25 and 0.30 respectively [2]. For the same solution-to-fly ash ratio of 0.30, but 18 M NaOH solution and oven-cure at 85°C during 20 h, developed initial compressive strength of almost 60 MPa [20]. Finally, to an 8 M concentration, 0.40 alkaline solution-to-fly ash ratio and oven-cure at 85°C, the results for compressive strength after 8 h, 20 h, 28 days and 60 days were practically constant at 20 MPa [22].

Based on the literature, the effect of various NaOH molarities (8 M, 12 M, 16 M) and curing temperatures (65 °C and 85 °C) on mechanical strength of fly ash geopolymer pastes were tested.

1.1 Justification

There is a synergy between the parameters studied that can be better understand by the mineralogical and microstructural characterization of raw materials and final products [23], which are determined by X-ray diffraction or thermogravimetric analysis [24-26]. Most of studies on the geopolymer development report only compressive strength and x-ray diffraction results. This study contributes presenting also flexural strength and thermogravimetric data. Based on the findings of this research, a relationship between the mechanical properties, the X-ray diffractometry and thermogravimetry data was observed and this is reported in the paper.

2. Materials and experimental program

2.1 Materials

A Class F fly ash from the Jorge Lacerda Thermoelectric Plant, Santa Catarina (Brazil), was tested. The ash was dried, milled and used as the aluminosilicate source. The chemical composition of fly ash determined by X-Ray Fluorescence (XRF) was presented in Table 1. The sum of reactive oxides (SiO_2, Al_2O_3 and Fe_2O_3) was 89.2% and the molar ratio was 2.8:1 (SiO_2:Al_2O_3). According to molar ratio exposed by [15], the geopolymer resulted is classified as a polysialate and can be used as low CO_2 cement.

The physical characterization consisted on the determination of density and fineness. The fly ash has a density of 2492 kg/m^3, determined by helium pycnometer. Finesses was investigated in terms of particle size analysis measured by the Microtrac s3500 laser diffraction particle size. The raw fly ash had presented a coarse particle size distribution. To improve its reactivity, was grinded in an AMEF ball mill during 550 minutes, as detailed in [27]. A comparison between milled and raw fly ash was presented on Figures 1 and 2 and the mean particle size (d50) was reduced from 90 μm to 17 μm.

Three activator solutions (8 M, 12 M and 16 M) were prepared through the dissolution of sodium hydroxide (NaOH pellets, 98% purity, Unipar Carbocloro, Brazil) in distilled water one day prior to their use due to heat generation from exothermic reaction. To prepare 500 mL of solution, it was weighted 160 g, 240 g and 320 g, respectively.

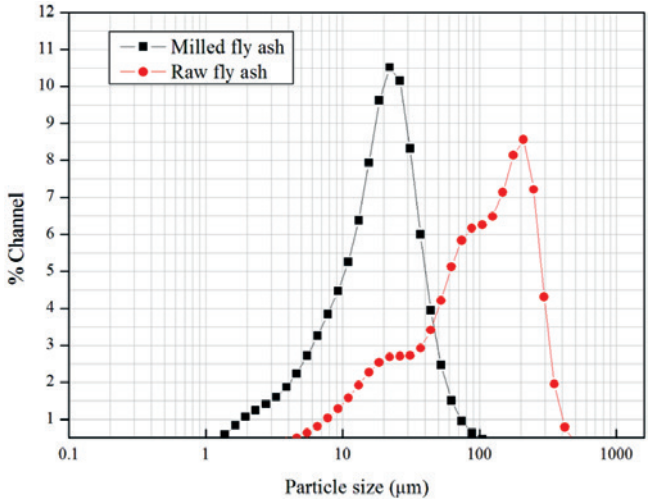

Figure 1
Particle size distribution of milled and raw fly ash, in terms of % channel

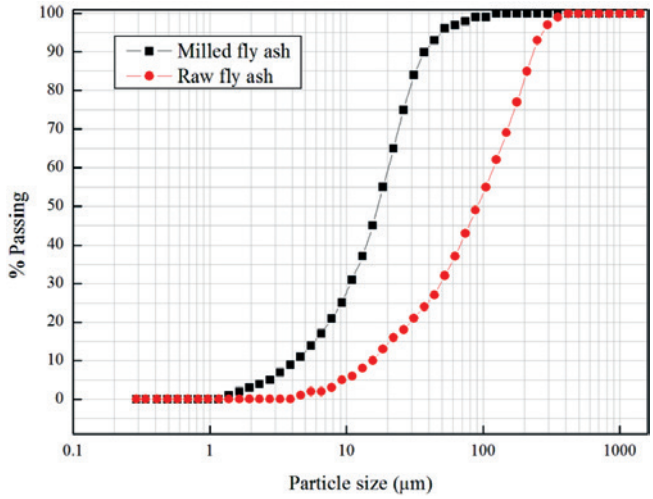

Figure 2
Particle size distribution of milled and raw fly ash, in terms of % passing

2.2 Mixing method and sample preparation

Pastes were produced by mixing the alkaline solutions previously prepared with the fly ash, in an activator solution-to-fly ash ratio of 0.55, for 5 minutes in a mechanical stirrer at 733 RPM. Table 2 presented the mix design of different mixtures.

The fresh pastes were poured into a 40 x 40 x 160 mm metallic prismatic mold. The specimens were cured in oven for 22 hours, applying two different curing regimes (65°C and 85°C). Additionally, a glass plate to prevent water evaporation covered them. Three samples were prepared for each composition. To facilitate the demolding process the metallic walls were enveloped with layers of plastic wrap (PVC film).

The hardened pastes were kept covered in air chamber for 27 days at 23 ± 1 °C and relative humidity > 95%. There is some concern about carbonation process in alkali-activated fly ash, especially when NaOH solution is used. It has been studied the lowering of pH due to process reduces the ash activation rate and consequently the strength developments. However, this can be prevented trough high relative humidity [1], as is practiced in air chambers.

Table 1
Main chemical composition of fly ash (% wt)

Component	Composition (%)
SiO_2	64,1
Al_2O_3	22,9
Fe_2O_3	2,2
K_2O	2,0
L.O.I.[a]	1,6
CaO	1,4
TiO_2	1,4
MgO	0,6
Na_2O	0,2
SO_3	0,2

[a] L.O.I. = Loss on ignition

2.3 Items of investigation

At the age of 28 days, prismatic specimens were tested for flexural and compressive strength, according to the standard BS EN 196-1:2005 [28]. Flexural strength was determined using three-point-bending test at a loading rate of (50±10) N/s, in an Instron 5569 press. It was calculated from Equation 1, where: R_f is the flexural strength (in MPa), F_f is the load applied to the middle of the prism at fracture (in N), l is the distance between the supports (in mm) and b is the side of the square section of the prism (in mm).

$$R_f = \frac{1.5.F_f.l}{b^3} \quad (1)$$

The resulted specimens (the both halves from the flexural specimens) were used for determination of compressive strength, in a Shimadzu 2000 kN universal press. The load was increased smoothly at the rate of (2400±200) N/s until fracture and the compressive strength was calculated from Equation 2, where R_c is the compressive strength (in MPa), F_c is the maximum load at fracture (in N) and 1600 is the area of the auxiliary plates (40 mm x 40 mm).

$$R_c = \frac{F_c}{1600} \quad (2)$$

Both fly ash and geopolymers were characterized by X-ray diffraction and thermogravimetric analysis. Samples of the hardened pastes were collected after the mechanical tests and stored in liquid nitrogen until the characterization tests were carried out. Prior to analysis, they were submitted to a freeze-drying process and then crushed.

Table 2
The sample compositions

Mixture ID	Fly ash (g)	Alkaline activator		
		Solution (g)	Content of solids (g)[a]	Content of water (g)[a]
M8	1,120.0	616.0	159.8	456.2
M12	1076.6	592.2	214.4	377.7
M16	1,103.7	607.1	275.7	331.3

[a] The content of solids and water was calculated based on [8] methodology.

The mineralogical characterization by X-ray diffraction (XRD) was carried out in a Rigaku Miniflex II powder diffractometer, under the following conditions: Copper x-ray tube (λ = 1.5418 Å), U = 30 kV, I = 15 mA. Scaning betwwen 5° to 60° 2θ, with a step of 0.05° and 1 s step count time.
Termogravimetry (TG) was conducted in a TA Instruments SDT-600 Simultaneous Thermal Analyzer. Samples were heated from ambient temperature to 1000°C at 20°C/min, in a nitrogen environment at 200 mL/min purge rate.

3. Results and discussions

3.1 Mechanical strength

The flexural and compressive strength of the geopolymers were summarized in Figs. 3 and 4, respectively. The error bars plotted above and below the mean values represent ± one standard deviation for the respective specimen. The results were evaluated through multifactorial analysis of variance, in significance level of 0.05, and it was concluded that both factors influence the mechanical strength. In terms of average values, strength was improved by increasing the solution molarity and curing temperature. The best results were achieved with the paste prepared with the 16 M alkaline solution and a curing temperature of 85 °C, which had a flexural strength of 4.20 MPa and compressive strength of 21.35 MPa.
The flexural strength results were mainly influenced by the NaOH content, while the use of the higher curing temperature had a more significant effect only in the case of pastes prepared with 16 M NaOH. It is known that the alkali concentration determined by the molar concentration is an important factor in relation to geopolymerization since it is responsible for dissolving the glassy structure of the fly ash, activating the silicon and aluminum to form the precursor gel [29,30]. Higher leaching of silica and alumina [31] promotes better binding between the solid particles in the final structure of the system [32] and it contributes to the mechanical properties of the hardened geopolymer [33].
However, the compressive strength was mainly influenced by the curing regime parameter. There were no statistically significant differences (significance level of 0.05), between the geopolymers prepared with solutions of different molarities cured at 65 °C.
Once there is a relationship between compressive strength and degree of reaction, it could be assumed that the geopolymerization reaction was incomplete, since a higher curing temperature is required for more efficient alkaline activation [34] and therefore to produce a better oriented molecular structure [35]. Nevertheless, on comparing the pastes prepared with the 8 M NaOH solution, it was noted that samples with a curing temperature of 85 °C had lower compressive strength than those obtained at 65 °C. Considering a longer curing time at higher temperatures might have harmful effects on the structure, due to loss of structural water resulting in cracking [36], there appears to be an ideal curing temperature for pastes prepared with the 8 M concentration.

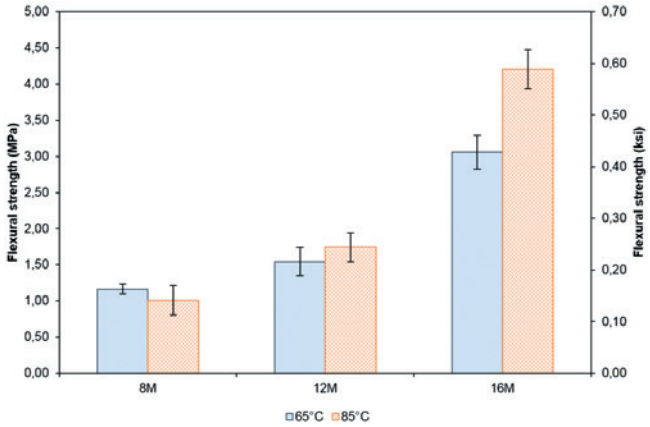

Figure 3
Flexural strength of geopolymers

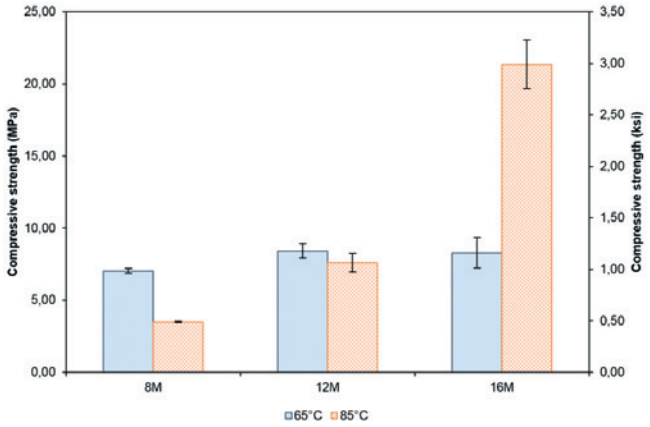

Figure 4
Compressive strength of geopolymer

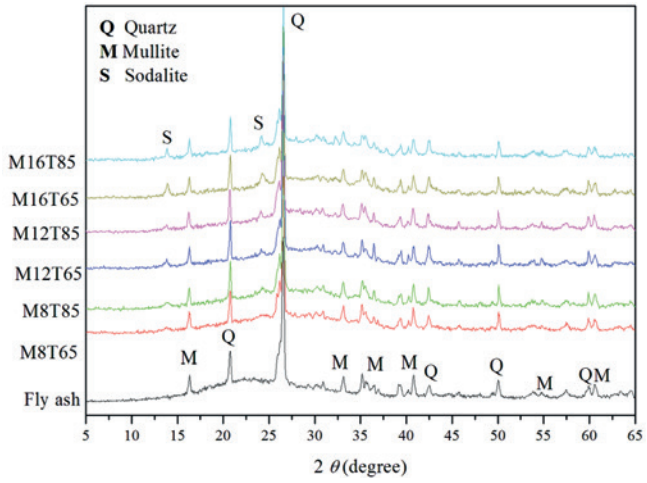

Figure 5
Diffraction patterns of fly ash and geopolymers

In the case of pastes prepared with the 16 M solution at a curing temperature of 65 °C, the compressive strength was lower than that observed applying a curing temperature of 85 °C. According to [37], an excess of OH- requires a higher curing temperature or a longer time to eliminate the excess water, due to the viscosity of the solution. Therefore, a higher curing temperature was necessary. The curing temperature affects the structural transition from the amorphous to the crystalline state during polycondensation [38] and thus for synthesis carried out at higher temperatures it is possible to achieve better results in terms of the strength until a threshold value [29,39].

The curing temperature also affects the reaction kinetics [2,8,20] due to an increase in the dissolution and polycondensation rates [40] and this is an important factor in the optimization of polymerization reactions [1]. However, after 28 days the degrees of geopolymerization of the different mixtures were similar, so the quality of the reaction product is the predominant factor rather than the curing temperature. This may explain the finding that the pastes with a NaOH concentration of 12 M treated using different regimes showed similar results for the average strength.

3.2 X-ray diffraction

The X-ray diffraction data collected for the unreacted fly ash and geopolymer pastes samples were shown in Figure 5 and patterns were labeled according to mix and curing parameters, so if the label is M8-T65 it is mean the paste were prepared with an 8 M solution and were cured at 65°C.

The unreacted fly ash was comprised most of a vitreous phase (with a halo registered between 17° and 32° 2θ) and some minor crystalline phases, such as quartz and mullite. The geopolymer diffraction pattern showed some differences in relation to the raw fly ash. For the geopolymer samples the halo associated with the silica peak was shifted to slightly higher angles (recorded between 22° and 38°) due to the formation of an aluminosilicate gel, the main product of geopolymerization [29, 41], which is highly disordered [33].

With the exception of the sample M8-T65, other pastes indicated the aluminosilicate gel, a new zeolitic phase identified as sodalite, the compound of the type Na2O-Al2O3- SiO2 with low and scattered bands responsible for mechanical strength. The hardened geopolymers contains quartz and mullite, which exist in the fly ash, but appeared in lower intensity. It indicates the presentation of products in a state of low-ordered crystalline structure [4].

The similar XRD patterns for the original fly ash and the geopolymer paste sample M18-T65 indicates undissolved fly ash particles [32] and lower production of the geopolymer [30].

Under better conditions for polycondensation (higher NaOH concentration and curing temperature), new phases were detected, suggesting high levels of geopolymerization and strength [30].

Finally, it is possible to relate the zeolitic phase with strength, once the better mechanical strengths were observed for geopolymers

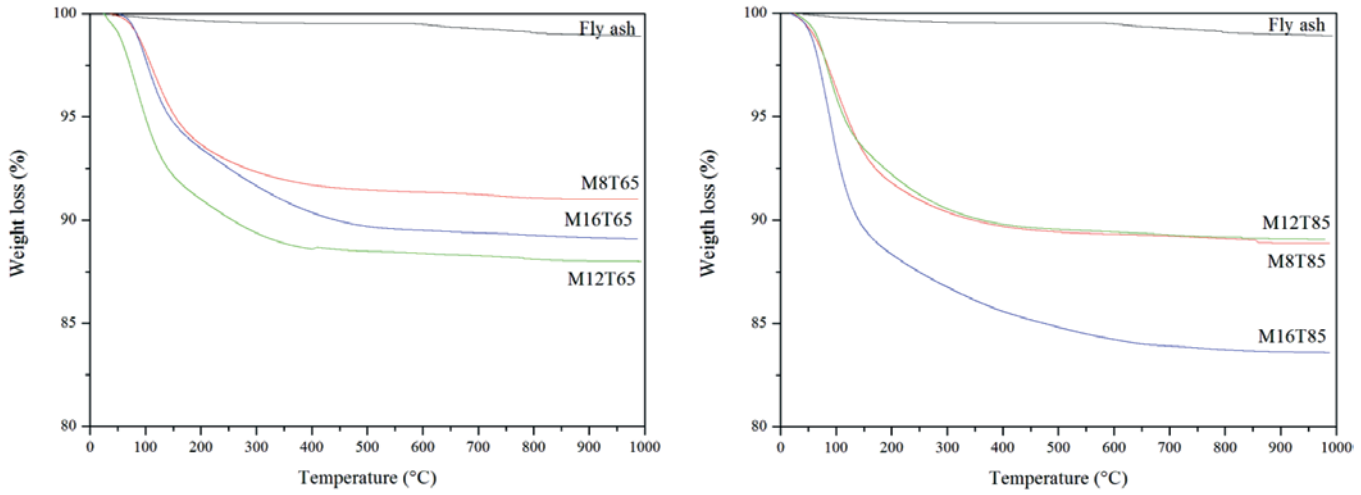

Figure 6
Thermograms (TG) of fly ash and geopolymers

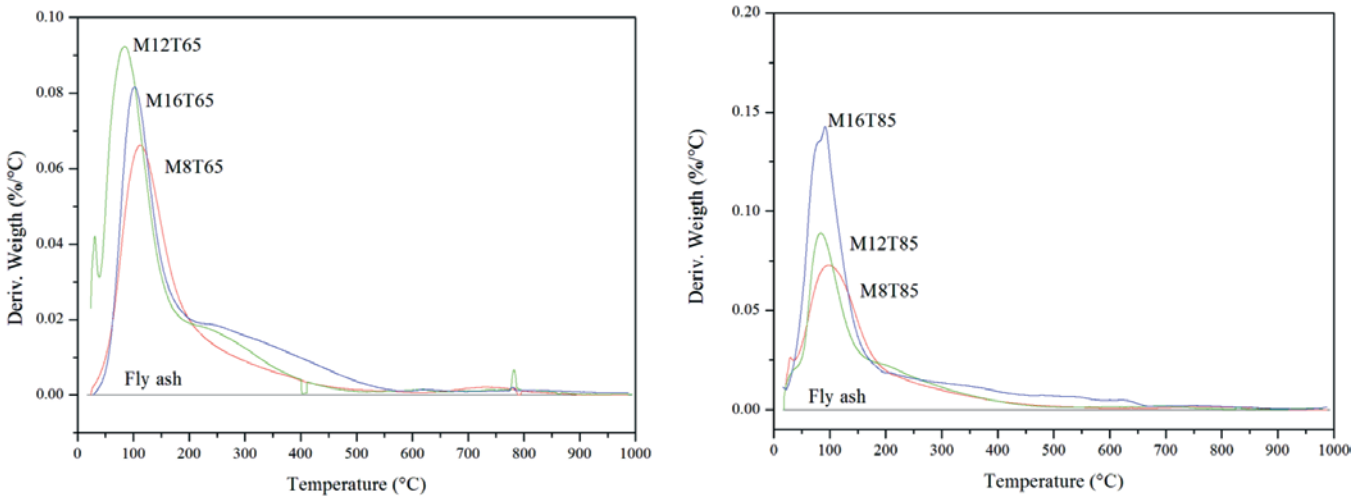

Figure 7
TThermograms (dTG) of fly ash and geopolymers

with a better-defined peak for sodalite. Highlighting M16-T85 sample that presented flexural strength of 4.20 MPa and compressive strength of 21.35 MPa.

3.3 Thermogravimetry

According to Davidovits [42], there are three types of water in a hardened geopolymer paste. The water released during heating is classified as physically bonded water, chemically bonded water and water formed from hydroxyl groups as result of polycondensation [39]. During the thermogravimetry test, the weight loss is mainly associated with the evaporation of free water [43]. Mechanical strength inversely relates with the amount of chemically bonded water, known as zeolitic water [39].

The thermograms (TG and DTG) for the geopolymers and unreacted fly ash were presented in Figs. 6 and 7. There was progressive weight loss until 200 °C, up to 13%, due to the evaporation of free water or condensation of hydroxyl groups [44,45]. At temperatures above 800°C the geopolymer was totally dehydrated and since no peaks were observed, no hydrates were present [46].

In fact, the main peak identified in differential thermogravimetric curves presents a minimum between 80°C to 111°C. The increases in intensity of this peak indicates a higher extent of geopolymeric gel produced, in accordance to higher compressive strength in pastes [47], in a special manner to geopolymer produced with 12 M and cured at 65°C, and a 16 M molar concentration and curing regime of 85°C. There were not significant peaks beyond related to free or condensation water, fact also observed by [43, 45, 46].

4. Conclusions

The results obtained in this study aid a better understanding of the geopolymerization process. The production of geopolymers with high mechanical strength was achieved by determining the ideal mixing composition (NaOH solution molarity) and curing regime (oven temperature). In general, the flexural and compressive strengths were better for higher curing temperature and solution molarity. Nevertheless, when pastes were cured at 65 °C the differences in the strength due to the NaOH content originating from the alkaline solution were not expressive. In addition, for a higher solution molarity it was observed that an increase in the curing temperature is required to achieve better results and trends according to the thermogravimetric data.

The use of the X-ray diffraction technique provided evidence of the geopolymerization process, through changes in the diffractograms according to the shift in the amorphous halo and the new phase identified as a zeolitic-type.

Likewise, TG/DTG analysis showed, by the increased intensity in the main peak, a relation between weight loss due to condensation water associated to gel formation and the compressive strength.

The results for both XRD and TG analysis reinforced the idea that the strength of a fly ash-based geopolymer paste is dependent on the synergy between the NaOH concentration and the curing temperature. Applying the same curing temperature (85°C) and increasing the solution molarity (16M) led to higher flexural and compressive strengths, as well as the formation of geopolymeric gel and an increase in the weight loss. However, for lower temperature (65°C), it is not indicated to exceed the 12 M molar concentration, since it is seems necessary to submit pastes in a longer curing regime to promote geopolymerization.

5. Acknowledgements

The authors gratefully acknowledge the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) and the Fundação de Amparo à Pesquisa e Inovação do Estado de Santa Catarina (FAPESC) for providing the financial support for this research, and the Tractebel Energia for donating the fly ash.

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