

## Adhesion of geopolymer bonded steel plates

Kelly Cristiane Gomes, Sandro Marden Torres, Silvio de Barros  
*Nucleo de Estudos e Pesquisas em Materiais NEPEM-UFPB,  
Joao Pessoa/PB – Brazil*

Normando Perazzo Barbosa  
*Laboratorio de Ensaios de Materiais e Estruturas LABEME-UFPB,  
Joao Pessoa/PB – Brazil*

### Abstract

Known since the 70's, geopolymers are synthetic materials of acknowledged interesting chemical, mechanical and thermal performance. As far as environmental issue is concerned, the use of alkali-activated industrial by-product precursors and the near-room casting temperature (around 60°C) can be seen as an advantageous choice for many applications such as in thermo-mechanical efficient steel coatings. Whereas most epoxy resin would char at temperatures above 300°C, geopolymers can uphold good mechanical performance in temperatures as high as 1000°C. Although they have mostly been studied for coatings, the number of studies on their adhesion mechanisms is considerably less than those regarding its other properties. The main thrust of this work is to evaluate the binding potential of alkali-activated clay-based waste in steel bonded plates. The adhesion of steel plates (40x160x5mm) was evaluated using three clay-based wastes and metakaolin (MK) as precursors, provided by X-Ray fluorescence (XRF) analyses. Epoxy compound (EC) was used as a reference binder. The best result was found for the waste systems, which iron content is extremely higher than the other precursors, indicating a better adhesion performance of this system, when compared to the widely used epoxy compound.

Keywords: adhesion, geopolymer, mechanical tests.

### 1 Introduction

The use of adhesive bond technology has been increasingly applied for the development of composites and laminate structures. This can be attributed to (i) the flexibility of the application of such materials which allows their use in several different geometries; (ii) better stress distribution of the bound parts when compared to rivetting and screwing and (iii) the inexistence of heat affected zone when compared to welding. The use of adhesives can also represent an advantageous choice when production interruption needs to be avoided. For instance, epoxic resins have been employed for repairing oil and gas pipes in the oilwell industry [1, 2]. Also, it is estimated that approximately five kilograms of

adhesive materials are used to joint some components of a popular car [3]. However, the mechanisms and durability of adhesion are very complex, therefore, demanding a broad spectrum of research [4]. Most adhesives are polymer based such as epoxy. Epoxic resins are known to have excellent mechanical properties. Nonetheless, its application is often limited to low temperature below 400°C.

Because most polymeric resins would char at temperatures above such threshold, there has been an increasing interest on other types of polymeric materials such as the geopolymers. These materials are found to uphold considerable mechanical properties in temperatures as high as 1000°C [5]. These inorganic polymers (geopolymers) were developed in France during the world oil crises of the 70s. These worldwide energy crises often help to bring up the quest for environmentally friendly engineering solutions, and the development of new materials become a paramount issue for the academics, the production sectors and governments [6]. One might consider that geopolymers can fit in the category of friendly materials. Firstly, their synthesis occur at a near-room casting temperature. Secondly, they can be obtained by precursors derived from by-products of mining and industrial activities.

Most materials used to produce geopolymers are aluminosilicate such as: (i) kaolinite containing clays and (ii) steel manufacturing wastes such as ground granulated blast furnace slag and fly ash. There are several other sources of aluminosilicate containing materials that have not been yet studied as geopolymer precursors, especially in places with low steel manufacturing industrial activities such as the Brazilian northeast. Nonetheless, this region contains important sources of clay minerals and other related by-products such as in the ceramic industry. Recent studies have aimed to evaluate the potential use of such types of wastes for the development of geopolymer. The results have shown interesting thermo-mechanical properties [7, 8].

The number of studies on the adhesion mechanisms of geopolymers are, nonetheless, considerably inferior when compared to studies on their syntheses and mechanical properties [9, 10]. The main thrust of this work is, therefore, to evaluate the binding potential of alkali-activated clay based mineral and industrial wastes as adhesive of steel bonded joints subjected to different temperatures.

## 2 Materials and methods

### 2.1 Materials

The chemical composition of the raw wastes obtained by X-Ray Fluorescence analyses is shown in Tab. 1, where:

- MK: Metakaolin was used as control, given the wide number of research in such material as geopolymer precursor.
- Waste A: Mineral waste from a local source.
- Waste B: Local industrial waste.
- Waste C: Mineral waste from a local source.

Table 1: Chemical composition of waste

|           | MK (%) | Waste A (%) | Waste B (%) | Waste C (%) |
|-----------|--------|-------------|-------------|-------------|
| $SiO_2$   | 62,96  | 51,18       | 62,25       | 61,87       |
| $Al_2O_3$ | 34,61  | 11,70       | 22,11       | 24,06       |
| $Fe_2O_3$ | 0,63   | 35,97       | 5,99        | 10,87       |
| $Na_2O$   | 0,07   | 0,00        | 1,06        | 0,00        |
| $K_2O$    | 1,34   | 0,02        | 1,24        | 0,19        |
| $CaO$     | 0,05   | 0,02        | 0,05        | 0,20        |

The oxide content of the activator is shown in Tab. 2.

Table 2: Chemical composition of activator

| $SiO_2$ (%) | $Na_2O$ (%) | $H_2O$ |
|-------------|-------------|--------|
| 36,25       | 16,63       | 47,12  |

The molar ratio composition of the mixes are shown in the Tab. 3. With the exception of the silica to alumina ratio, the other parameters were kept as close as possible, although such ratio differences can be expected given the differences in both the waste composition and the texture of the powders.

Table 3: Molar ratio of geopolymer

|         | $SiO_2/Al_2O_3$ | $H_2O/M_2O$ | $Al_2O_3/M_2O$ | $M_2O/SiO_2$ |
|---------|-----------------|-------------|----------------|--------------|
| MK      | 5,43            | 9,73        | 0,96           | 0,19         |
| Waste A | 9,33            | 9,76        | 1,19           | 0,09         |
| Waste B | 7,09            | 9,06        | 0,91           | 0,16         |
| Waste C | 6,66            | 9,76        | 0,98           | 0,15         |

## 2.2 Sample preparation

The steel plates (160x40x4 mm) surfaces were lapped and polished in order to keep an uniform metal substrate. The prospective bonding faces were washed with acetone and then rinsed with ethanol prior to bonding process. The adhesive materials were poured on the treated surfaces and both plates were pressed together by a mechanical device in order to keep the adhesive thickness of 0,5mm in all samples.

## 2.3 Curing regimes

In order to investigate the effect of the temperature on the adhesive properties, the bonded plates were subjected to four types of curing temperature regimes, as shown in Tab. 4. In the first, the bonded steel plates were subjected to a classical curing regime, which was used as control. In the second and followings, the samples were subjected to increasing temperatures and subsequently placed in a temperature and humidity controlled environment. The highest temperature level was 400°C in order to assess the binding properties of the geopolymer in comparison with the epoxy adhesive.

Table 4: Curing regimes

| Regime | 22°C(day) | 55°C(day) | 22°C(day) | 200°C(day) | 400°C(day) | 22°C(day) |
|--------|-----------|-----------|-----------|------------|------------|-----------|
| 01     | 01        | 01        | 05        | -          | -          | -         |
| 02     | 01        | 01        | 01        | 01         | -          | 05        |
| 03     | 01        | 01        | 01        | -          | 01         | 05        |

## 2.4 Crack propagations tests

Among several classical adhesion assessment techniques, the mixed-mode flexure test (MMF) is one of the simplest to be performed. In a MMF test, both propagation modes, shear and opening, can be evaluated at the same time, in order to provide a more realistic geometry and mode of loading as in actual structures. The tests were performed using a Shimadzu Servopulser machine. The load speed applied was 0.01 mm/sec and the displacements were continuously recorded in a built-in data acquisition system.

## 3 Results and discussions

### 3.1 Near-room temperature

Figure 1 shows the crack propagation profile of the waste systems under the MMF mode at curing regime 1. It can be seen that the waste A type adhesive reached the highest crack propagation load

level, followed by the industrial waste B and mineral waste C geopolymer type adhesives, respectively.

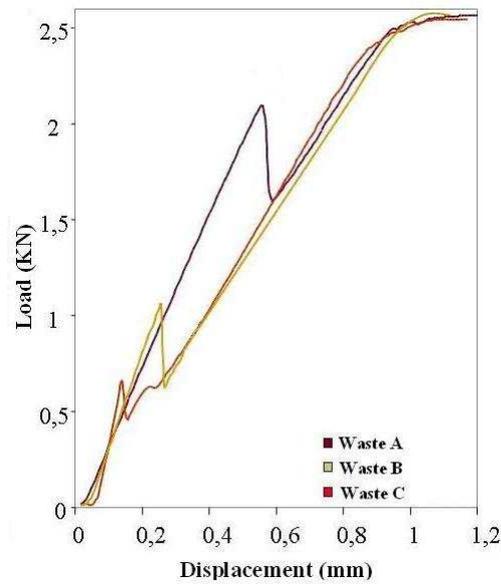


Figure 1: Crack propagation tests of curing regime 1.

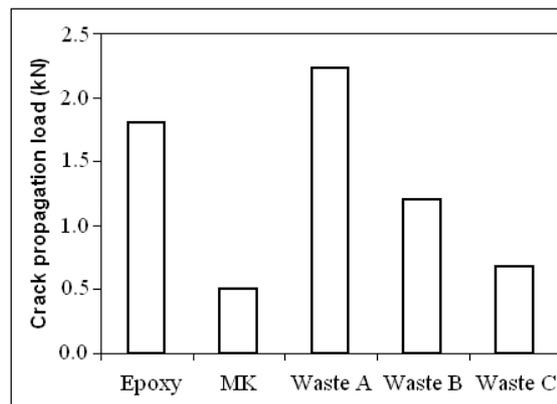


Figure 2: Crack propagation load of curing regime 1.

Figure 2 shows the crack load level of all five adhesive types tested at curing regime 1. In the epoxy system, the first propagation occurred at 1.8kN. Metakaolin system presented the lowest level, reaching around 0.5kN. When compared together, the obvious superiority of epoxy adhesive might explain the lack of studies on the adhesive properties of geopolymers at near room temperature. Indeed, metakaolin is the most widely spread precursor for this type of inorganic polymers. Also, it can be seen that all the other geopolymeric systems reached superior crack propagation load when compared to MK system alone. The mineral waste A reached the highest crack propagation load, slightly superior then the epoxy based system.

Although waste C system showed the lowest crack propagation strength, its load level can be considered equivalent to the MK one. This can be quite expected given the similar chemical composition of both MK and waste C (Tab. 1). The highest Al<sub>2</sub>O<sub>3</sub> content was observed in MK (34.61%), followed by Waste C (24.06%). Also, the Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> ratio of both MK (5.43) and waste C (6.66) are similar, slightly higher for waste C (Tab. 3). Experimental studies have proposed that the higher this parameter, the greater the strength of the bulk material. Consequently, it appears that such parameter is also related with the adhesive properties as well. The highest value of crack propagation load observed in waste A can be associated with its highest Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> ratio (9.33). The waste B system showed a rather higher crack propagation load then system C, although their composition are quite alike in terms of aluminium and silicon oxides.

The fact that the Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> ratio of waste B (7.09) be greater than the waste C (6.66) seems to corroborate to the view that such parameter also has a role on the adhesive properties of geopolymers.

The highest load value was observed for the waste A systems in which the lowest level of alumina was observed (11.70%). Although this level might suggest a low adhesive properties alone, this precursor presented the greatest value of Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> ratio (9.33). It is important to point out that iron content (35.97%) is extremely higher than the other precursors, nearly four times greater than the second highest (waste B). The results seem to indicate that the iron content might be another important parameter for the adhesive properties, having a positive influence on adhesion of steel systems. The greater the iron content, the higher the crack propagation load of the studied systems.

### 3.2 High temperature

The effect of the temperature on the crack propagation load can be observed on the Fig. 3. As expected for the epoxy based adhesive, its performance is limited to temperatures below 400°C. Although epoxy system reached the second highest crack propagation load level, this system failed without even the submission of the MMF test.

As far as the effect of temperature is concerned, it can be seen that the performance of the metakaolin geopolymer adhesive was quite constant up to 400°C. This result is in accordance with what is reported in the literature for the thermal-mechanical properties of such material, which shows geopolymers withstanding excellent mechanical properties at much higher temperatures [8, 11]

The effect of the temperature on the performance of the waste based geopolymers followed a similar trend of load decreament as observed for the epoxy system. Although greater crack propagation loads were obtained in all the waste based geopolymeric adhesives, the crack propagation load level decreased as the temperature increased. Nevertheless, they all retained similar load level when compared with

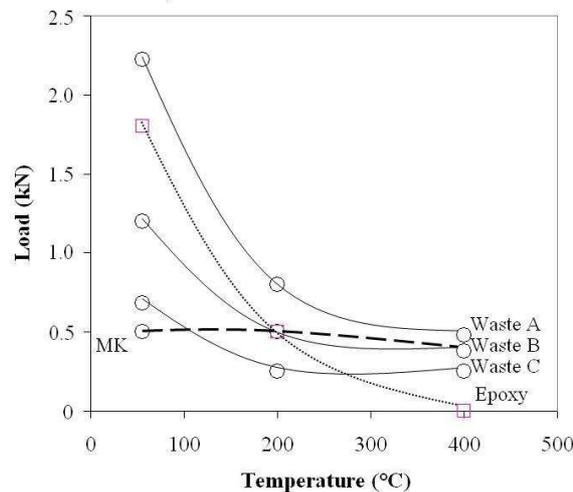


Figure 3: Crack propagation load versus increase in temperature.

the metakaolin geopolymer adhesive system. The load level decreament was more intense in Waste A, followed by Waste B and C, respectively. This decreament seems to be associated with the iron oxide content of the precursors, which decreased in the same order (see Tab. 1). This association might also explain why MK load level was kept almost constant up to the maximum tested temperature system as it had the least iron oxide content.

#### 4 Conclusions

The results can indicate that other alkali activated aluminosilicate materials had better performance then classical geopolymer precursor (MK), as far as adhesive properties are concerned. Two different mineral and one industrial wastes based geopolymers were successfully used as adhesive in steel plates with relative thermal efficiency. Geopolymers retained load under temperature as high as 400°C, giving superior performance when compared to an epoxy based adhesive. Although the metakaolinite geopolymer system was not significantly affected by temperature increase up to 400°C, all other adhesives presented a load decreament with temperature. The role of the Si:Al ratio on the adhesive properties of geopolymers seems to follow similar trend as occur for bulk compressive strength. The greater the Si:Al ratio of the precursor, the greater the crack propagation load in all systems. Nevertheless, it seems that the greater the iron oxide content, the greater the load decreament of all systems. Hence, care must be taken concerning the presence of gas generating reactions that might occur as in the case of some hydrated iron rich phases that might be present in some mineral and industrial wastes. This reaction can be responsible to reduce the mechanical efficiency of the steel plates bond.

### Responsibility notice

The author(s) is (are) the only responsible for the printed material included in this paper.

### References

- [1] Sampaio, R.F., Reis, J.M.L., Perrut, V.A. & Costa-Mattos, H.S., Epoxy rehabilitation of corroded steel pipelines with through-thickness damage. *Journal of Pipeline Engineering*, **7**, p. 1, 2007.
- [2] Stroganov, V.F., Strakhov, D.E., Alekseev, K.P. & Stroganov, I.V., Epoxy polymers in adhesive technologies of pipeline joint. *Polymer Science, Ser C*, **49(3)**, pp. 269–271, 2004.
- [3] Barquins, M. & Fadel, K., Adhesion et collage. *Decouverte*, **271**, pp. 31–46, 1999.
- [4] Dillard, D.A. & Pocius, A.V., The mechanics of adhesion. *Adhesion science and engineering*, **1**, 2002.
- [5] Davidovits, J., Properties of geopolymer cements. *Proceedings First International Conference on Alkaline Cements and Concretes*, Kiev, Ukraine, volume 131, 1994.
- [6] Davidovits, J., Geopolymers: inorganic polymeric new materials. *Journal of Thermal Analysis*, **37**, pp. 16–33, 1991.
- [7] Gomes, K.C., Nóbrega, A.F., Vieira, A.A.P., Torres, S.M., de Barros, S. & Barbosa, N.P., Ativação alcalina de resíduos de caulim. *International Conference on Non-Conventional Materials and Technologies - NOCMAT2007*, Alagoas, Brasil, 2007.
- [8] Barbosa, V.F.F. & Mackenzie, K.J.D., Thermal behaviour of inorganic geopolymers and composites derived from sodium polysialate. *Materials Research Bulletin*, **38**, pp. 319–331, 2002.
- [9] Latella, B.A., Perera, D.S., Escott, T.R. & Cassidy, D.J., Adhesion of glass to steel using a geopolymer. *Journal of Material Science*, **41**, pp. 1261–1264, 2006.
- [10] Wang, H., Li, H. & Yan, F., Synthesis and mechanical properties of metakaolinite-based geopolymer. *Colloids and Surfaces A: Physicochem Eng Aspects*, **268**, pp. 1–6, 2005.
- [11] Palomo, A., Varela, M.T.B., Granizo, M.T., Puertas, F., Varquez, T. & Grutzeck, M.W., Chemical stability of cementitious materials based on metakaolin. *Cement and Concrete Research*, **29**, pp. 997–1004, 1999.