Doctoral thesis proposal

Start : 2024

Thesis title: Contribution of paste-aggregate interfaces on the evaluation of geopolymer concrete tightness

Keywords: geopolymer concrete, natural aggregates, recycled aggregates, ITZ, cracking, damage, permeability, microstructural characterization

Laboratory: **GeM** - UMR CNRS 6183 - Institut de recherche en **G**énie civil **e**t **M**écanique
UTR (Thematic Research Unit) : DURPRO - Procédés et durabilité des matériaux et des structures

Location of the thesis: Saint-Nazaire – Nantes

Thesis director:	Co- Supervisors:
Full name: **CHOINSKA COLOMBEL Marta** Tel : +33 674012060 Mail : marta.choinska@univ-nantes.fr	1. Full name: **HILLOULIN Benoit** Mail : benoit.hilloulin@ec-nantes.fr 2. Full name: **RANAIVOMANANA Harifidy** Mail : harifidy.ranaivomanana@univ-nantes.fr

Description of the thesis

Context and objective

The study of the paste-aggregate interface, and in particular of the interfacial transition zone (ITZ), has interested many researchers, both for materials with hydraulic binders, and more recently for materials with geopolymer binders. The results obtained for geopolymer concretes are very promising and encouraging with regard to the recovery of these alternative construction materials, based on (local) industrial waste [Duzy et al., 2022; Duzy et al., 2021], therefore more environmentally friendly and thus less carbon-intensive than certain hydraulic concretes.

The results found in the literature show that the use of a geopolymer binder in concrete can improve the adhesion properties of aggregates, leading to a significantly stronger interfacial transition zone (ITZ) [Shi and Xie, 1998; Luo et al., 2021]. In addition, geopolymer binders are a priori acceptable materials to use in recycled aggregate concretes, thus improving their mechanical characteristics which are often inferior to natural aggregate concretes [Gopalakrishna and Dinakar, 2023].

The recycled aggregates mentioned here result from the transformation of inorganic materials previously used in construction. Most recycled aggregates are made up of two phases which come firstly from the granular skeleton of the old concrete and from its cementitious matrix [PN RECYBETON 2011; Yammine et al., 2020]. With a view to saving natural resources, waste management and the circular economy, the new edition of the hydraulic concrete standard [NF EN 206+A2/CN], published in November 2022, authorizes the use recycled gravel in quantities practically twice as large as in the previous edition (depending on the exposure class, etc.), but also authorizes the use of recycled sands. If we wish to bring geopolymer concrete back into use, the incorporation of recycled aggregates is thus an important subject from an ecological point of view and probably also economically for the future.

Indeed, a recent study [Skariah Thomas et al., 2022] reported that in the case of recycled aggregates, where the old cementitious paste attached to the original natural aggregate would be extremely porous, this paste could absorb the alkaline solution, thus resulting in geopolymerization of the new ITZ, which would improve the mechanical properties of such geopolymer concrete. As a result, geopolymer concrete has significant potential as a green construction material.

However, the properties of the ITZ interface have only been identified in the literature for very few types of geopolymer binders and very few types of aggregates.

The objective of this thesis work would thus be to characterize the paste-aggregate interfaces (natural and recycled aggregates) of geopolymer concretes, in the initial state, but also in the damaged state, then to identify the impact of these interfaces (damaged or not) on the tightness of geopolymer concretes.

Methodology

Experimental approach

In this thesis we propose to carry out an experimental study allowing initially to characterize the properties of the ITZ of geopolymer concretes, with natural aggregates and recycled aggregates. The methods implemented for the characterization of the ITZ at fine scales will be SEM microscopy associated with EDS (energy dispersive spectroscopy) analysis and instrumented nanoindentation. With this last method, we could deduce a spatial distribution of the micromechanical properties at the level of the aggregate, the paste and the interface, such as the microelastic modulus, the microhardness or the cracking energy associated with this scale. For the interface, this represents data expected to feed the input parameters of mesoscopic models, such as the one used in the work of Hayder Al-Khazraji [Al-Khazraji, 2017]. Indeed, the weak point of the description of the model was, among other things, the lack of data on the paste – aggregate interface and thus the attribution to this zone of the properties of the paste, with a width almost zero.

Another aspect of this study concerns the coupling of ITZ properties with cracking, due to mechanical loading. Among the characteristics of cracking, we mean initiation, propagation, crack opening, cracking energy and failure mode.

With regard to mechanical loading and stresses, we would like to highlight the effect of static and dynamic cyclic loading. A cyclic dynamic loading can be, on the one hand, representative for structures loaded by traffic or, on the other hand, representative to simulate seismic effects, natural or induced by human activity linked for example to the underground storage of pressurized fluids.

Under static loading, fracture mechanics predicts growth of larger defects. Thus, the microcracking must propagate from the larger pores or from a weak interface. Under dynamic loading, we can expect a privileged growth of certain pore sizes depending on the frequency content of the applied signal [Maurel et al., 2010] but this remains to be confirmed experimentally. Mechanical tests will be carried out on cylindrical specimens of materials loaded in indirect tension by splitting using a device developed and present in the laboratory, associated with acoustic emission. This will make it possible to detect the initiation and propagation of cracking in static or dynamic approach and to evaluate the gas permeability (indicator of tightness) according to the state of stress.

As part of this study, we will thus characterize the evolution of permeability at the macroscopic scale of geopolymer concretes under different types of loading, static or dynamic, but in parallel with this, we will use a technique of microscopic visualization by X-ray tomography to make the link between the evolution of (damaged) interfaces and (micro)cracking, or even network connectivity. These measurements will be supplemented by pore size distributions (MIP), carried out at different levels of damage.

Contribution to modeling

The distributions of pore sizes at different levels of damage will be used to investigate a permeability evaluation tool, based on the "random hierarchical bundle model", initially developed by [Garcia-Bengochea, 1979; Khaddour et al., 2018]. As part of this thesis work, we would like to be able to enrich this model by considering the flow properties at the (damaged) interfaces, based on the results of X-ray tomography. In addition, by employing a differential analysis between the distribution of pore sizes in a concrete and in a paste, we could access the distribution of pore sizes at the level of the interfaces, then at the level of cracks and damaged interfaces for different levels of damage. Thus, we will be able to obtain a multimodal representation for the set of phases at the MIP scale, which we could then transpose to a macroscopic scale and compare with experimental results on a cylindrical specimen of material.

Bibliographic references

Duzy P., Choinska M., Hager I., Amiri O., Claverie J., Mechanical Strength and Chloride Ions' Penetration of Alkali-Activated Concretes (AAC) with Blended Precursor. Materials (Basel). 2022 Jun 24;15(13):4475. doi: 10.3390/ma15134475.

Duzy, P., Sitarz, M., Adamczyk, M., Choińska, M., Hager, I., Chloride ions' penetration of fly ash and ground granulated blast furnace slags-based alkali-activated mortars, Materials, 2021, 14(21), 6583.

Shi C., Xie P., Interface between cement paste and quartz sand in alkali-activated slag mortars, Cem. Concr. Res. 28 (1998) 887–896.

Luo Z., Wengui Li, Kejin Wang, Arnaud Castel, Surendra P. Shah, Comparison on the properties of ITZs in fly ash-based geopolymer and Portland cement concretes with equivalent flowability, Cement and Concrete Research, Volume 143, 2021, https://doi.org/10.1016/j.cemconres.2021.106392.

Gopalakrishna B., Pasla Dinakar, Mix design development of fly ash-GGBS based recycled aggregate geopolymer concrete, Journal of Building Engineering, Volume 63, Part A, 2023, https://doi.org/10.1016/j.jobe.2022.105551.

PN RECYBETON, 2011, «RECYclage complet des BETONs, Étude de faisabilité», Projet National, IREX (Institut pour la recherche appliquée et l'expérimentation en génie civil).

Yammine A., Leklou N., Choinska M., Bignonnet F., Mechling J.-M., DEF damage in heat cured mortars made of recycled concrete sand aggregate, Construction and Building Materials 252 (2020) ⟨10.1016/j.conbuildmat.2020.119059⟩

NF EN 206+A2/CN, 2022, Béton - Spécification, performance, production et conformité - Complément national à la norme NF EN 206+A2, Normes nationales et documents normatifs nationaux.

Skariah Thomas B., Yang J., A. Bahurudeen, S.N. Chinnu, Jamal A. Abdalla, Rami A. Hawileh, Hussein M. Hamada, Geopolymer concrete incorporating recycled ggregates: A comprehensive review, Cleaner Materials, Volume 3, 2022, https://doi.org/10.1016/j.clema.2022.100056.

Al-Khazraji H., 2017, « Étude du couplage fissuration/perméabilité dans l'essai de fendage Brésilien », Thèse de l'Université de Nantes, Directeur de thèse : Pr. A. Khelidj, Co-encadrement : M. Choinska et N. Benkemoun.

Maurel, O., Reess, T., Matallah, M., De Ferron, A., Chen, W., La Borderie, C., Pijaudier-Cabot, G., Jacques, A., Rey-Bethbeder, F., Dec. 2010. Electrohydraulic shock wave generation as a means to increase intrinsic permeability of mortar. Cement and Concrete Research 40 (12), 1631–1638.

Garcia-Bengochea I., A.G. Altschaeffl, C.W. Lovell, Pore distribution and permeability of silty clays, J. Geotech. Eng. Div. 105 (1979) 839–856.

Khaddour F., D. Grégoire, G. Pijaudier-Cabot, A hierarchical model for the computation of permeation properties of porous materials and their enhancement due to microcracks, J. Eng. Mech. 144 (2018) 04017160, https://doi.org/10.1061/ (ASCE)EM.1943-7889.0001392

Required Skills

Knowledge of materials, Taste for experimentation, Adaptability and teamwork skills, Autonomy, Organizational skills.

Additional Comments

Planned funding: Contrat Doctoral Établissement ("CDE" grant, demand in progress)

Documents to attach to your application:
Cover letter
CV
Diplomas and transcripts
Letter(s) of recommendation