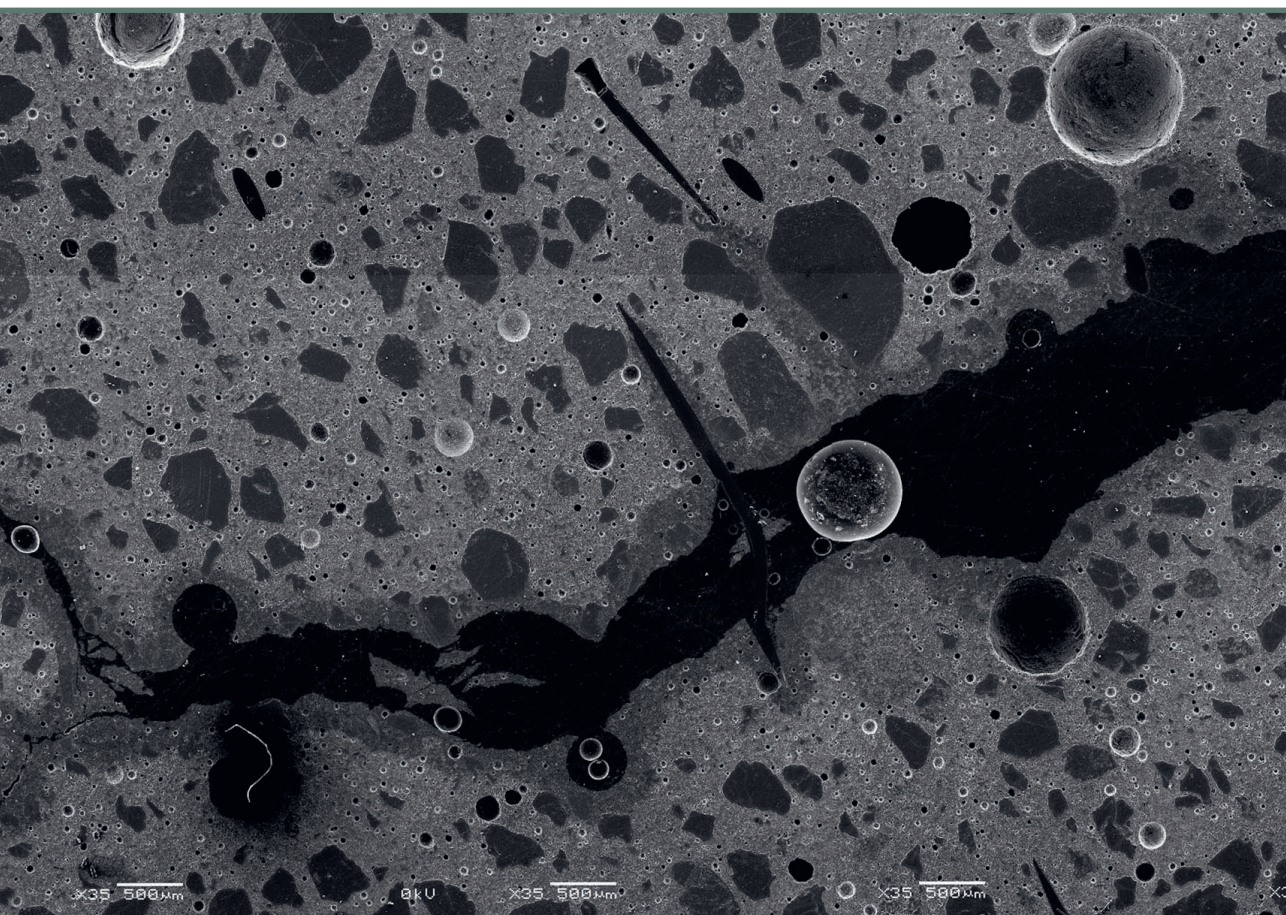


Rihards Gailītis

**METHODOLOGY FOR ASSESSING LONG-TERM
DEFORMATION INFLUENCE ON GEOPOLYMER
COMPOSITE MICROSTRUCTURE**

Summary of the Doctoral Thesis



RIGA TECHNICAL UNIVERSITY

Faculty of Civil Engineering
Institute of Structural Engineering

Rihards Gailītis

Doctoral Student of the Study Programme “Civil Engineering”

**METHODOLOGY FOR ASSESSING
LONG-TERM DEFORMATION INFLUENCE
ON GEOPOLYMER COMPOSITE
MICROSTRUCTURE**

Summary of the Doctoral Thesis

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DOCTORAL THESIS PROPOSED TO RIGA TECHNICAL UNIVERSITY FOR THE PROMOTION TO THE SCIENTIFIC DEGREE OF DOCTOR OF SCIENCE

To be granted the scientific degree of Doctor of Science (Ph. D.), the present Doctoral Thesis has been submitted for the defence at the open meeting of RTU Promotion Council on 10th of November 2023 at 14.15 at the Faculty of Civil Engineering of Riga Technical University, Kļīpsalas 6a, Room 342.

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DECLARATION OF ACADEMIC INTEGRITY

I hereby declare that the Doctoral Thesis submitted for review to Riga Technical University for promotion to the scientific degree of Doctor of Science (Ph. D.) is my own. I confirm that this Doctoral Thesis has not been submitted to any other university for promotion to a scientific degree.

Rihards Gailītis (signature)

Date:

The Doctoral Thesis has been written in English as a summary of scientific publications. It consists of a summary and nine scientific publications. Seven of the publications are in English, and their total length is 65 pages. Two of the publications are Latvian national patents and are in Latvian, and their total length is 34 pages. The Bibliography contains 185 titles.

Acknowledgement

This Doctoral Thesis was written from 2018 till 2023 at the Institute of Structural Engineering of Faculty of Civil Engineering of Riga Technical University.

I would like to thank the personnel of the departments of the Faculty of Civil Engineering of Riga Technical University – the Department of Structural Engineering, Department of Building Materials and Products, Department of Structural Mechanics, Department of Composite Materials and Structures – as well as the employees of the Faculty of Material Engineering and Physics of the Cracow University of Technology.

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IEGULDĪJUMS TAVĀ NĀKOTNĒ

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ABBREVIATIONS

ADPF – abiotic depletion of fossil fuels
BA – bottom ash
BFA – biomass fly ash
Ca(OH)₂ – calcium hydroxide
CAC – calcium aluminate cement
CaCO₃ – calcium carbonate
CT – curing time
EFC – Earth Friendly Concrete
FA – fly ash
FS – ferrochrome slag
GBFS – granulated blast furnace slag
GGBFS – ground granulated blast furnace slag
GLSS – granulated lead smelter slag
GP – geopolymer
GPC – geopolymer concrete
IOT – iron ore tailing
KHFA – ultra-fine kaolite high-performance ash
KL – kaolin
LS – limestone
LWA – lightweight aggregate
M – molarity
MK – metakaolin
NaCl – sodium chloride
NaOH – sodium hydroxide
NS – Nano-silica
OPC – Ordinary Portland cement
PAN – polyacrylonitrile
PE – polyethylene
PET – polyethylene terephthalate
PCFA – powder coal fly ash
POFA – palm oil fuel ash
PP – polypropylene
PVA – polyvinyl alcohol
RHA – rice husk ash
SD – sawdust
SEM – scanning electron microscope
SF – silica fume

SiO₂/Al₂O₃ – silicon dioxide / aluminium oxide

SMF – silico-manganese fume

SSD – saturated surface dry

GENERAL DESCRIPTION OF THE THESIS

The aim of the work

1. Evaluate mechanical and long-term properties of geopolymer composite reinforced with different fibers in compression, tension and three-point bending.
2. Determine fiber reinforced geopolymer composite long-term deformation influence on microstructure in compression, tension and three-point bending.

Tasks

1. Develop geopolymer composites that have their properties enhanced by:
 - a) polyvinyl alcohol fibers;
 - b) steel fibers;
 - c) polypropylene fibers;
 - d) carbon fibers;
 - e) waste steel fibers from car tire recycling.
2. Determine the developed composites' properties:
 - a) compressive, tensile and bending strength;
 - b) creep and drying shrinkage.
3. Develop a method for the long-term load application effect assessment on cement composites in various stress-strain states.
4. Determine possible links between the specimen cross-section microstructure composition and the long-term deformations.

Scientific novelty

1. A new methodology has been developed and patented for long-term property determination in compression, tension and three-point bending for concrete and cement composites.
2. Patent application The Technique for Outside Effect Determination on Concrete and Cement Composite Microstructure in Various Stress-Strain States has been developed and filed.
3. The effect of fiber reinforcement effect on geopolymer composite mechanical and long-term properties has been tested and analysed.
4. Long-term load effect on geopolymer composite microstructure in compression, tension and three-point bending has been assessed.

Practical novelty

1. A new method of long-term property determination in laboratory conditions has been developed for concrete and cement composites that further leads to increased information about novel concrete and cement composites and their application in civil engineering.
2. A patent was developed and filed for a new technique for outside effect determination on concrete and cement composite microstructure in various stress-strain states by testing polished section specimens in a scanning electron microscope. It allows to increase information regarding long-term load application effect on concrete and cement composite microstructure.
3. Compositions of geopolymer composites have been developed to whom mechanical and long-term properties have been determined and can be used for structure development for application in civil engineering.
4. Increased information is provided on fly ash-based geopolymer composite mechanical and long-term properties and fiber reinforcement influence on their long-term properties, thus increasing application of these composites in civil engineering.

Structure of the Thesis

The Thesis is a summary of scientific publications focused on long-term and mechanical property assessment of plain and fiber reinforced geopolymer composites and the determination of long-term deformation effect on geopolymer composite microstructure.

Approbation and publications

The results of the Thesis have been published in 22 SCI journals and conference proceedings (13 of them are published in conference proceedings and 2 are patents) and have been presented in 15 international conferences.

List of papers

1. **Gailitis, R.**, Pudans, P., Ziemelis, K., Bumanis, G., Sprince, A. Early-Age Creep and Shrinkage Properties of Printed and Cast Cement Composite. (2023). Materials Proceedings. 13(1) 35.
2. **Gailitis, R.**, Sprince, A., Kozlovskis, T., Pakrastins, L., Volkova, V. Impact of Polypropylene, Steel, and PVA Fibre Reinforcement on Geopolymer Composite Creep and Shrinkage Deformations. (2023) Journal of Physics: Conference Series, 2423 (1).
3. Radina, L., Sprince, A., Borodinecs, A., Pakrastins, L., **Gailitis, R.**, Sakale, G. Foamed Geopolymers: A Review of Recent Studies. (2023) Journal of Physics: Conference Series, 2423 (1).

4. **Gailitis, R.**, Pakrastins, L., Sprince, A., Radina, L., Sakale, G., Miernik, K. Different Fiber Reinforcement Effect on Fly Ash Based Geopolymer Long-Term Deflection in Three-Point Bending and Microstructure. (2022) *Materials*, 15 (23).
5. Pupure, L., Varna, J., **Gailitis, R.**, Al-Maqdasi, Z., Pakrastins, L. Development of Methodology for Experimental Parameter Identification for Inelastic 3D Material Model (2022) *ECCM 2022 – Proceedings of the 20th European Conference on Composite Materials: Composites Meet Sustainability*, 6, pp. 282–289.
6. **Gailitis, R.**, Figiela, B., Abelkalns, K., Sprince, A., Sahmenko, G., Choinska, M., Guigou, M. D. Creep and shrinkage behaviour of disintegrated and non-disintegrated cement mortar. (2021) *Materials*, 14 (24).
7. Sprince, A., Kozlovskis, T., **Gailitis, R.**, Valivonis, J., Korniejenko, K., Castel, A. Tensile creep of cement and concrete composites: Monitoring by means of 2D-digital image correlation. (2021) *Applied Sciences (Switzerland)*, 11 (18).
8. Kozub, B., Bazan, P., **Gailitis, R.**, Korniejenko, K., Mierzwiński, D. Foamed geopolymer composites with the addition of glass wool waste. (2021) *Materials*, 14 (17).
9. **Gailitis, R.**, Sprince, A., Kozlovskis, T., Radina, L., Pakrastins, L., Vatin, N. Long-term properties of different fiber reinforcement effect on fly ash-based geopolymer composite. (2021) *Crystals*, 11 (7).
10. Sprince, A., **Gailitis, R.**, Pakrastins, L., Kozlovskis, T., Vatin, N. Long-term properties of cement mortar under compression, tension, and 3-point bending. (2021) *Magazine of Civil Engineering*, 105 (5).
11. **Gailitis, R.**, Sprince, A., Pakrastins, L., Bazan, P., Korniejenko, K. Plain and PVA fibre-reinforced geopolymer compact tension specimen critical area surface composition assessment. (2021) *Vide. Tehnologija. Resursi – Environment, Technology, Resources*, 3, pp. 72–77.
12. **Gailitis, R.**, Sprince, A., Pakrastins, L., Korniejenko, K., Kozlovskis, T. Plain Geopolymer Concrete Cross-Section Surface Analysis After Creep and Shrinkage Tests in Compression and Tension. (2021) *RILEM Book series*, 31, pp. 13–24.
13. **Gailitis, R.**, Sprince, A., Pakrastins, L., Korniejenko, K., Kozlovskis, T. Reinforced and Plain Geopolymer Concrete Specimen Cross-section Composition Influence on Creep Strains. (2021) *Proceedings of 4th International RILEM conference on Microstructure Related Durability of Cementitious Composites (Microdurability2020)* (Indexation pending).
14. **Gailitis, R.**, Korniejenko, K., Sprince, A., Pakrastins, L. Comparison of the long-term properties of foamed concrete and geopolymer concrete in compression. (2020) *AIP Conference Proceedings*, 2239.

15. **Gailītis, R.**, Sliseris, J., Korniejenko, K., Miķuļa, J., Łach, M., Pakrastins, L., Sprince, A. Long-Term Deformation Properties of a Carbon-Fiber-Reinforced Alkali-Activated Cement Composite. (2020) *Mechanics of Composite Materials*, 56 (1), pp. 85–92.
16. Sprince, A., Pakrastins, L., **Gailītis, R.** Long-Term Parameters of New Cement Composites. (2020) *RILEM Book series*, 24, pp. 85–94.
17. **Gailītis, R.**, Korniejenko, K., Łach, M., Sliseris, J., Morán, J., Rodriguez, E., Miķuļa, J. Mechanical Properties of Geopolymer Concretes Reinforced with Waste Steel Fibers. (2019) *IOP Conference Series: Materials Science and Engineering*, 660 (1).
18. **Gailītis, R.**, Sprince, A., Pakrastins, L., Sahmenko, G., Kozlovskis, T. Drying Shrinkage Deformation Comparison between Foam Concrete, Geopolymer Concrete, Disintegrated, and Non-disintegrated Cement Mortar. (2019) *IOP Conference Series: Materials Science and Engineering*, 660 (1).
19. **Gailītis, R.**, Sprince, A., Pakrastins, L., Shakhmenko, G., Kozlovskis, T., Radina, L. Long-Term Properties of Foamed Concrete. (2019) *Proceeding of 13th International Conference Modern Building Materials, Structures and Techniques (MBMST 2019)* (Indexation pending).
20. **Gailītis, R.**, Sprince, A., Pakrastins, L., Shakhmenko, G., Kozlovskis, T. Comparison of the long-term properties in compression of different size foamed concrete (2019) *Vide. Tehnoloģija. Resursi – Environment. Technology. Resources*, 3, pp. 41–44.

National patents

1. Sprince, A., Pakrastins, L., Radina, L., **Gailītis, R.**, Kozlovskis T. Paņēmiens betona un cementa kompozītu ilglaicīgo īpašību noteikšanai dažādos sprieguma stāvokļos/ Method for Determination of Long-Term Properties of Concrete and Cement Composites in Various Stress Conditions/ Latvian patent No. **15659B**.
2. **Gailītis, R.**, Sprince, A., Pakrastins, L. Paņēmiens ārējo iedarbju ietekmes noteikšanai uz betonu un cementa kompozītmateriālu mikrostruktūru dažādos sprieguma stāvokļos/ Technique for Outside Effect Determination on Concrete and Cement Composite Microstructure in Various Stress-Strain States/ Latvian patent application No. **LVP2023000039 (Filed for patent)**.

Results of the research were presented at the following conferences

1. 10th Scientific-Technical Conference on Material Problems in Civil Engineering MatBud'2023, Cracow, Poland, 19–21 April 2023.
2. 5th International Conference on Innovative Materials, Structures and Technologies, IMST 2022, Riga, Latvia, 28–30 September 2022.
3. 2022 Global Conference on Polymers, Plastics and Composites (PPC2022), Budapest, Hungary, March 21–22, 2022.
4. RTU 62. starptautiskā zinātniskā konference apakšsekcija “Būvniecība”, tiešsaiste, 28. oktobris 2021.

5. World Symposium on Mechanical-Materials Engineering & Science (WMMES2021) Prague, Czech Republic, 9–11 September 2021.
6. 13th International Scientific and Practical Conference. Environment. Technology. Resources, online, 17–18 June 2021.
7. The Biot-Bažant Conference on Engineering Mechanics and Physics of Porous Materials (A One-time Fusion of Concreep and the Biot Conference on Poromechanics), online, 1–3 June 2021.
8. 4th International RILEM conference on Microstructure Related Durability of Cementitious Composites (Microdurability2020), online, 29 April – 25 May 2021.
9. International RILEM Conference on Early-age and Long-term Cracking in RC Structures (CRC2021), online, 9–10 April 2021.
10. RTU 61. starptautiskā zinātniskā konference apakšsekcija “Būvniecība”, tiešsaiste, 22. Oktobris, 2020.
11. 1st International Scientific Conference Advanced Construction and Architecture 2020 (ACA2020), online, 23–25 September 2020.
12. 4th International Conference on Innovative Materials, Structures and Technologies, IMST 2019, Riga, Latvia, 25–27 September 2019.
13. 4th Polish Congress of Mechanics, PCM 2019 and the 23rd International Conference on Computer Methods in Mechanics, CMM 2019, Cracow, Poland, 8–12 September 2019.
14. 12th International Scientific and Practical Conference “Environment. Technology. Resources”, RTA, Rezekne, June 20–22, 2019 (Plenary Session).
15. 13th International Conference “Modern Building Materials, Structures and Techniques”, Vilnius, Lithuania, May 16–17, 2019.

Declaration of the authorship for the papers

Rihards Gailītis has conducted a major part of the experimental work, evaluated the results, and written all of the appended papers and Patent II. In general, the co-authors contributed with experiment planning, provision of materials, SEM internship arranging, specific specimen preparation, and constructive criticism/advice on the obtained results and their representation in the publications, which further increased the scientific quality of the publications.

Paper No.	Reference	Corresponding author	R. Gailitis' contribution
Paper I	Gailītis, R. , Korniejenko, K., Łach, M., Sliseris, J., Morán, J., Rodriguez, E., Miķūla, J. Mechanical Properties of Geopolymer Concretes Reinforced with Waste Steel	R. Gailitis	95 % of the mechanical property testing and evaluation, test result formatting and assessment, manuscript preparation.

	Fibers. (2019) IOP Conference Series: Materials Science and Engineering, 660 (1), art. no. 012007, Open Access, SNIP 0.344.		
Paper II	Gailitis, R., Sliseris, J., Kornijejenko, K., Mikula, J., Łach, M., Pakrastins, L., Sprince, A. Long-Term Deformation Properties of a Carbon-Fiber-Reinforced Alkali-Activated Cement Composite. (2020) Mechanics of Composite Materials, 56 (1), pp. 85–92, SNIP 0.832.	R. Gailitis	95 % of specimen preparation for the long-term and mechanical property tests and the tests, result evaluation preparation of the manuscript, and structure of SEM micro analysis.
Paper III	Gailitis, R., Sprince, A., Kozlovskis, T., Radina, L., Pakrastins, L., Vatin, N. Long-term Properties of Different Fiber Reinforcement Effect on Fly Ash-based Geopolymer Composite. (2021) Crystals, 11 (7), art. no. 760, Open Access, SNIP 0.821.	R. Gailitis	Preparation of the geopolymer composite specimens, 80 % of the experimental work that includes their adaptation to creep and shrinkage tests, all the mechanical property assessment, creep and shrinkage test result assessments, result formatting, and manuscript preparation and revision.
Paper IV	Gailitis, R., Sprince, A., Pakrastins, L., Kornijejenko, K., Kozlovskis, T. Reinforced and Plain Geopolymer Concrete Specimen Cross-section Composition Influence on Creep Strains. (2021) Proceedings of 4th International RILEM Conference on Microstructure Related Durability of Cementitious Composites	R. Gailitis	Specimen preparation, 85 % of the long-term testing, development, and adaptation of microstructure specimen preparation and investigation, and further specimen investigation with SEM and result quantitative analysis, preparation, and presentation of the scientific paper and findings.

	(Microdurability2020), Open Access.		
Paper V	Gailitis, R. , Sprince, A., Pakrastins, L., Kornijejenko, K., Kozlovskis, T. Plain Geopolymer Concrete Cross-Section Surface Analysis After Creep and Shrinkage Tests in Compression and Tension (2021) RILEM Book series, 31, pp. 13–24. SNIP 0.320.	R. Gailitis	The specimen preparation, 65 % of the long-term mechanical property testing, mechanical property assessment and creep and shrinkage test result assessment, the polished section preparation and analysis specifically adapted for the geopolymer composite testing purposes, result formatting, and manuscript preparation and revision.
Paper VI	Gailitis, R. , Sprince, A., Pakrastins, L., Bazan, P., Kornijejenko, K. Plain and PVA Fibre-reinforced Geopolymer Compact Tension Specimen Critical Area Surface Composition Assessment. (2021) Vide. Tehnologija. Resursi/ Environment. Technology. Resources, 3, pp. 72–77, Open Access.	R. Gailitis	80 % of the specimen preparation, 80 % of the testing of the long-term properties, 100 % of the microstructure tests, preparation of the manuscript, revision, and presenting in the conference.
Paper VII	Gailitis, R. , Pakrastins, L., Sprince, A., Radina, L., Sakale, G., Miernik, K. Different Fiber Reinforcement Effect on Fly-Ash Based Geopolymer Long-Term Deflection in Three-Point Bending and Microstructure (2022) Materials, 15 (23), Open Access, SNIP 1.137.	R. Gailitis	100 % of the specimen preparation, 60 % of the specimen long-term testing, 100 % of the microstructure specimen preparation and testing, and 100 % of the manuscript preparation and all the revision cycles.
Patent I	Sprince, A., Pakrastins, L., Radina, L., Gailitis, R. , Kozlovskis, T., Paņēmiens betona un cementa kompozītu ilglaicīgo īpašību noteikšanai	A. Sprince	Testing in the laboratory environment for method validation of the developed methods for long-term property determination in compression,

	dažādos sprieguma stāvokļos/ Method for Determination of Long-Term Properties of Concrete and Cement Composites in Various Stress Conditions/ Latvian patent No. 15659B.		tension, and 3-point bending of the cement composites.
Patent II	Gailitis, R., Sprince, A., Pakrastins, L. Paņēmiens ārējo iedarbju ietekmes noteikšanai uz betonu un cementa kompozītmateriālu mikrostruktūru dažādos sprieguma stāvokļos/ Technique for Outside Effect Determination on Concrete and Cement Composite Microstructure in Various Stress-Strain States/ Latvian patent application No. LVP2023000039	R. Gailitis	R. Gailitis developed the methods for polished section preparation and surface analysis and assessment procedures of specimens that have been subject to long-term testing. Also, methodology has been tested, and the results were published in conference proceedings as well as full-text publications. He prepared the patent application together with the RTU patent specialist.

Thesis to defend

1. Polypropylene fiber with an amount of 1–5 % and 5 % waste steel cord fiber incorporation into the geopolymer matrix reduces creep in compression, and 1 % steel fiber incorporation reduces creep in three-point bending in contrast to creep exhibited of the plain geopolymer specimens. In the cases of 1 % carbon fiber or 1 % steel fiber incorporation for creep in compression, 1 % PVA fiber incorporation for specimens tested in tension and 0.5 % PVA/0.5 % steel, and 1 % PVA fiber introduction for specimens tested in three-point bending raises creep strains. This is mainly due to the significantly higher specific surface area of polypropylene fibers and bonding properties between the geopolymer matrix and steel fibers.
2. Fiber incorporation into geopolymer composite does reduce shrinkage strains only in the cases of 1–5 % polypropylene fiber, 1 % steel fiber, and 5 % waste steel cord fiber incorporation in compression and 1 % polyvinyl alcohol fiber introduction in tension. By adding 1 % carbon fibers, shrinkage is not reduced significantly. This is because of increased air entrapment due to fiber incorporation and increased micro crack amount in the specimen.
3. Developed and validated new methodology “Method for Determining the Long-Term Properties of Concrete and Cement Composites in Various Stress-strain Conditions” No. 15659B.
4. Developed and validated a new methodology “Technique for Outside Effect Determination on Concrete and Cement Composite Microstructure in Various Stress-Strain States”, No. LVP2023000039.
5. As a result of the creep specimen, tested in a linear state, and shrinkage specimen microstructure analysis, it is determined that in tension, micro cracks have a significant influence on long-term properties. The amount of them in creep and shrinkage specimens is similar. The same results are visible in specimens subjected to three-point bending. For the specimens meant for long-term testing in compression, micro cracks have not been discovered. The amount of micro cracks differs due to the specimen surface area and size differences.

1. INTRODUCTION

Ordinary Portland cement (OPC) is used as a common binder in most of the conventional concrete structures. The buildings and infrastructure made from concrete have been safe and durable. Furthermore, properties and their development for regular OPC-based concrete are well known [1]. This makes the demand for OPC high, and it is expected to rise in the coming years due to rapid urbanization and the high demand for residential units [2]. Annual OPC production in the next 30-year period is expected to increase by 50 % [3], [4]. Despite all the advantages that OPC provides, the biggest drawback is OPC's energy intensity that further means high CO₂ emissions during the manufacturing process.

Carbon dioxide and other pollutants are the main cause of global warming. In Fig. 1, where CO₂ emissions worldwide are presented, it is visible that the main source of CO₂ emissions from a usage perspective is energy production, which produces twice as much CO₂ than industry. If we compare each country's contribution to CO₂ emission, we see that the biggest pollutant is China, that emits 1/3 greater amount of CO₂ than the USA and nearly twice as much as EU. The United States Geological Survey (see Fig. 2) shows unbalanced OPC production in the world. The largest producers are based in Eastern Asia [5].

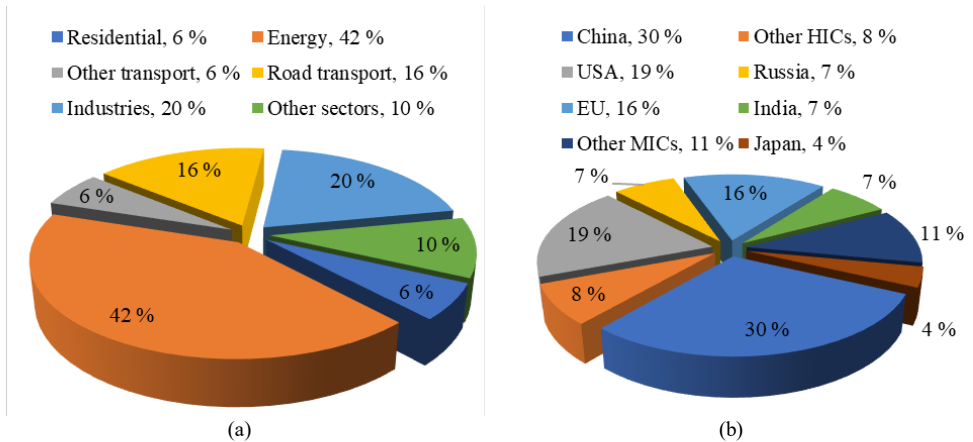


Fig. 1. CO₂ emissions worldwide (a); by sectors (b) by countries [6].

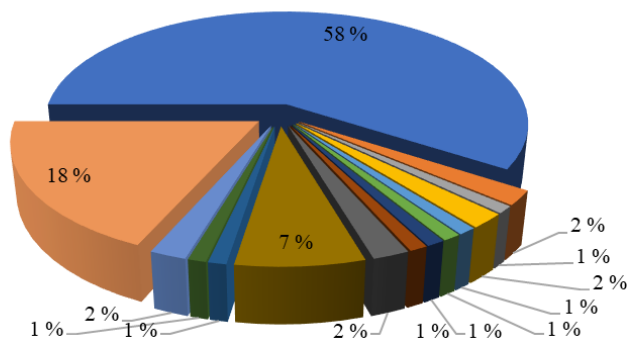
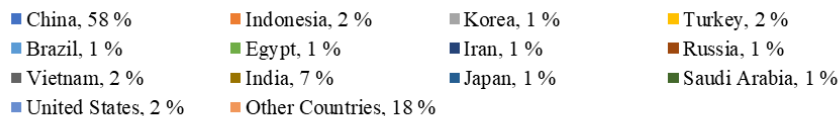


Fig. 2. OPC production around the world [5].

Depending on the source, the cement production contribution to CO₂ emission is reported to be 5–30 %. In [7], it was reported that cement production contributes 5 % of all CO₂ emissions, others estimated that cement clinker production was responsible for 8 % of the world CO₂ emissions in 2017 [8], [9] that dropped to 4 % in 2019 [10]. Others claim that all in all, concrete production is responsible for up to 30 % of all CO₂ emissions [11].

The calcination process and raw material burning are pointed out as the main sources of greenhouse gas emission sources [12], [13]. Emission amounts of CO₂ are alarming, and recent reductions in them are mainly caused by the COVID-19 pandemic and further disruption in worldwide supply chains not by demand reduction or novel material application introduction in the building environment. Therefore, it is a high priority to take measures and drastically reduce CO₂ emissions, taking meaningful actions when choosing building materials.

CO₂ emissions from the production of concrete and OPC can be reduced by different approaches:

- cement replacement by secondary raw materials and/or secondary cementitious materials;
- use of alternative fuel and energy resources in clinker cement production;
- use of alternative binders in the manufacturing of cement;
- changes in manufacturing processes and techniques [14].

One of the possible solutions is geopolymeric material that can be produced from recycled materials such as fly ash, silica fume, various slags, meta-kaolin, and others that would totally or in part replace the cement in concrete [15], [16]. It is known that alkali activated concrete has up to 60 % lower energy consumption than the Portland cement concrete [17]. Furthermore, it is claimed that geopolymer composites have the same or even better mechanical properties than OPC

composites. Even though a lot of research for the purposes of geopolymer wider commercial application has been done, there is a need for more investigations on long-term behavior and durability [18]. It has been determined that geopolymer composites have higher shrinkage while polymerized as well as the creep values in elevated temperatures and other specific situations [19]–[27]. But there is a lack of information about the creep and shrinkage properties of the polymerized geopolymer composite elements, and it is not known if they have similar long-term property decrease as the OPC based composites.

2. GEOPOLYMER COMPOSITES

In 1978, a new reaction of solid aluminosilicate with alkali solution was found by Davidovits to produce a geopolymer (GP). Geopolymer is a binder that is created in the reaction of aluminium oxide (Al_2O_3) and silicon dioxide (SiO_2). This binder, in its basic form, has a low Ca amount, unlike OPC. This binder has a tetrahedral framework that is connected with long-range covalent bonds [28]. It has been found that the efficiency and rate of GP forming are greatly affected by hydroxide or hydroxide base plus silicate [29]. To this day, various silicate and aluminate silicate materials have been used to create GPs, for instance, red mud, rice husk ash, ground granulated blast furnace slag, fly ash, and metakaolin [30]–[32].

In the literature regarding geopolymer it is claimed that in most cases, geopolymer composites show the same or superior properties as the OPC based composites. General property differences between geopolymer concrete and ordinary Portland cement are shown in Table 1.

Table 1

Differences in the Properties of GPC and OPC [1], [33]–[41]

Properties	Geopolymer concrete	Ordinary Portland cement concrete	Comments
Compressive strength	Higher	Lower	At an early age. GPC gains higher strength compared to OPC. The main factors influencing GPC strength are aluminosilicate source, type of activator, curing conditions and length, and reactivity of the precursors
Water absorption	Moderate	Slightly lower	Internal porosity in the GPC matrix slightly increases its water absorption in contrast to OPC
Setting time	Faster	Slower	In general, GPC is considered to have a short setting time, but unlike the OPC setting time, it is more affected by the factors such as activator type, source material properties, and exposure to temperature
Shrinkage	Moderate	Lower	Curing conditions and the mixture's moisture content affect shrinkage-related crack development to GPC
Tensile strength	Higher	Lower	GPC shows higher tensile strength and compressive strength
Durability	Higher	Lower	Presence of silicate and alumina products provides better durability of GPC
Resistance to acids	Higher resistance	Lower resistance	Alumino-silicates in GPC contribute to better acid resistance than in OPC
Fire resistance	Typically, higher	Limited	Due to its chemical composition, GPC degrades less in fire, while OPC shows less fire resistance
Freezing and thawing cycles	Less susceptible	More susceptible	GPC shows greater resistance to aggressive environments and to rapid temperature changes in the environment
CO ₂ emissions	Low	High	Overall, GPC poses smaller potential CO ₂ emissions than OPC during the life cycle
Porosity	Moderate	Lower	GPC shows a greater number of pores in the cross-section
Insulating properties	Higher	Limited	Depending on the curing condition, precursor and activator types of GPC show better insulating properties than OPC

In the next sections, geopolymer properties and its microstructure and environmental influence will be discussed. In the subsections, geopolymer constituents, properties, microstructure, and its influence and life cycle assessment will be discussed in detail.

2.1.Constituents

2.1.1. Aluminosilicates

Aluminosilicates, or in other words, materials that are composed of aluminium, silicone, and oxygen, are the base constituent of GP materials. They are sourced from waste utilization and are indispensable in making the GP matrix. Fly ash (FA) is a primarily used waste product for manufacturing GP. Other aluminosilicate sources and individuals in combination with each other and as standalone aluminosilicates have also been reported. The aluminosilicate materials as well as additives for the creation of GPs are shown in Table 2.

Table 2

Aluminosilicates as Source Materials for Geopolymer Composites [42]

Type	Abbreviation
(a) Aluminosilicate as source material	
Fly ash	FA
Bottom ash	BA
Granulated blast furnace slag	GBFS
Metakaolin	MK
Natural zeolites	
Kaolin	KL
Palm oil fuel ash	POFA
Granulated lead smelter slag	GLSS
Rice husk ash	RHA
Ferrochrome slag	FS
Ultra-fine kaolite high-performance ash	KHFA
Biomass fly ash	BFA
Silico-manganese fume	SMF
(b) Additives as source material	
Calcium aluminate cement	CAC
Nano-silica	NS
Calcium hydroxide	Ca(OH) ₂
Ordinary Portland cement	OPC
Silica fume	SF

2.1.1.1. Fly ash

Fly ash is generated during the burning process by industries that consume coal as an energy source or manufacturing ingredients. Mainly, these are power plants and metallurgic plants. The scheme of coal power plant operating, where fly ash is obtained, is shown in Fig. 3.

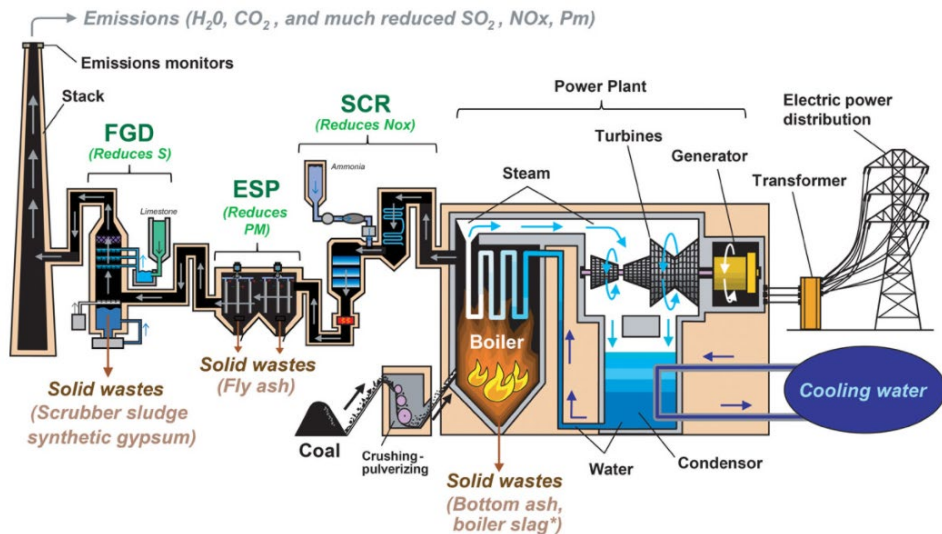


Fig. 3. Coal power plant's operational scheme [43].

For power plant application, the coal is crushed and introduced into the ignition chamber together with air. Here the crushed coal combusts generating heat and molten minerals. Boiler tubes remove all the heat from the kiln. As a result, fuel gas gets cooler and mineral residue hardens and forms ash. Coarse ash drops to the bottom of the ignition chamber and is further characterized as slag. Fly ash particles are retained in the fuel gas. As the gas rises, fly ash particles are captured in electrostatic precipitators or fabric filters that are placed for particle emission requirements.

Fly ash contains Al_2O_3 and SiO_2 that are key ingredients to form cementitious substances. Fly ash reacts with lime $\text{Ca}(\text{OH})_2$ in the presence of water to form compounds identical to cement. Its usage in concrete has a significant effect, and it is found that it has high potential as a supportive material to GP. Fly ash containing concrete has greater strength and is more durable as compared to conventional concrete. By using fly ash, the cost of concrete is reduced and sulphate resistance is increased [44].

Using fly ash, landfill areas can be conserved and even reduced, water consumption and energy needs reduced, and greenhouse gas emissions minimized. Using fly ash, the needed amount of OPC is reduced and therefore CO_2 emissions are greatly reduced as well. Per ton fly ash used the CO_2 emissions are reduced by a ton. Utilizing a whole year's supply of fly ash in concrete is equivalent to 25 % disposal of CO_2 released by vehicles worldwide [45].

In the author's research, described in **Papers I–VII** [46]–[52], fly ash from the coal power plant located in Skawina, Poland, was used. It was determined that this fly ash is particularly suitable for geopolymer due to high SiO_2 (47.81 %) and Al_2O_3 (22.80 %) content.

2.1.2. Reinforcement

In field practice, fibers are added to concrete to reduce or even altogether avoid early age cracking caused by shrinkage creep. Fibers also increase the concrete's tensile strength and crack resistance in the later stages of its life [53]. Often the material properties of fibers are more dominant than the binder properties in altering the performance of fiber reinforced geopolymer composite [54]. A good example is poly-propylene fibers that regardless of the binder type (OPC-based or geopolymer) would show weak binder and fiber interaction that further contributes to reduced compressive strength of the composite [55]–[59]. The physical and mechanical properties of the fibers used as reinforcement are compiled in Table 3.

Table 3

Reinforcement Fiber's Physical and Mechanical Properties [60]–[65]

Material category			Density (g/cm ³)	Tensile strength (MPa)	Modulus of elasticity (GPa)	Ultimate elongation (%)
Metallic		Steel	7.65–7.85	345–2850	200–210	0.5–3.5
Carbon based	Synthetic	PAN	1.8–1.9	2500–7000	250–500	0.6–2.5
		Rayon	1.4–1.7	500–1500	35–60	2.5
		Mesophase patch	1.6–2.2	1500–3500	200–900	0.3–0.9
		Graphene		130000	1000	
		Carbon nanotube		11000–63000	1000–1800	
		Polyvinyl alcohol	1.2–1.3	800–2500	29–42	5.7–7
		Polypropylene	0.9–0.95	240–760	1.5–10	15–80
		Polyethylene	0.92–0.97	80–3500	5–113	3–100
		Aramid	1.38–1.47	2300–3500	63–120	2–4.5
		Acrylic	1.16–1.18	270–1000	13.8–19.3	
		Polyethylene terephthalate	1.3–1.4	420–450	3.1–10	11.2
	Polyester	1.22–1.38	580–1100	15	35.0	
	Nylon	1.13–1.41	440–1000	4.1–5.2	16–20	
	Natural	Jute	1.3–1.5	250–350	26–32	1.5–1.9
		Sisal	1.34–1.45	280–750	13–26	3.0–5.0
		Coconut	0.87–1.4	120–200	19–26	10.0–25.0
		Bamboo	0.6–1.1	140–800	11–32	2.5–3.7
		Cotton	1.5–1.6	390–600	5.8–11	6.0–10.0
		Palm	1.3–1.46	21–60	0.6	
		Wool	1.3	160	3.5	
		Hemp	1.4–1.5	270–900	23.5–90	1–3.5
		Kenaf	1.4	223–930	14.5–53	1.5–2.7
		Coir	1.15–1.46	95–230	2.8–6	15–51.4
		Banana	1.4	500	12.0	1.5–9
		Flax fabric	1.5	500–1500	50–70	
		Bagasse	1.3	222–290	17–27	1.1
		Abaca	1.5	400–980	6.2–20	1–10
Inorganic		E-glass	2.5–2.62	3100–3800	72.4	4.8
		S-glass	2.46–2.49	4020–4650	86.9	5.4
	C-glass	2.6	3310	69	4.8	
	AR-glass	2.7	3240	73	4.4	
	Basalt	2.65–2.80	3000–4840	89–110	3.00–3.15	
	Asbestos	2.55	620	160		
	Alumina	3.3–3.95	1700–2000	300–380	0.4	
	Alumina- silica	3.4	1590–2550	200–248	0.8–1	
	Silicone Nitride		2500–4800	195–300		
	Silicone Carbide	2.5–2.7	2200–3450	221–250		
	Boron nitride	7.65–7.85	2100	345		

In further subsections, the most commonly industrially used fibers are described.

2.1.2.1. Steel fibers

Steel fibers are used mainly due to their high mechanical strength, flexibility and availability. According to ASTM A820-16 steel fibers can be arranged into 5 groups depending on their usage and purposes. They are: (1) pieces of smooth or deformed cold-draw wire; (2) smooth or deformed cut sheet; (3) melt-extracted; (4) mill cut; and (5) modified cold-drawn wire steel fibers. These fibers have a wide range of tensile strength (310–2850 MPa) and ultimate elongation (0.5–3.5 %) that differs according to the material and fabrication process [66]–[68].

The main disadvantage of steel fiber application is corrosion. To limit the corrosion's effect, mainly two solutions are applied: (1) usage of stainless steel alloys, for example, austenitic, ferritic, martensitic, duplex, and precipitation hardenable steels [69], or (2) sacrificial coating on fibers like copper and zinc [70], [71].

In **Papers I, III, IV, and VII** [46], [48], [49], [52], steel fibers were used. In **Papers I and IV** [46], [49], waste steel fibers from car tyre recycling were used. As steel chords from tyres are not useful for metallurgical application, they mostly end up in the landfill. In specimen preparation, it was observed that due to the different chord cross-section dimensions and length, it was hard or, in the case of cylindrical specimen preparation, nearly impossible to incorporate tyre chords into the geopolymer composition while mixing, like for regular steel fiber reinforcement. Therefore, a more suitable way of incorporation of waste steel fiber reinforcement is putting it into the mold and then fill and vibrate the matrix into it. In **Papers III and VII** [48], [52], steel fibers La Gramigna gold (0.3 x 20 mm) were used. It was observed that low amounts of steel fiber addition do not significantly affect geopolymer composite flowability and consistency.

2.1.2.2. Inorganic fibers

Inorganic fibers consist of a mixture of silica and alumina. Those fibers have a high melting point that makes them suitable for thermal application. These fibers have low cost, high tensile strength and chemical stability, and good insulating properties [60]. Silica fibers, aluminosilicate, alumina fibers, and basalt fibers are the most widely used inorganic fibers.

2.1.2.3. Carbon fibers

Reinforcement fibers, known for having the highest specific strength and lightness, are carbon fibers. Carbon fibers show outstanding tensile strength and modulus at high temperatures, chemical and thermal stability, low thermal expansion, and high electrical and thermal conductivity. Also, carbon fibers show good elastic properties that are not affected by fatigue deformations while loading and unloading are happening. According to fiber geometric characteristics, they can be divided into two groups: (1) fibers that are continuous in length and (2) carbon nanofibers [72], [73].

In **Paper II**, mechanical and long-term properties of carbon fiber reinforced geopolymer specimens were examined [47]. It was observed that 1 % of carbon fiber reinforcement introduction into the matrix slightly increases thickness of the geopolymer composite but does not significantly affect its flowability.

2.1.2.4. Polymeric fibers

As it is known, polymers are long chains of repeating monomers. They are held together through strong intermolecular bonds [74]. Depending on the chain order, polymers are divided into crystalline (over 80 % crystallinity), semi-crystalline (crystallinity between 10 % and 80 %), and amorphous (crystallinity is less than 10 %) polymers [64], [75]. When crystallinity is increased it also can enhance the polymer's mechanical properties, rigidity, environmental stability, and surface roughness. Based on the source material and the specifics of the production process, polymer fibers can be categorized as synthetic or natural.

Synthetic polymer fibers can be manufactured from raw material or recycled plastic waste. It has been claimed that the usage of recycled fibers in construction is a good solution for such widely used plastic disposal as polyethylene terephthalate (PET) and polypropylene (PP) [74]. For reinforcement of cementitious materials, the most widely used fibers are based on PP, polyvinyl alcohol (PVA), polyethylene (PE), and PET [76]. The main advantages of PP fibers are low cost, inert characteristics at high pH environment of the cement composite, ability to control plastic shrinkage-caused cracking in the concrete, and easy dispersion [77]. The downside for these fibers are low thermal resistance, low modulus of elasticity, and poor interfacial contact with cementitious matrixes due to their inherent hydrophobic characteristics [59], [78]–[80]. PET has similar mechanical properties as PP and nylon fibers. In contrast to previously mentioned fibers, PET fiber manufacturing is more cost effective and environmentally friendly. Recent developments in PET bottle recycling to produce PET fibers show promise for the construction industry [81]. The PVA fibers have higher tensile strength and modulus of elasticity than the abovementioned fibers. Those fibers also show stronger chemical bonding with cementitious matrix due to hydroxyl groups in its molecular chains [82], [83].

In **Papers III, IV, and VII**, PP and PVA fiber reinforced geopolymer composite's mechanical and long-term properties are researched [48], [49], [52].

2.1.2.5. Natural fibers

It is considered that jute, hemp, kenaf, bagasse and sisal are natural fibers. These fibers are common, widely available, and have low price. Furthermore, they have low density, reduced thermal conductivity, and good mechanical properties. The main drawbacks of these fibers are their low durability. At high fiber concentration, fibers significantly reduce the workability of the mix, and are the cause of inconsistent material properties and poor bonding/interaction with matrix [84]–[86].

2.2. Geopolymer composite properties

2.2.1. Mechanical properties

One of the most important and preliminary defining property of concrete is compressive strength. High initial compressive strength is of immense importance for construction materials. Therefore, flexural strength, tensile strength, and modulus of elasticity are closely linked with

compressive strength. Mechanical properties are greatly affected by the properties of the utilized source material. It has been found that the compressive strength of GPC is significantly influenced by the composition of the mix, the surface area, and particle shape of the fly ash [87]. The strength of GP composites is affected by many factors. The main factors are calcium content, molarity of alkali solution (NaOH, KOH), binder/aggregate ratio, solution/binder ratio, and silicate/hydroxide ratio. Furthermore, the development of strength is equally affected by the source material's properties, curing conditions, and filler particle size distribution [88]. It has been reported that heat cured fly ash-based GP composite achieves its complete compressive strength within one day. Other reports say that nearly 90 % of compressive strength can be achieved within a couple of hours if it is cured at temperatures around 80 °C to 90 °C. In **Paper II**, the geopolymer composite modulus of elasticity development was evaluated throughout the testing time. The specimens were polymerized for 24 hours at 75 °C. The alkali solution had 12 mol concentration. It was determined that as the tested specimens' age was 28 days, the modulus of elasticity from the 28th day till the 62nd day on average increased by 0.8 % each day and dropped the development to 0.4 % per day from the 62nd till the 150th day. Furthermore, the compressive strength had increased by 7 % and 27 % (for plain and carbon fiber reinforced specimens, respectively) from the beginning of the tests till the end. It was also determined that if the geopolymer specimen is subjected to water saturation for 24 hours, then its compressive strength decreases significantly. Compressive strength decreased by 2.2 % for plain specimens and 14.3 % to 35.1 % for carbon fiber reinforced specimens [47]. The strength development of GPC cured in ambient temperature is very close to OPC strength development. Nevertheless, all of the curing conditions result in long-term strength, and the additional heat to ambient temperature just speeds up the strength development and achieving of ultimate compressive strength for the specific GPC mix.

Some researchers have found that using 8-12M NaOH activation solutions and curing samples at 85 °C for 1 day results in specimens showing compressive strength from 35 MPa to 40 MPa. The compressive strength can be significantly increased if sodium silicate is added to NaOH ($\text{SiO}_2/\text{Na}_2\text{O} = 1.23$), reaching up to 90 MPa [89]. In **Papers IV and V**, the compressive strength values for the cylindrical specimens, made using 10M NaOH solution and cured for 24 hours in 75 °C, were from 30.37MPa to 36.33 MPa for plain specimens and 44.52 MPa for waste steel cord reinforced specimens [49], [50]. With the same NaOH solution and curing conditions, in **Paper III**, the compressive strength at the age of 28 days is 52.5 MPa, 55.1 MPa, 33.9 MPa, and 48.4 MPa for plain geopolymer composite, geopolymer composite with 1 % and 5 % PP fiber reinforcement and 1 % steel fiber reinforcement, respectively [48]. Specimens, made with the 12M NaOH solution, at the age of 28 days showed 48.16 MPa and 45.48 MPa for specimens with 1 % carbon fiber and without them (see **Paper II**). The curing procedure for these specimens was done for 24 hours at 75 °C [47]. The cubic geopolymer composite specimens mentioned in **Paper I** exhibit compressive strength values of 113.97 MPa, 81.07 MPa, and 85.2 MPa for 3.5 %, 2 % steel cord reinforced and plain geopolymer composites. These specimens were made using 10M NaOH solution. They were cured for 24 hours at 75 °C [46].

Water/binder ratio also plays a significant role in GPC compressive strength. It has been found that in order to gain the maximum compressive strength of fly ash-based GP composite, the optimal $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio has to be 15.9 [90]. It was also found that compressive strength was affected by the molarity of the activation solution. An increase was observed in compressive strength after 7, 14, 28 and 56 days when using 12–14 M activation solutions. The decrease in compressive strength was observed using activation solutions with molarities of 14 M and higher. The relations are showed in Fig. 4 [91].

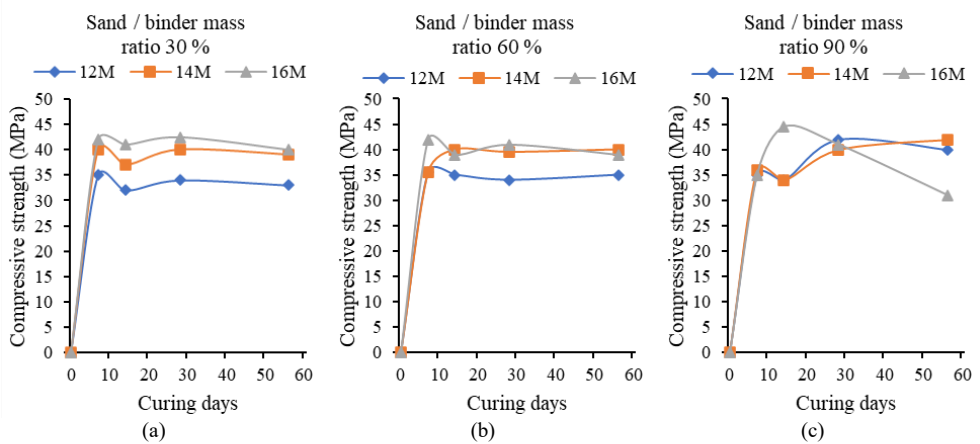


Fig. 4. Geopolymer mortar's compressive strength according to curing days, sodium hydroxide concentration, and sand/binder ratio [91].

In the early days of the GP composite research, tests were done with GP based on kaolinite, fly ash, NaOH, water and sodium silica mix. It was found that compressive strength was affected not only by the curing time but also by the curing temperature. Specimens cured at 60 °C for 48 hours had the highest strength (see Fig. 5) [92].

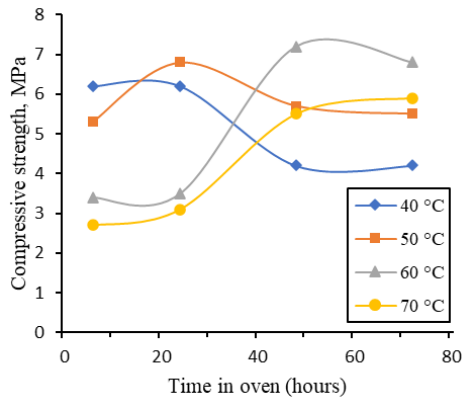


Fig. 5. Compressive strength development of fly ash-based geopolymer composite cured at different temperatures [92].

In Table 4, the important effect of variable parts on compressive strength on paste, mortar and concrete are summarized.

Table 4

Geopolymer Composite's Compressive Strength Variation Due to Different Variables [42]

Category	Source materials	Molarity	t (°C)	Curing time (h)	Compressive strength (MPa)
Paste	FA	12	70	24	65.0
	Fluidized bed FA-MK	10	40	24	72.0
	FA and SD	10	40	24	67.0
	RHA and WTS	10	60	24	24.0
	FA and coir fiber	8	75	24	31.4
	FA-MK-Sepiolite	10	20	24	52.0
	FA and POFA	14	65	48	72.7
	Nano TiO ₂ in fluidized bed FA-MK	10	40	72	77.0
FA and IOT	10	7 thermal cycles at 200	N/A	36.0	
Mortar	MK with CG	N/A	22	24	97.0
	GGBFS, MK and RHA	14	65	24	47.9
	FA	16	65	24	56.0
	FA	14	75	22	80.0
Concrete	FA	12	75	24	62.3
	FA	15	80	24	48.7
	FA	16	60	24	53.6
	FA	14	25	24	34.3
	BA	14	25	24	0.6
	FA and BA	14	25	24	8.4
	FA	14	46	24	42.5
	FA	14	46	24	40.6
	FA and LWA	12	70	24	17.0
	FA and KL	14	100	72	54.0
	FA and SF	14	100	72	51.2
	FA and nano-silica	12	25	24	46.4
	FA and nano-silica	8	80	24	47.0
	FA and LS	8	60	24	32.0
	FA and GGBFS	12	75	18	53.2
	FA with SSD	8	60	24	62.0
	FA, POFA, and OPS	14	65	48	30.1
	PCFA, GGBFS, and LWA	3	20	24	9.0
	POFA, MK, OPS, and steel fiber	14	65	48	31.9
	POFA, GGBFS, and MK	14	65	24	41.5

As for the tensile strength of geopolymer composites, in **Papers V and VI**, plain and 1 % PVA fiber reinforced specimens achieved 5.13 MPa and 4.95 MPa. These specimens were made using a 10 M NaOH solution and were cured at 75 °C for 24 hours [49], [51].

Regarding fly ash-based geopolymer bending strength with and without various fibers, similar as in **Paper VII** [52], plain and polymer fiber reinforced specimens show the best bending strength in contrast to specimens reinforced with low amount of steel fibers or with the combination of steel and PVA fibers. The bending strength for plain GP is 8.07 MPa and is followed by the 1 %PVA fiber, 0.5 % PVA/0.5 %steel fiber, and 1 % steel fiber reinforced specimens with a bending strength of 7.32 MPa, 6.93 MPa, and 6.20 MPa, respectively. Previously mentioned GP compositions with the best initial bending strength also have the largest deterioration of bending strength throughout the long-term deflection test. For the plain and 1 % PVA fiber reinforced, the flexural strength deterioration after the long-term test is 13 % and 15.15 %, respectively. For the remaining composites the reduction of bending strength is less than 1 %. Furthermore, the measurement errors of plain and 1 % PVA fiber reinforced GP are the largest. The errors vary from ± 0.49 MPa to ± 0.86 MPa for plain GP and ± 0.50 MPa to ± 0.70 MPa for 1 % PVA fiber reinforced GP.

2.2.2. Geopolymer composite durability

The resistance to weathering, degradation, abrasion, and chemical resistance are essential to any structural element. Absorption capability, noticeable volume of pours, water and chloride absorption, sulfate and/or other acid influence are important parameters that show GPC stability and suitability for structural use. GP composites usually show higher endurance to aggressive environment impact than OPC.

The highest durability issue arises from water absorption. High water absorption greatly reduces the compressive strength of GP composite [93]. Acid attacks, liquid absorption, strength degradation and weight loss, chloride ingress, discontinuous pores and voids, and wetting-drying cycles have relatively less impact on durability. GP composites are considered acid resistant. Nevertheless, acids can have a significant effect on GP composites, their durability, and composition stability. GP resistance to acids mainly relies on the acid properties and GPC specimen physical state and chemical composition [94]. Absorption ratios are highly important because the higher they become, the higher the capillary water level in GPC mix [95]. Strength loss mainly would be generated by imperfections in the chemical composition of the GP and reduced compactness that leads to reduced split tensile strength, flexural strength, and compressive strength [96]. Significant weight loss of specimens leads to durability loss mainly while specimens are submerged in solutions that hold sulphates from sodium and magnesium, sulphuric acid, and NaCl. In the beginning, it appears that the specimen's weight increases as the chemicals penetrate into a structure. Afterward, it is observed that absorption of the solutions have also led to the expansion of the specimen that further causes a significant amount of micro cracks that further reduce the durability [97]. Chloride penetration into the specimen would create the same results as weight loss. As the specimen is subjected to chloride solution, it would penetrate specimen and would

expand in the specimen that leads to micro and macro cracks that further significantly reduce the durability of the specimen [98]. Discontinuous pores increase GPC workability and resilience under severe environmental conditions [99]. Heating and cooling and exposure to moisture cycles influence the microstructure of the specimen and further affect mechanical properties. The higher the weight loss during these cycles, the greater reduction in durability of this specimen [100].

Shrinkage-introduced cracks in most cases have less impact on durability than the previously stated situations. They can be introduced during the polymerization process and through moisture cycles. In most cases, shrinkage would not cause a high amount of cracks, but if a high amount of shrinkage cracks are present, then the durability of the specimen can be significantly impacted and mechanical properties reduced [101].

2.2.3. Geopolymer composite's thermal endurance

Unlike OPC that would lose 20–30 % of strength if it is heated at 800 °C to 1000 °C, GPC shows different thermal properties in elevated temperatures. Geopolymer composites exhibit decent fire resistance properties at high temperatures due to nano-pore presence in the microstructure. The melting temperature of GP composites is about 1200 °C with no signs of spalling [102]. Micro-pores allow evaporation and migration of water vapor in the structure of GP without posing harm to the aluminosilicate network. While elevated temperatures are applied to specimens, various processes are happening, for instance, water that has been absorbed by N-A-S-H gel evaporates, water-free products are formed, melting and crystallization of stable water free phases that leads to further GP matrix destruction. It has been observed that if GP paste that contains fly ash is exposed to 800 °C, the residual strength is increased by 6 %, but metakaolin containing GP paste would show 34 % decrease in residual strength [103].

2.2.4. Time dependent properties of geopolymer composite

Creep and shrinkage can cause a significant increase in strain with time. Thus, for material's use in the long-term, it is of high importance that these properties are studied. Often creep and shrinkage are considered as independent phenomena, but there are some studies that claim that these phenomena have strong poromechanical (studies of porous material permeated by interconnected network of pores) couplings [104]–[106].

Nevertheless, there is still lack of information and research regarding creep and shrinkage behavior of geopolymer composites in various stresses, especially in tension and three-point bending.

Methodology for determining the long-term properties of concrete and cement composites in various stress-strain conditions (**Patent I**) was developed and verified not only for the purpose to measure creep strains but also to record shrinkage and modulus of elasticity changes [107]. It also regulates how to carry long-term tests in compression, tension and three-point bending. The developed method regulates the specimen's preparation, loading and unloading procedures, and length and environmental necessities for long-term property laboratory testing.

2.2.4.1. Creep properties

Creep is defined as a phenomenon where strain increases in time while the applied stress remains constant [108]. Creep is a very important factor in any cement-based composite. Creep affects stress distribution and deformations throughout the specimen's or the structural element's cross-section. A survey on concrete bridges in 2011 [109] showed the importance of reliable estimation of the basic creep compliance function of concrete to accurately predict the long-term deflection of the bridge over a 20-year period. Basic creep characterization of concrete is also important in the prediction of relaxation of prestressed cables to nuclear vessels [110].

Creep for hardened concrete most often is divided into two occurrences:

- (1) basic creep that happens while the specimen has constant moisture; this process is irreversible;
- (2) drying creep (also called Pickett effect or stress-induced shrinkage) happens due to drying and is partially reversible [111].

Furthermore, drying creep is attributed to a micro cracking effect due to the shrinkage strain gradient between the external and internal layers of specimen upon drying [112]. Basic creep can be divided into short-term and long-term creep, referencing it to different kinetics [112]–[114]. It has been reported that the main factors effecting creep for OPC based materials are mixture proportions, curing age, temperature and humidity of the surrounding environment, and the level of applied stress [115]. For geopolymer composites, it is expected that the same factors are affecting creep development. Creep curve is generally divided into the transient elastic deformation stage (instant creep), primary creep stage (decelerating creep), steady creep stage (secondary creep or isokinetic creep), and accelerated creep stage (tertiary creep or creep failure). They are dependent from creep rate and creep time relations, as it is shown in Fig. 6 [116]. Due to the nature of geopolymer polymerization process, the factors that affect OPC composite creep would have different effects on geopolymer composites.

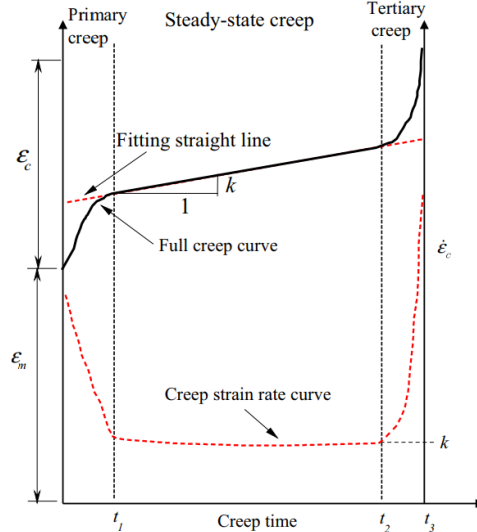


Fig. 6. Schematic diagram of a usual creep curve and creep strain rate [116].

As geopolymer binder is a fairly recently developed material, there has been increased interest in creep properties of geopolymer in recent years. It has been showed that fly ash-based geopolymer concrete exhibits low creep values over a 12-week testing period [19]. Furthermore, it has been found that fly ash-based geopolymer has lower specific creep (creep strain normalized with applied stress) [20] and creep coefficient (the ratio between creep strain and elastic strain) [20], [21] than OPC concrete. This matches with the results in **Papers II–VII** [47]–[52], where it was observed that creep properties for plain specimens are low, and in **Paper II** [47] it was shown that they are lower than for OPC by around 13 % to 23 %. It was further concluded in **Papers II and III** [47], [48] that insufficient amount of fiber incorporation into geopolymer composite would lead to higher creep strains. Still, if the reinforcement is in sufficient amount and has the necessary properties for the specific service state, the creep is limited. In **Paper IV** [49], recycled tyre cord reinforcement decreases creep strains by 30 %. It has been claimed [22] that this low creep is attributed to a micro aggregation effect of unreacted fly ash in the binder.

Most of the studies that have looked into the creep properties of the geopolymer composites have determined creep in compression [23]–[25], [101]. In general, the fly ash-based GP composite would have a lower creep than OPC based concrete. It has been claimed that GP concrete cured at 60 °C for 24 hours has up to 50 % less compressive creep than OPC based concrete [22]. Similar creep properties have been observed for the specimens cured for 6 hours at 60 °C [101]. There have been creep measurements for GP composites cured from 40 °C to 80 °C for different durations. It was observed that GPs that have been cured at 40 °C for three days have insignificant or the same creep properties as OPC based specimens, but compressive creep strains of GP specimens that have

been cured for 7 days at 80 °C have been significantly reduced and got negligible [20]. In **Paper II**, up to 23 % reduction in creep strains was observed for the geopolymer composite that has been cured for 24 hours at 75 °C [47].

The creep in tension has a different manner than creep in compression. Tensile creep has a critical role in assessment of the early age cracking risk [117]. Tensile creep shows more linear behavior throughout time than creep in compression. The development rate of creep strain in compression throughout time would decrease. Some researchers claim that creep coefficients measured for compressive stresses can be multiplied by factor 1 to 3 to get creep coefficient in tension [118]. The mechanisms of creep in tension are different to those in compression. The tensile creep strain rate does not reduce at the same rate as the creep strains in the compression [119]. Tensile creep determination for concrete at early age can be done using the direct tension experiment or the indirect tension test [120], [121]. The ring test is a simplified indirect tension method that is quite popular to determine tensile creep and restrained shrinkage cracking risk [122].

Some researchers [123] claim that at an early age, creep in compression for cement composites would be bigger than creep in tension, while others [124] claim the opposite. In **Paper V** [50], it was determined that creep strain for plain geopolymer composite in compression is around 35.8 % higher than in tension. Furthermore, creep strains in compression develop and increase throughout testing time, the creep strains in tension do not exhibit significant increases throughout test. As for the specific creep values, here significantly, specimens that have been tested in tension show higher values. They have, on average, 85.92 % higher specific creep than the specimens tested in compression, as shown in Fig. 7.

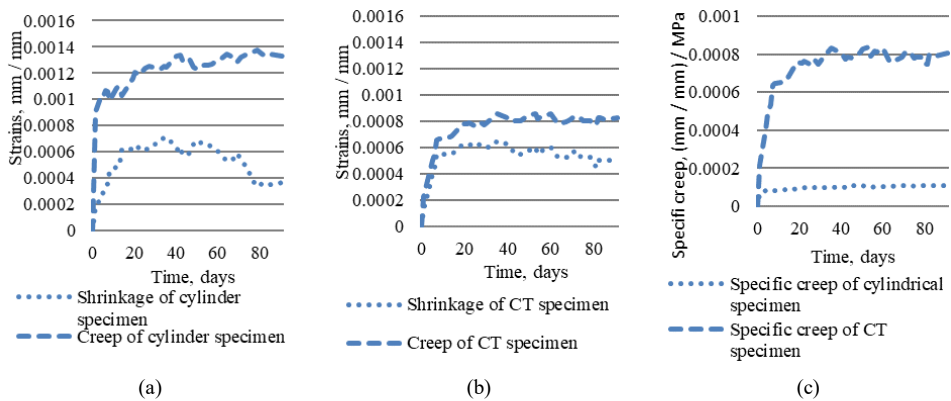


Fig. 7. Creep and shrinkage curves of compression (a) and tension (b), and specific creep (c) of compression and tension of specimens [50].

In **Paper VI** [51], it was determined that 1 % PVA fiber reinforced geopolymer composites exhibit similar creep strains as plain GP. Tensile strength also is similar. It also has to be mentioned that the 1 % PVA fiber reinforced GP specimens have larger creep strains in the early stage, but

after 40 days of testing, creep strains are equal to the plain and 1 % PVA fiber reinforced specimens.

Just like the OPC based concrete, also GP concrete shows low tensile capacity. The authors claim that for reinforced GP structures it is inevitable to show cracking in service life [119]. At the early stage of structure's life, a restrained shrinkage is one of the factors that induces early age cracking. The evolution of restrained shrinkage in the first few days would introduce time dependent tensile stress development in concrete [125]. In the concrete member that has been restrained by reinforcement, the shrinkage increases the tensile stress while tensile creep is relaxing tensile stress. All of the previously referred studies are focusing on quantitative analysis of experimental data but information regarding creep and shrinkage in GP is limited.

The long-term deflection properties of geopolymer composites are not widely studied. Some of the studies [126], [127] that looked into long-term properties of specimens tested in deflection show that geopolymer composites have close correlation with the Portland cement based composite long-term deflection properties. **Paper VII** [52] shows similarities with [126] the acquired deflection characteristics. As for the quantitative comparison, it is impossible to compare the results presented in **Paper VII** with other results, for instance, the results published in [126] where the deflection strain graphs are represented using absolute strain measurements not relative (measurements not dependent from the measured specimen's size and applied load impact) strains. From the results in **Paper VII** [52] it becomes clear that the usage of steel fibers is beneficial in contrast to the PVA fiber or combined PVA and steel fiber usage. Furthermore, while creep properties of geopolymer composites reinforced with 1 % steel fibers in compression and tension show the highest creep and specific creep properties in long-term deflection, they show the lowest long-term deflection that is 39 % less than the next GP composition.

In **Patent I**, the test setup and procedures necessary for creep testing in compression, tension and bending are described.

2.2.4.2. Shrinkage properties

Shrinkage of concrete materials in general comes from the shrinkage of the paste (matrix). It is due to high dimensional stability of the aggregates [128], [129]. The shrinkage of materials can be divided into groups based on mechanisms that influence shrinkage development. These are plastic shrinkage, carbonation shrinkage, autogenous or chemical shrinkage and drying shrinkage. Plastic shrinkage happens instantly when mixture is poured into mould. Carbonation shrinkage happens due to CO_2 from the environment penetration into the outer surface of the specimen and reaction with Ca(OH)_2 from the hydration products. That further produces CaCO_3 and water molecules. Autogenous shrinkage is caused by self-desiccation of the composites. It is an unavoidable process that reduces volume of the element and is very likely to happen to composites with low water/cement ratio [130]. Drying shrinkage is the volume reduction of the specimen that is caused by water evaporation from the gel pore surface. Most of it happens in the hardening process of the specimens [131]. For geopolymer composites and alkali activated composites in general, the main shrinkage comes from autogenous and drying shrinkage. For geopolymers, autogenous shrinkage

means loss of internal water during polymerization that further creates capillary stress. Drying shrinkage happens due to water evaporation into the environment, and it is proportional to the moisture loss from the composite [128], [132]–[134]. In contrast to autogenous shrinkage, drying shrinkage is more affected by alkali concentration and outside environment effects. The tests done in [26] showed that the geopolymer binder drying shrinkage increased with the increase of alkali concentration (Na_2O). In the meantime, autogenous shrinkage stayed relatively constant, as is shown in Fig. 8.

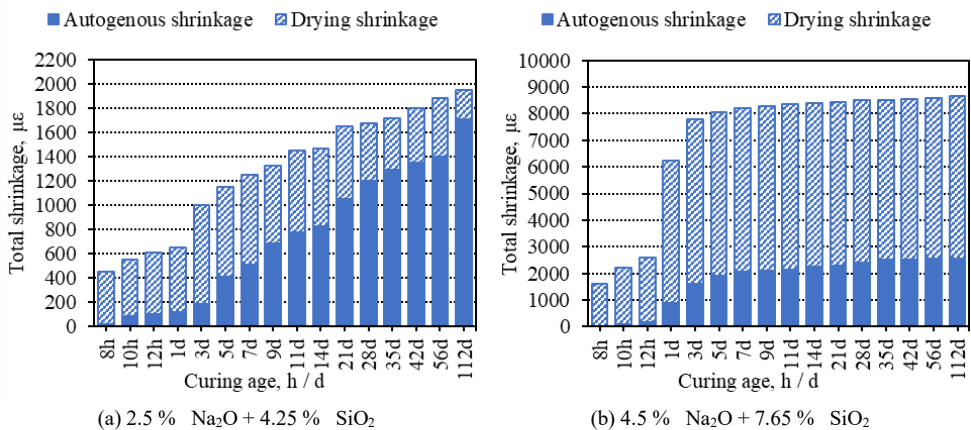


Fig. 8. Autogenous and drying shrinkage of alkali activated composites with different alkali concentration [26].

Other researchers state that it is hard to accurately determine the level of autogenous and drying shrinkage from one another for the composites mainly due to the fact that autogenous and drying shrinkage happen simultaneously if the specimen is not sealed. Therefore, measured drying shrinkage often has a fraction of autogenous shrinkage as well. The shrinkage, especially for alkali activated composites, is closely linked to polymerization process and used raw material properties that greatly influence the moisture loss and the pore structure development of the composites [27], [128], [135].

In **Papers II–VI** [47]–[51], drying shrinkage strains were measured. It was determined that fiber reinforcement plays a significant role in shrinkage reduction for specimens tested in compression and tension. For instance, in **Paper IV**, specimens that have been reinforced with recycled tyre steel cords show on average 50 % less shrinkage than plain geopolymer composites [49]. Furthermore, in **Paper VI**, the 1 % PVA fiber reinforced specimen tested in tension showed on average 54.21 % lower shrinkage than plain specimens [51]. Also, in **Paper V**, shrinkage strains were determined in compression and tension [50]. There shrinkage strains between compression and tension specimens on average were 15.8 % less for the specimens meant for testing in tension.

The mechanisms that influence shrinkage can be divided into four groups: capillary stress, interlayer water loss, disjoining pressure, and surface energy [133]. These shrinkage mechanisms influence cement-based materials and alkali activated cement-based composites. While mechanisms are the same, the shrinkage values for the OPC based and geopolymer composites differ, mainly due to differences in hardening reaction mechanisms [136]. In general, the alkali activated cement composites, slag-based composites in particular, show larger shrinkage than OPC based composites. This geopolymer shrinkage increase is mainly due to the differences in pore size distribution and reaction products in the paste mix. These results of the previously mentioned research are based on quantitative analysis of experimental data.

In **Patent I**, the test setup and procedures necessary for drying shrinkage testing in compression, tension and bending are described.

3. GEOPOLYMER MICROSTRUCTURE

3.1.Characteristics and processes of alkali-activating reaction

Some studies have shown that the alkali-activated reaction of fly ash-based GP is affected by certain factors, such as the fineness of fly ash particles, curing conditions, and alkali activation solution concentration [137]–[141]. The dissolution process of Al and Si happens when fly ash is subjected to alkali solution. Larger molecules condense into a gel (polymerization and nucleation), and as the alkali effect on fly ash continues, larger spheres open to reveal smaller spheres inside. Smaller spheres then would be dissolved almost fully with the formation of reaction products inside and outside the large spheres. The AlO_4 and SiO_4 formation by silicon and aluminium-rich fly ash dissolution forms three types of monomers by combining with oxygen atoms. Then, these three types of monomers form a gel with a simple structure. After that, the gel is restructured and then polymerized and hardened in the disordered structure with high mechanical strength and forms an alkali-activated cementitious material [142]–[147]. The polymerization process is shown in the scheme in Fig. 9.

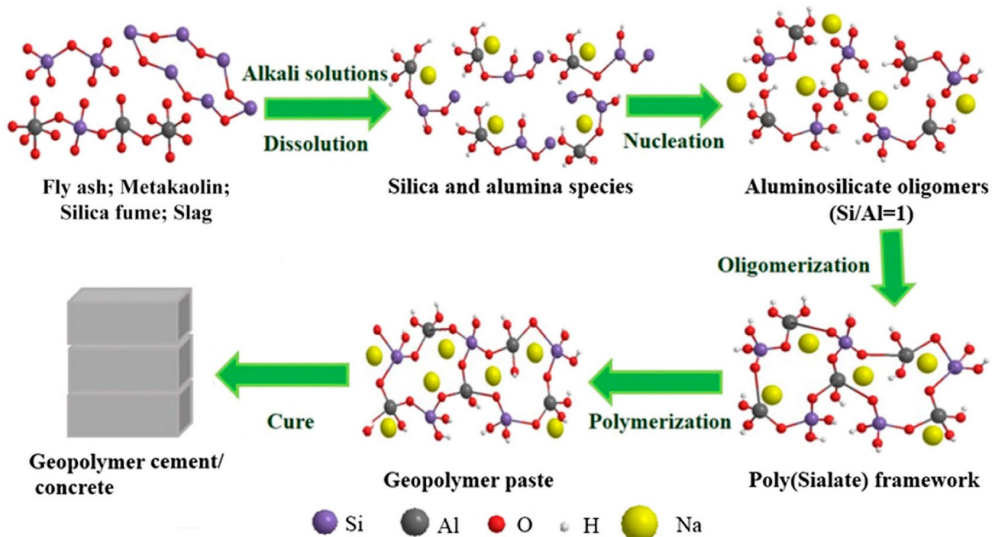


Fig. 9. Fly ash-based geopolymer cement/concrete chemical development scheme[148].

The influence of the fly ash particle size on the polymerization reaction shows significance when the reaction temperature is low. Fly ash with a smaller particle size has higher solubility and stronger reactivity.

3.2. Pore structure of the fly ash-based geopolymer

In general terms pore structure in fly ash-based GP composites is affected by curing conditions. Geopolymer pores are mainly within the small pore size range. This is unlike OPC based composites. There the hydration products do not have a filling or optimizing effect on the pores. Some researchers [149] observed fly ash-based GP gel pores and capillary pores that have been cured for 7 and 28 days and concluded that pores were concentrated in the small pore size range. That differs from OPC composites. Furthermore, in the fly ash based GP, the C-A-S-H gel was distributed uniformly, and no capillary pores were found in it. Nevertheless, a small amount of large cavities from partial fly ash particle dissolution were observed. In OPC based composites, C-S-H gel was concentrated, and capillary pores were formed. In other studies [150] it was observed that the pore volume increased and morphology varied significantly when the setting time was increased for OPC composites. It was determined that the main factor could be the expansion of the basic silica gel and the merging of pores. Fly ash based composites showed the filling effect of the fly ash particles and gel. This contributes to negligible variations in pore volume and morphology. Furthermore, variations in micropore diameter and volume were linked to the polymerization level of fly ash during curing.

Also, silicon content in the alkali activator plays a crucial role in the pore structure development of fly ash-based GP. A larger quantity of silicone contributes to the larger quantity of gel and contributes to the uniform distribution of the gel with the pore filling effect at the micro scale [151], [152]. An alkali activator with high silicon content contributes to the formation of uniform and dense microstructure [149]. Despite this, in [153] it was determined that high silicon content in alkali solution does not influence the refinement of pore structure of fly ash-based GP. It was pointed out that the pore size distribution was concentrated within the sub-mesoporous region (0.22–3.6 nm), and the pore network was more refined when the silicon content in alkali solution was low. Pore distribution in mesoporous region was from 3.6 nm to 50 nm, and the pore refinement degraded when the silicon amount in alkali solution was high. Furthermore, longer curing time can promote formation of the N-A-S-H gel with pore filling effect and even more reduce porosity [154], [155]. To a certain extent the volume of capillary pores decreases with the increase of curing temperature [155].

There are many possibilities to measure the porosity of the material. The most often used ones are [156]:

- mercury porosimetry,
- helium pycnometry,
- image analysis,
- water absorption.

To determine porosity, in **Papers IV–VII**, a technique for outside effect determination on concrete and cement composite microstructure in various stress-strain states (**Patent II**) [157] was developed and used. The technique is based on polished section specimens that are examined in

the scanning electron microscope (SEM) or optical microscope with a specific image recording equipment. The acquired images from polished sections are compiled and rendered and quantitative image analysis is done. This technique (**Patent II**) can be used not only for porosity determination but also for the specimen cross section characterization purposes. It details and regulates the acquiring and storage of specimens. It also regulates the specimen preparation procedures and image acquiring sequence and recommends the best ways to render and quantitatively analyse images and their composition.

In **Papers IV–VI**, it was determined that fiber incorporation into geopolymer composite leads to increased porosity. Porosity was increased by 0.61 % and 2.26 % in the specimens reinforced by the steel fibers from recycled car tyres [49]. Also, it was determined that specimens that have been subjected to load (tested for creep properties) had around 1.17 % less air voids than the specimens that had not been subjected to load. For the specimens without reinforcement, no significant reduction of air voids due to loading was established. In **Paper V**, it was determined that the specimens meant for testing in tension had a significantly higher air void amount than the specimens used in compression tests [50]. The specimens used in tension have a 2.14 % and 3.01 % higher amount of air voids than the compression specimens. Also, as these are plain specimens, the difference between the specimens meant for compression testing and those that have or have not been subjected to load has not been established. For the specimens used in tension testing there is a difference in porosity. There is a 0.69 % reduction for specimens subjected to load. Furthermore, in **Paper VI**, it is determined that for the specimens used in tension, the testing fiber introduction significantly increases the air void amount [51]. In specimens with a 1 % PVA fibers, the air void amount is increased by 1.88 % and 2.50 %. In a three-point bending for the long-term deflection tested specimens, the differences in air voids between plain, 1 % PVA, 0.5 % PVA/0.5 % steel and 1 % steel fiber reinforced specimens is within a 1.37 % difference, as it is shown in **Paper VII** [52]. The highest air voids are found in plain specimens with 5.02 % of analysed surface area, and lowest in 0.5 % PVA/0.5 % steel fiber reinforced specimens with 3.65 % of the analysed surface area consisting of air voids. Significant differences become apparent when the specimen's cross section surface analysis is done for the specimens tested for bending strength. Then the air void and crack area differ from 14.50 % and 16.48 % for 0.5 % PVA/0.5 % steel and 1 % steel fiber reinforced specimens up to 18.57 % and 22.00 % for plain and 1 % PVA fiber reinforced specimens. This leads to the conclusion that for thin specimens with large surface area, fiber incorporation allows an entrapped air release.

3.3. Geopolymer and OPC composite microstructure differences

As it was mentioned in the previous section, hardened GP shows a denser structure than the cured OPC composite. In GP system, C-A-S-H matrix chains have been found to be longer than the C-S-H gel chains in OPC systems. It is mainly because of Al^{3+} substituting Si^{4+} in bridging positions. C-A-S-H gels showed a lower amount of Ca/Si ratio and a higher ratio of Al/Si than C-

S-H. There were indications of tobermorite 1.4 nm existence with a chain length of 11 nm and tobermorite 1.1 nm with a chain length of 14 nm tetrahedra. Furthermore, the modulus of elasticity for tobermorite 1.1 nm was 77.3 MPa and for tobermorite 1.4 nm – 49.9 MPa. This is because of higher interlayer cohesion of bonding between bridging tetrahedral of conservative layers [158].

If the differences in gel composition of GP and OPC composite systems are compared, the main difference is that the C-S-H forming in OPC composite shows lower Al and higher Ca content than the formation of C-A-S-H in slag-based GP composites. If the fly ash is introduced in the system of GP, N-(C)-A-S-H can be identified with low Ca content, but N-A-S-H can only be stable at pH lower than 12 [159], [160].

The slag-based GP pore size varies in the field of mesopores (pore size <50 nm). These GPs also show lower porosity than OPC based composites. If the pore size distribution is compared to OPC based paste and slag based GP paste, it becomes apparent that most of the pores for the OPC based paste are within a range of 10 nm to 100 nm, while for the GP paste, most of the pores were below 20 nm [161]–[163].

3.4. Microstructure development of geopolymers composite incorporating lime and silica

As fly ash stocks in the future can be exhausted and necessity of elevated temperatures for such GP polymerization can be an overwhelming boundary, other alternative source materials have been tested as more suitable base materials for GPs. By introducing lime and silica fume in the GP matrix [164], compressive strength and microstructure for 7-, 14-, and 28-year old specimens were determined. The researchers replaced fly ash partially with lime and silica fume within the range of 5–10 % and 1–3 %, respectively. It was found that by partially replacing fly ash with lime, the setting time and workability of such material would be reduced, while the usage of silica fume would increase these properties. The compressive strength for these specimens increases if 7.5 % lime and 2 % silica fume is used. It was also observed that at the age of 7 days specimens show a less homogeneous microstructure with some unreacted fly ash particles. It was deduced that fly ash reacted throughout time as polymerization continued. At the age of 28 days the microstructure appeared homogeneous and compact. Microstructure appearance directly contributed to the compressive strength values.

4. GEOPOLYMER COMPOSITE APPLICATION IN CIVIL ENGINEERING

High compressive strength, higher durability to acid attacks and thermal durability, low carbon emissions and low processing energy consumption and others are the properties that justify the GP application in civil engineering in contrast to conventional Portland cement and other cement-like materials that have lime in them.

4.1. Geopolymer application in soil stabilization

It has been claimed that GP usage in soil stabilization is feasible especially on sites where there are soft or weak soils [165]. Some authors [166] have found that very effective in the stabilization of deep soil are fly ash slurry. It has to be pointed out that these slurries are calcium-based GPs. Others [167] have deduced that alkali activated metakaolin shows promising results in soil stabilization in contrast to cement-stabilized soil. It was found that in an unconfined compressive strength test at the age of 28 days metakaolin (15 %) stabilized soil had 4 MPa, while the cement (5 %) stabilized soil strength was 3.5 MPa and non-treated soil showed 0.5 MPa strength. Soils that have been stabilized using metakaolin appear more ductile [168]. These soils show lower shrinkage. It is significant at 11 % metakaolin concentration.

Researchers have used Class F fly ash (with low Ca content) together with alkali solution (with sodium hydroxide and sodium silicate). The alkali solution was prepared in 10 M, 12.5 M, and 15 M concentrations. At the early age, the 15 M solution showed higher strength, but at the age of 90 days and 365 days, 12.5 M concentration was much higher. Alkali solutions with concentration of 15 M handled several times were too viscous to handle and crystallized at lower temperatures. Furthermore, as the sodium silicate and sodium hydroxide are quite expensive, the 12.5 M solution seems more suitable from this standpoint [166].

It has been concluded that the usage of alkali activated compound in jet grouting is suitable for soil stabilization and comparable to traditional cementitious grouting methods, however the strength development has to be researched further [169].

4.2. Geopolymer application in buildings and infrastructure

There have been various claims of the first building in the world that would have its structure entirely made from GP. Some authors claim that it is the Global Change Institute of the University of Queensland building (Fig. 10) that was constructed in 2013 by HASSEL in conjunction with Bligh Tanner and Wagners [170]. The building is made from precast elements. They were made from slag/fly ash-based GP, called earth friendly concrete (EFC) that is the Wagners brand name for their commercial form of GP concrete.



Fig. 10. The Global Change Institute of the University of Queensland building: (a) one of 33 floor panel montage and (b) finished building [170].

Other researchers claim that the first two buildings were built in the 1960s in Ukraine, Mariupol. Two 9-storey residential buildings were constructed from alkali activated material developed by the Ukrainian scientist Glukhovsky. This material had some Portland cement in it. The first building made from alkali-activated concrete without Portland cement was built in 1989 in Lipetsk, Russia, and it had 20 floors [171].

Besides the previously mentioned cases there have not been any claims of other residential buildings made from GP composites.

In contrast to applications in residential buildings, the use of GP composites for infrastructure purposes is much more common. For instance, in Australia, the Rocla Research Center has produced and successfully implemented sewer pipes, railway sleepers, cemetery crypts, box culverts, and wall panels [172]. In 2015, Wagners Australia and Glasby documented large scale commercial application of GP concrete at the Brisbane West Wellcamp Airport. Approximately 40000 cubic meters of fly ash-based GP concrete was used to make 435 mm thick heavy duty pavements in the northern end of the runway, aircraft turning areas, taxiway on the western side, and hangars on the eastern side of the runway [169]. In India, there has been a successful experience with pavement stone development and usage on paved road surfaces [173]. It was concluded that the fresh properties of GP composite (slump value and spread) were equivalent to the concrete guidelines. Additionally, strength values were greater than the target values.

Dense microstructure of GP composites results in low permeability that further reduces and inhibits penetration of seawater, thus making GP composites suitable for marine applications [174]. The authors of [175] have claimed that aluminosilicate geopolymeric gels are chemically stable in sea water and can be a sustainable alternative to Portland cement based marine structures. Some researchers [176] have proposed the use of fly ash and steel furnace slag as source materials for high density GP composite for coastal protection structures. The composites achieved a compressive strength value up to 37 MPa and a size reduction of breakwater structures by 30 % to 40% was proposed without compromising the structural performance. This resulted in the reduction of material requirements that further reduces overall carbon footprint. The authors of [177] have

had similar reports on enhanced properties of fly ash-based GP composites exposed to harsh environments such as sea water and acidic environments.

Also, in India they have had success with GP concrete road creation. In 2017, at the Council of Scientific and Industrial Research in the Central Building of the Research Institute in Roorkee, a 50-meter long and 3-meter wide road segment was laid. In Uttar Pradesh, at NTPC Energy Technology Research Alliance, a 100-meter long and 6.5-meter wide road segment was laid. In 2017, several haul roads were laid for heavy load carrying from the mines in Bhubaneswar. In Lanjigarh, Sesa Sterlite Ltd in cooperation with IMMT laid a 40-meter long GP road. In late 2019, in Ramagundam, NTPC in association with NETRA laid a 500-meter long fly ash based GP concrete road [178].

All in all, the GP technology is more advanced and technically more applicable in precast state. The main factor is that GP is quite sensitive while it is setting (polymerizing). It is easier to provide a high temperature curing environment and safe alkali processing environment in specific location than on a building site and have all of the necessary technological means moveable. This can be concluded for the developed and tested geopolymer compositions in **Papers I–VII**. As all of the tested geopolymer specimens were developed, prepared and tested in laboratory conditions, the acquired properties are only possible in certain conditions. These conditions are molded structures and polymerized in a controlled environment. Therefore, the developed geopolymer composite compositions in previously mentioned papers would be suitable for precast structure development. Also, it would be significantly easier to abide health and environment requirements in a precast element factory than on site, as the alkali solution is caustic and hazardous to the environment.

If today's GP manufacturing technology had to be made mobile, the on-site GP construction cost would be unaffordable and GP usage financially unreasonable. The only place where low calcium GP composite usage on building sites seems feasible is in the countries and at time when the average daily temperature is high to sustain the polymerization reaction of the GP composite.

4.3. Geopolymer application as mortars

Geopolymer mortars possess similar properties as natural rocks like granite and marble. Geopolymer composite's durability characteristics, especially in harsh environments, poses it as an alternative to conventional cement mortars in building restoration. Researchers [179] have reported that metakaolin based GP with calcium carbonate (CaCO_3) and calcium hydroxide (Ca(OH)_2) in it can be used as mortar for the restoration purposes of historical buildings.

In 2012, Zeobond reported the use of a commercial GP concrete ready mix with steel reinforcement for the creation of slabs and footpaths in Melbourne (Australia). In 2011 and 2012, Zeobond and Rocla produced and tested according to Australian standards precast pipes, railway sleepers, and pavers and installed them in several construction projects [169].

4.4. Geopolymer application as fire resistant layer

In recent research, scientists investigated GP performance under elevated temperatures and compared it with OPC concrete specimens. They used Class C fly ash that was activated with the solution consisting of sodium hydroxide and sodium silicate. Both GP and OPC concrete specimens were exposed to temperatures up to 1200 °C. The OPC based specimens showed severe cracking at temperatures above 800 °C while GP specimens showed an insignificant amount of visible cracks and no spalling even at high temperatures. The residual compressive strength for OPC based specimens (49 MPa) was lower than for GP specimens (54 MPa). From these results it was concluded that GP is suitable to be used as fire resistant coating or as whole material for structures where fire resistance and structural performance are crucial [38].

4.5. Geopolymer application as insulating layer

Researchers [180] have found that when metakaolin-based GP matrix is mixed together with sawdust, the resulting material can act as effective insulation material for buildings. If the water to biomass ratio is up to 2, then the material exhibits low heat conductivity (0.118–0.125 W/mK). This material poses potential for practical application as building insulation.

As all of the geopolymer compositions that have been subjected to microstructure assessments (in Papers IV–VII) have porosity and air void amount less than 10 % in uncracked state, they would not be suitable to be applied as an insulating material. Thus, there have been various studies with similar base compositions with the addition of foaming agent that have found it suitable for insulation purposes.

5. GEOPOLYMER COMPOSITE LONG-TERM ENVIRONMENTAL INFLUENCE

5.1. Environmental assessment of geopolymer long-term impact on the environment based on life cycle assessment

Life cycle assessment has been the main analytical mechanism that till today has supported valid claims that GP is an environmentally friendlier material than OPC based composites. A research done in Australia [181] looked into the hybrid life cycle assessment (hLCA) of greenhouse gas emissions from cement, concrete, and GP concrete.

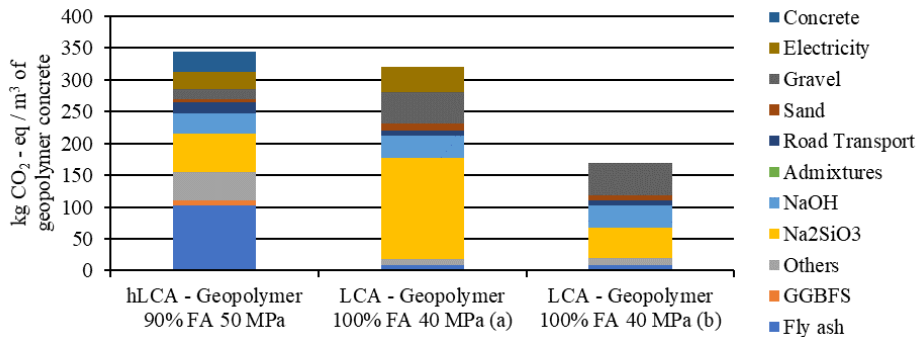


Fig. 11. Comparison of fly ash geopolymer concrete carbon footprint intensities of hLCA and LCA, made by the authors in [7] and [182], based on [7].

The researchers concluded that unlike life cycle assessment research that had previously been conducted by Davidovits and Grant hLCA (Fig. 12) shows an increase in greenhouse gas emissions for 50 MPa OPC concrete: 25 MPa OPC concrete, 25 MPa blended cement concrete, and 50 MPa GP concrete by 29 %, 22 %, 11–50 %, and 48–103 % (depending on emission allocation with sourcing of FA), respectively (Fig. 12).

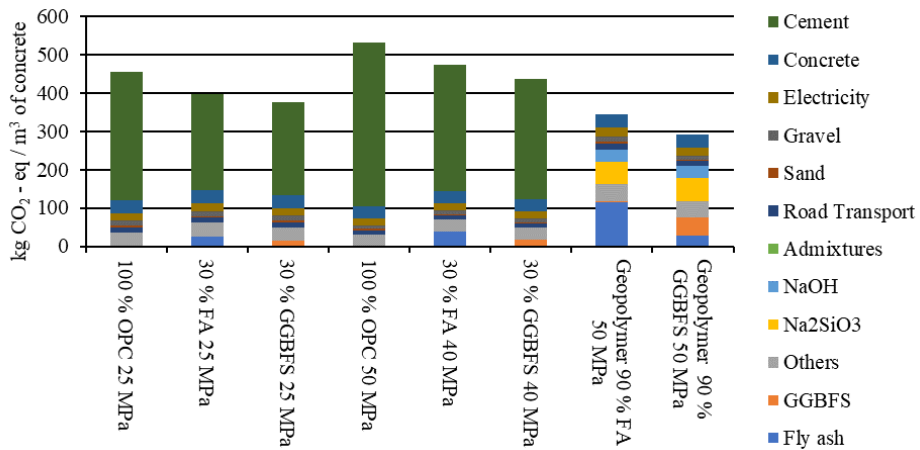


Fig. 12. Carbon footprint of 8 types of concrete according to hLCA [181].

Furthermore, it was concluded that the findings go hand in hand with other life cycle assessment research findings. In other words, GP concrete has the greatest potential to reduce greenhouse gas emissions in comparison to OPC and blended cement-based concrete. Fly ash and ground granulated blast furnace slag-based GP concrete can substantially reduce greenhouse gas emissions by 32 % and 43 %, respectively, as compared to OPC concrete with no loss in compressive strength using the economic allocation method. When compared with blended cement concrete, ground granulated blast furnace slag-based GP (16 %) has a higher reduction of greenhouse gas emissions than fly ash based GP (just 9 %). Other research [183] has a similar conclusion – GP has a 37 % lower global warming potential than OPC. It was also concluded that GP concrete shows worse results regarding energy requirements for its production. GP concrete shows a 287 % greater use of fossil fuel resources than OPC based concrete. It has to be mentioned that in this regard the literature is not consistent because some authors [184] claim that energy consumption related to GP concrete production is 48 % higher than for OPC concrete production, others [40] report that abiotic depletion of fossil fuels (ADPF) for GP concrete is 26 % lower than for OPC based concrete.

The authors in [185] have made similar life cycle assessment evaluations of GP concrete and OPC concrete usage impact on the environment. They concluded that considering factors such as ecosystem, human health and resources GP concrete shows less negative impact on the environment than cement-based concrete. The whole situation is represented in Fig. 13. Cement in concrete makes the greatest impact (76.42 %) of all ingredients in the concrete, while sodium hydroxide and sodium silicate in GP have only a combined impact of 59.97 % of all GP ingredients. If the sodium silicate is replaced with silica fume, then the impact is even more reduced. The global warming potential for GP concrete is reduced to 148 kg, 135 kg, and 133 kg of CO₂-e for GP without silica fume, GP with silica fume, and GP with silica fume and 47.61 % reduction of sodium

silicate, respectively. In the meantime, cement-based concrete has 597.54 kg of CO₂ impact. It was also found that the impact of GP concrete on the environment is more affected by the transportation of raw materials than of ordinary cement concrete. For GP concrete it varies from 20.83–29.01 %, while for cement-based concrete it is 9.71 %. Researchers also claim that replacing cement with GP in concrete can reduce the costs of concrete by 10.87–17.77 %.

All in all, the environment gains from using GP composites instead of OPC composites lie in material location. The GP, researched in **Papers I–VII**, would be less environmentally damaging if they were used near the source of fly ash and alkali manufacturing facilities. Otherwise, all the gains from fly ash utilization in GP are lost by shipping impact on the environment. As fly ash is considered a waste material, it has to be used in areas where this waste is located or as close to the location as possible. The only way that the GP composite usage would be justified would be in the structures where extra resistance to the acidic or harsh outside environment impact is necessary. Otherwise, GP composites usage would have a higher negative impact on the environment than that of OPC based composites.

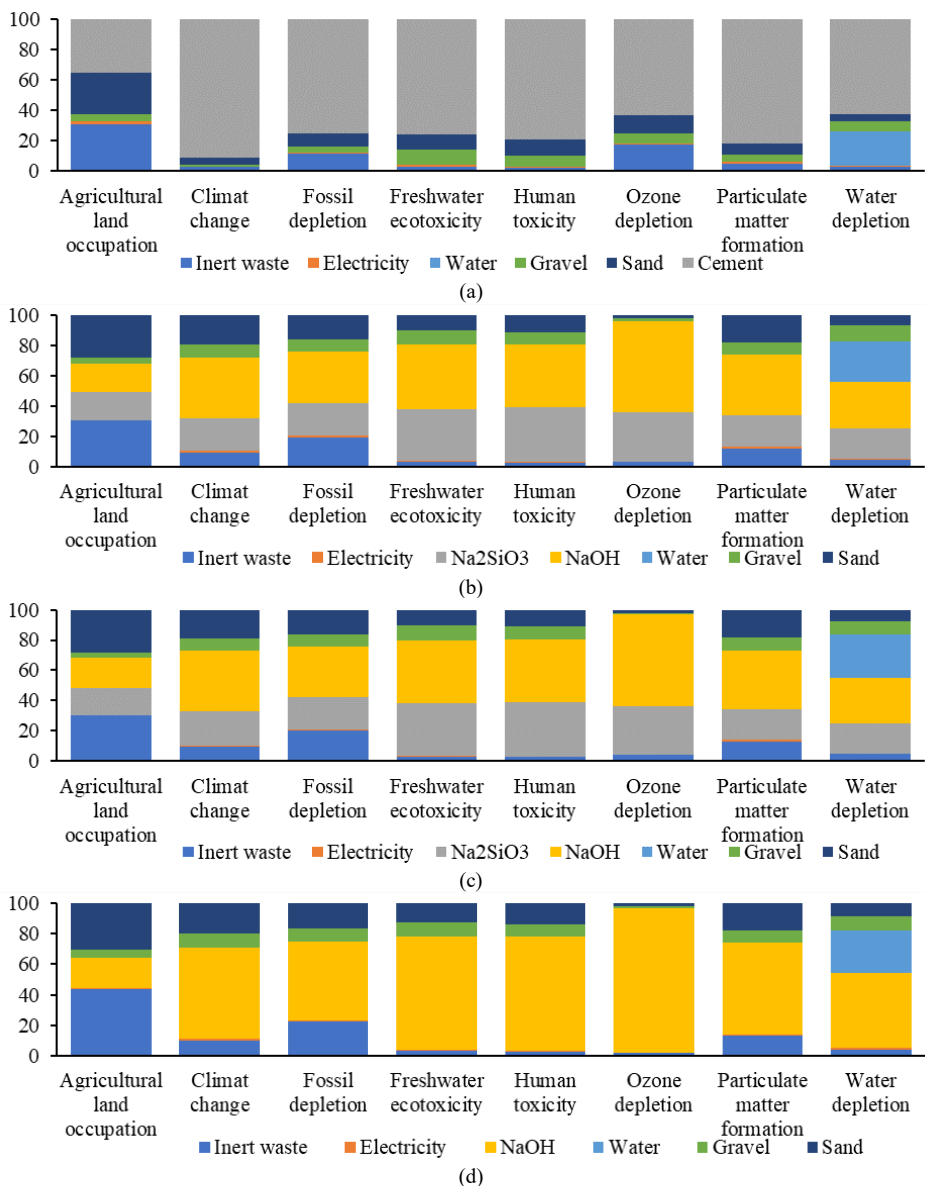


Fig. 13. Environmental impact from the ingredients of cement-based concrete (a), geopolymer concrete (b), geopolymer concrete with silica fume and sodium silicate (c), and geopolymer concrete with silica fume and without sodium silicate (d) [185].

CONCLUSIONS

The Thesis focuses on methodology development for long-term deformation influence assessment on innovative cement composite microstructure. The main conclusions are:

1. Geopolymer composites based on fly ash show remarkable mechanical properties. Plain geopolymer composites at the age of 7 days show compressive strength equal to C30/37 class Portland cement concrete. At the age of 28 days, plain geopolymer composites exhibit compressive strength not lower than C40/45 class Portland cement concrete, reaching up to 61.44 MPa for cylindrical specimens and 92.0 MPa for cube specimens.
2. Fiber reinforcement introduction into geopolymer composite is not beneficial in all cases. For compact tension specimens, the 1 % PVA fiber introduction led to 3.7 % lower tensile strength. In three-point bending, 1 % PVA fiber, 0.5 % PVA/0.5 % steel fiber and 1 % steel fiber reinforcement introduction led to 9 %, 14.2 %, and 23.2 % flexural strength reduction, respectively.
3. Fiber reinforcement introduction to geopolymer composite for creep strain reduction is beneficial in the case of flexural stress. Geopolymer composites with 1 % steel and 0.5 % PVA/0.5 % steel fiber reinforcement show 51.7 % and 2.7 % creep reduction in contrast to plain specimens. Creep strains for compact tension specimens do not decrease significantly when 1 % PVA fibers are introduced. In compression, 1 % and 5 % PP fiber introduction reduces creep strains by 21.2 % and 49.7 % in contrast to plain geopolymer.
4. The specific creep of geopolymer composites in compression is on average 85.92 % less than in tension. It shows that in tension, geopolymer composites have 7.5 higher specific creep than in compression that indicates that in tension, geopolymer composites are more prone to creep. In three-point bending, the 1 % steel fiber amount shows 37.1 % less specific creep than the plain geopolymer composite. Geopolymer composites in three-point bending show 99.18 % less specific creep than in compression and 99.88 % less than in tension. Furthermore, in compression, geopolymer composites show 13 % to 23 % lower specific creep than ordinary Portland cement-based composites.
5. Fiber introduction shows benefits in shrinkage strain reduction. For compact tension geopolymer composites, the 1 % PVA fiber reduces shrinkage strains by 54.21 %.
6. Regarding long-term property results, the methodology of long-term property determination in various stress-strain conditions was developed (**Patent I**) and patented.
7. Microstructure analysis of specimens that were exposed to long-term compressive load showed that fiber incorporation into the geopolymer matrix significantly increases the amount of air void into the mix. Fiber addition of 1 % would increase air entrapment by 0.61 % to 2.26 % in steel fibers, 1.88 % in 2.50 % PVA fibers, and 1 % to 2.26 % in waste steel fiber reinforced specimens. It also shows that air entrapment throughout the

specimen cross section decreases from 4.7 % to 10.3 % in contrast to the outer layers of cross section.

8. Air inclusion for compact tension specimens is on average 5.15 % higher than for the compression specimens. Furthermore, for PVA fiber reinforced compact tension specimens, it is on average 19.85 % to 26.29 % higher than for the plain geopolymer composites.
9. There are clear indications that the long-term properties of geopolymer composites are linked with microstructure composition – shrinkage cracks play a huge role, and in this case, fiber reinforcement improves the mechanical and long-term properties and reduces shrinkage effects.
10. Regarding the loading effect on the specimen cross section composition and further interaction with creep strains, at least with the applied load amount of 20 % from ultimate load, in compression, no indications of specimen cross section disintegration or rupture were found. In tension and three-point bending, cracks were observed for the loaded specimens. It further leads to conclusions that specimen shape, especially for heat cured geopolymer composite, leads to inner stresses that due to shrinkage, create micro cracks, and while in compression, micro cracking would not have an immediate visible effect on the long-term property amount in tension and three-point bending, where some part or all cross section is subjected to tensile stress, the long-term properties are influenced immediately.
11. Regarding the results of quantitative image analysis of the polished sections of long-term tested geopolymer composites, the method for determining the outside factor impact on concrete and cement composite microstructure in various stress-strain states (**Patent II**) was developed and applied in the patent.

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