Bio-based sodium alginate treatment for natural fibre as partial aggregate replacement for Geopolymer Concrete.

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Abstract

MNEXT (formerly the Center of Expertise – Biobased Economy (CoE BBE)) is researching about the link between Geopolymer, Alginate and Natural fibres treated using Alginate, more specifically about the potential usage of Sodium Alginate and Alginate-treated natural fibres as biological/sustainable binder/activator and reinforcement for Geopolymer concrete tiles for civil engineering application in the “Geopolymer concrete tiles using Alginate and Natural Fibre” research project group. Untreated Miscanthus fibres tend to absorb water and have poor physical properties and interfacial bond between the fibre and the geopolymer matrix, thus fibre needs to be treated with chemicals before it can be used in producing geopolymer concrete tiles. This research attempt to design a geopolymer concrete mixture with partial aggregate substitution with Miscanthus fibre, and perform a preliminary investigation into the viability of Sodium Alginate solution as a pretreatment method for Miscanthus fibre for followup research by MNEXT and Joint Research Center Zeeland. Sodium Alginate solution by the results of this research is a viable pretreatment method for Miscanthus fibre. Geopolymer with Miscanthus fibre substitution pretreated by Sodium Alginate perform almost to peer with geopolymer concrete with Miscanthus fibre pretreated conventionally by NaOH by the metrics of Compression Strength and Modulus of Rupture, and outperform NaOH when the metrics of workability and sustainability into account.

Keywords: geopolymer concrete, alginates, natural fibre, miscanthus fibres, mechanical properties, compressive strength, modulus of rupture.

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1. Introduction

1.1. Background Information

With the global ongoing movement towards renewable energy and sustainable economy, the construction industry remains one of the fields that are still far behind. While Ordinary Portland Cement (OPC) concrete is one of the most economical and effective construction material worldwide, OPC concrete production still accounts for approximately 10% of global human CO2 emissions (PBL Netherlands Environmental Assessment Agency, 2014) due to the highly energy-intensive and CO2-emitting production of Portland cement clinker. In the effort to reduce the embodied CO2 emissions of concrete and increase sustainability through reusing industrial byproducts, there have been many research into alternative Portland cement blends that partially substitute the Portland cement clinker with alternatives such as ground granulated blast furnace slag (GGBFS), bottom ash or fly ash (FA), and researches to find an alternative concrete binder to Portland cement. One highly promising alternative that have been the centre of much interest in the recent years is Geopolymer Concrete (GPC). Geopolymer Concrete (GPC) is concrete which completely substitute the Portland cement binder with geopolymer, a type of alkali-activated binders formed from the polymerization of aluminosilicate precursor (typically used are fly ash, blast-furnace slag and metakaolin) with an alkali activator solution (typically used are Sodium Hydroxide (NaOH) and Sodium Silicate (Na2SiO3)) (Davidovits, States of the Geopolymer R&D 2017, 2017). Geopolymer Concrete uses no Portland cement and utilizes industrial byproducts such as fly ash and GGBFS as the aluminosilicate precursor for the reaction, and consequently has been found to has a significantly lower embodied carbon emission and environmental impact compared to Portland cement concrete of equivalent strength (Soo Huey Teh, 2017). Furthermore, different from Portland cement binder which undergoes the hydration process wherein water is a reactant and required in large quantity for both the reaction and workability, geopolymer undergoes the polymerization process which require only a small amount of water to dissolve the reactants and achieve sufficient workability for the fresh slurry, reducing the amount of water used in concrete construction.

While Geopolymer Concrete is a highly promising more sustainable alternative to Portland-cement concrete, but as a concrete, Geopolymer concrete still require a large volume of aggregates in its mixture. Since 2016, the annual global demand from the construction industry for aggregates is more than 40 billion metric tons and growing, and their extraction has led to both depletion of local resource and damaging to the environment (Sutton, 2020; Marco Hernandez, 2021). With the risk of depleting natural resource, soaring raw material prices and in order to make geopolymer concrete to be even more sustainable, one potential development is to substitute the aggregate with more sustainable, biobased aggregate alternatives. Adding fibres (such as steel fibre, glass fibres and different natural fibres) as reinforcement to the cementitious matrix is a well-established method to enhance the strength and other mechanical properties of concrete, and natural fibre has received much scientific attention as a potential biobased aggregate alternative or substitution for OPC concrete. Natural fibres are used as a renewable reinforcement and aggregate substitute for concrete in region where they are abundant, typically utilizing agricultural wastes and by-products such as shredded coconut shell (Radha Tomar, 2021), palm oil shell (Mahmud, 2012), rice husks (Morgan Chabannes, 2014), hemp (Samer Ghosn, 2020), and particularly of note is Miscanthus fibre (Ezechiels, 2017; Acikel, 2011; Fan Wu, 2023; Y. X. Chen, 2020). According to Santos et al (Santos, 2015), natural fibres have several advantages: low density, lower but still acceptable mechanical strength, is bio-based and renewable as well as being widely available in area with agriculture industry. However untreated natural fibres also have many disadvantages, such as poorer mechanical properties, are hydrophilic and more importantly have poor interfacial bond between the fibre and cementitious matrix, and thus the material need to be chemically pretreated before they can be used in producing geopolymer concrete. Natural fibres are typically treated using synthesized chemicals such as Sodium Hydroxide, Silane and Sodium Silicate, and researches into using bio-based chemicals such as Sodium Alginate to treat natural fibres such as coir, banana, sisal

(Mani, 1990) and flax fibre (Maya, 2022) as reinforcement for plastic composites were promising but inconclusive.

Among natural fibre, Miscanthus fibre is a highly promising candidate as aggregate replacement for geopolymer concrete in the Netherlands due to its rising availability in Europe, low cost and good mechanical properties of the fibre. Miscanthus x giganteus (also called ‘Elephant grass’) is a perennial biomass crop that is increasingly being cultivated in Europe as an energy crop (Anderson, et al., 2014) and ornamental crop (Schiphol Airport, 2021) due to their high adaptability to European climate, low water and fertilizer need, the ability to be grown in barren land (Davey CL, 2017) and high dry biomass yield (John Clifton-Brown, 2017). In addition to being an ideal energy crop, in recent years there have been a growing interest in utilizing miscanthus fibre as a biobased sustainable building material. In the Netherlands, Miscanthus fibre is also grown as natural, green bird control system in airport, and the agriculture waste from that purpose represents a potential source of cheap biomass for construction materials. Studies on the use of Miscanthus fibre for OPC concrete are scarce, even more so for geopolymer concrete, but existing studies (Ezechiels, 2017; Acikel, 2011; Fan Wu, 2023; Y. X. Chen, 2020) suggested that Miscanthus fibre is highly suitable as a reinforcement and aggregate substitute for OPC concrete.

Alginate is a relatively low-cost, green and naturally occurring polymer and gellant extracted from brown algae by an alkaline treatment, typically using Sodium Hydroxide, and commercially available as water-soluble sodium alginate powder (US Patent No. 2036922, 1936; M., 2008). Alginate is extensively used and researched as a stabilizer/thickener in food technology, and Alginate polymer is a highly promising natural replacement for synthetic polymers and show promises as an additive and bio-admixture that can potentially increase the mechanical strength of concrete (R. Ramasubramani, 2016). Sodium Alginate also show promises as a biobased pretreatment method for natural fibre for application in plastic composites by coating the exterior of the fibre with a protective layer. However, the efficacies of this pretreatment method for concrete and especially geopolymer concrete are untested, and the hydrophilic nature of Sodium Alginate may negatively affect the workability and strength of the concrete mixture the fibre would be used in.

Overall, existing research into using Sodium Alginate to treat natural fibre for use in geopolymer or concrete is scarce, and so is into using chopped Miscanthus fibre as a coarse aggregate substitute for geopolymer concrete. Furthermore, due to the difference in chemistry and matrix formation between OPC binder and geopolymer binder and geopolymer concrete’s higher alkalinity could affect the compatibility between Miscanthus fibre and the concrete matrix differently, and as the result affect the produced concrete’s strength differently. This research would thus develop a concrete using geopolymer as binder and containing Miscanthus x giganteus fibre pretreated using Sodium Alginate, comparing the properties of different geopolymer concrete mixtures using different pretreatment methods for the Miscanthus x giganteus fibre aggregate substitute versus plain geopolymer concrete.

This research paper is part of the “Geopolymer concrete tiles using Alginate and Natural Fibre” research project group, a collaboration between the research group MNEXT and Vibers B.V., the Netherlands. MNEXT, formerly the Centre of Expertise – Biobased Economy (CoE BBE), is a Centre of Expertise formed in partnership between HZ University of Applied Sciences and Avans University of Applied Sciences, with the purpose of developing more sustainable and biobased materials and construction methods in the global transition towards a green, sustainable, and circular economy. Vibers B.V. (not to be confused with Rakuten Viber) is a green paper, bioplastic and construction material supplier specialized in products utilizing Miscanthus fibre in the Netherlands, and is the supplier of the processed Miscanthus fibre used by this project. Following MNEXT’s purpose, this research paper’s goal is to perform a preliminary investigation into the viability of using Sodium Alginate as a pre-treatment method for Miscanthus x giganteus fibre for use as partial aggregate substitution for fly-ash-based geopolymer concrete for future research by MNEXT’s Geopolymer research group.

1.2. Problem Statement

Geopolymer concrete is a more sustainable alternative to Portland cement concrete. However, as a concrete, Geopolymer concrete still require a large volume of aggregates in its mixture. One way to make geopolymer concrete more sustainable and biobased is to use natural fibre as partial aggregate substitute for the mixture, specifically using Miscanthus fibre. All natural fibre needs pretreatment before it can be used in a concrete mixture, however the chemical processes of Portland cement concrete and fly-ash based geopolymer are fundamentally different and the same pretreatment method can have different effectiveness between normal Portland cement concrete and fly-ash based geopolymer concrete. Sodium Alginate is one potential biobased and sustainable natural fibre pretreatment method, having been tested on coir and sisal fibre for normal Portland cement concrete. As of right now the effectiveness of Sodium Alginate pretreatment for Miscanthus fibre for usage in geopolymer concrete is still unclear, and should be further investigated. This research seeks to develop a biobased geopolymer concrete mixture using chopped Miscanthus fibre pretreated using Sodium Alginate as aggregate substitution, comparing the effectiveness between this, GPC with Miscanthus fibre using conventional Sodium Hydroxide pretreatment method and plain GPC without natural fibre substitution.

1.3. Objective

As part of this broader research project, the main objective of this research paper is to develop a bio-based geopolymer concrete using short-strand Miscanthus x Giganteus fibre treated with Sodium Alginate as partial substitute for coarse aggregate, with the desired properties.

In order to complete this objective, the following goals must be achieved:

1) Determine the workability, compressive strength of Geopolymer concrete with Sodium Alginate-treated Miscanthus fibre aggregate replacing at least 10% of its total volume.
2) Determine the effectiveness of Sodium Alginate pretreatment method compared to conventional pretreatment method, and whether it is worth further investigation.
3) Develop an in-house methodology/procedure for the production of alginate-treated natural fibre for future research by MNEXT Geopolymer research group.

1.4. Research Question

Through discussions with the research group MNEXT, the following research questions were defined:

"Is it possible to use Miscanthus x Giganteus fibre pre-treated with Sodium Alginate as partial replacement for aggregate in Geopolymer concrete mixture?"

To answer this, the following sub-questions need to be answered:

- How can alginate be used to pre-treat Miscanthus fibre?
- What is the optimum Sodium Alginate solution to pre-treat Miscanthus fibre?
- How does Geopolymer concrete with Sodium Alginate-pretreated Miscanthus fibre as partial replacement for aggregate in the mixture perform against plain Geopolymer concrete without natural fibre in the mixture, and against Geopolymer concrete with conventional NaOH-pretreated Miscanthus fibre as partial aggregate replacement?

1.5. Boundary Conditions

Based on client expectations and comparable typical properties of geopolymer concrete mixture (see below section 2.2. Geopolymer concrete), we have the following requirements:

- ❖ Technical requirements:
  - Miscanthus fibre pre-treated using Sodium Alginate consist of at least 10% of total mixture volume.
  - Compressive strength reaching 30 MPa.
  - Density lower than conventional concrete (2400 kg/m^3).

- Functional requirements:
  - Good workability: Slump between 50-90 mm, uniform throughout the mix, no collapse or shear happen in slump testing.

2. Theoretical Framework – Literature review

2.1. Geopolymer

Geopolymer is a type of ceramic-like inorganic polymer created from rock-forming minerals of geological origins, hence the term “geopolymer” (Davidovits, States of the Geopolymer R&D 2017, 2017), with many applications in naval, aerospace, automobile and civil engineering as high-performance material that can be used independently, as a fire-resistant coatings and protective layer, or as binder for concrete. In recent years, Geopolymer have received much interest as an alternative for Portland cement as a binding agent for concrete (A. Palomo, 1999). Geopolymer as a modern concept was first defined in 1978 by Joseph Davidovits, a French researcher at the Cordi-Géopolymère private research lab, Saint-Quentin, France (Davidovits, Geopolymer Chemistry and Applications, 5th Edition, 2020), however earlier scientific researches into alkali-activated binders existed as early as the 1940s, and methods of producing alkali-activated slag cement/binder were independently invented in Belgium (Trief cement - (Trief Victor, 1950-1954)) and Ukraine (Gruntosilikaty ‘soil silicates’ - (Glukhovsky, 1959)). Geopolymer is defined by Joseph Davidovits as “an amorphous (non-crystalline, at ambient and medium temperature) to quasi- or nano-crystalline (at temperatures > 500°C), highly-connected three-dimensional matrix formed from chains or networks of inorganic mineral molecules linked with co-valent bonds”, with different synthesis routes and precursors, such as via an acidic medium using Phosphoric acid precursor, or via an alkaline medium using (Na/K/Ca)-hydroxides and alkali-silicate precursor, calcium (Ca, K, Na)-sialate (steel slag) precursor, silica precursor, kaolinite/hydrosodalite precursor, or metakaolin precursor, ferro-sialate precursor, or fly-ash precursor (Davidovits, Geopolymer Chemistry and Applications, 5th Edition, 2020).

The chemical hardening process of geopolymer is fundamentally different from that of Portland cement. When Portland cement is mixed with water and aggregates to form a mortar or concrete slurry, the compounds making up Portland cement undergoes a hydration reaction with the water in the mixture causing the mixture to gain strength; the chemical reaction stops when the mixture runs out of cement or water. Thus, the water in a Portland cement mixture contributes to the strength and the workability of the mixture – insufficient water can lead to the mixture drying out before the mixture have fully reacted and gained its potential strength, and excess water can reduce the strength of the resultant concrete, increasing shrinkage and the amount of cracking (University of Illinois Urbana-Champaign). In geopolymer or geopolymer concrete mixture, the aluminosilicate precursor and alkali activator undergo a poly-condensation (polymerization) reaction without reacting with water, the water in the mixture only serve as the solution medium where the reaction occur and to provide workability to the mixture (Davidovits, Geopolymer Chemistry and Applications, 5th Edition, 2020). Thus, in geopolymer concrete mixture, insufficient water may lead to difficulty with mixing and casting the mixture but does not have a chemical effect on geopolymer strength, and excess water can similarly cause reductions in strength, especially the potential to cause leaching when the water evaporates from the geopolymer mixture.

2.2. Geopolymer concrete

Concrete is a type of composite material in which a filler (typically fine and coarse aggregate) is held together by a cementitious matrix acting as binder. The most commonly used kind of concrete is ordinary Portland-cement (OPC) concrete, which use a slurry from Portland-cement and water as the binder. Geopolymer concrete (GPC) is a concrete which utilizes Geopolymer as a binder in place of Ordinary Portland cement, and is defined as “a concrete which comprises of a geopolymer binder, aggregates, water and admixtures”. For usage as concrete binder, Geopolymer binder can be defined as “a type/sub-group of alkali-activated binders formed from an Aluminosilicate precursor (typically used are fly ash, blast-furnace slag and metakaolin) with an alkali activator solution (typically used are Sodium Hydroxide and Sodium Silicate)". For this usage, the most commonly utilized types of geopolymer to look into is heat-cured or ambient-cured fly ash-based geopolymer or calcium (steel slag)-based geopolymer, due to these precursor's prevalence as industrial byproducts abundantly available in developed and developing countries, having received much attention in the recent years (Patankar S. V. G. Y., 2015; Djwantoro Hardjito, 2005; M. Sofi, 2007; Soo Huey Teh, 2017). For construction application, GPC have many advantages over OPC concrete: geopolymer concrete use much less water, geopolymer production process can make use of industrial byproducts such as fly ash and blast-furnace slag as the aluminosilicate precursor material meaning geopolymer potentially have a much lower carbon emission and environmental impact than Portland cement, while still able to produce a concrete with an equivalent to higher compression strength to Portland-cement concrete, and furthermore depending on the mixture and precursor base, geopolymer potentially has a shorter curing time until the material reach full strength (A. Palomo, 1999). Without taking the production of fly ash and granulated blast furnace slag and the temperature curing process into account, GPC has up to 70% less embodied emission than OPC concrete (Davidovits, Technical Paper #24, 2015). When taking the production of fly ash, granulated blast furnace slag and the temperature curing process into account, fly-ash GPC has been found to have significantly (approx. 20%) less embodied emissions than OPC concrete (Soo Huey Teh, 2017)

There are multiple factors that affect the strength and workability of a fly-ash based geopolymer concrete mixture (Patankar S. V. G. Y., 2015), including water content to achieve desired workability (Jamkar S. S., 2013), fly ash quantity, content and fineness (Patankar S. V. J. S., 2012), ratio of solution to binder (Patankar S. V. J. S., Effect of solution-to-fly ash ratio on flow and compressive strength of geopolymer concrete, 2012), ratio of solution to fly ash (Patankar S. V. J. S., Effect of grading of fine aggregate on Flow and compressive strength of geopolymer concrete, 2014), ratio of sodium silicate to sodium hydroxide (Patankar S. V. J. S., Effect of water-to-geopolymer binder ratio on the production of fly ash based geopolymer concrete., 2013), grading and quantity of fine aggregates (Patankar S. V. J. S., Selection of suitable quantity of water, degree and duration of heat curing for geopolymer concrete production, 2014; Patankar S. V. J. S., Effect of grading of fine aggregate on Flow and compressive strength of geopolymer concrete, 2014), and the curing process. For this research paper, particularly of importance is the factor of water/fly ash ratio due to natural fibre filler's tendency to absorb water from the concrete mixture.

B. Singh reported on the typical properties of geopolymer concrete mixes in 2015 (B. Singh, 2015), comparing a variety of fly-ash-based geopolymer concrete mixtures based on their compressive, tensile, and flexural strength, different curing condition/time, and other mechanical properties. Heat-cured fly ash-based GPC can achieve compressive strength from 30 up to 80 MPa and flexural strength from 2,25 to 12 MPa in only 24 hours (Djwantoro Hardjito, 2005; A. Fernandez-Jimenez, 2005; E.I. Diaz-Loya, 2011; Z. Pan, 2011), but the high-temperature curing condition is difficult to replicate on-site. With the addition of steel slag, GPC can achieve 47-56,5 MPa in compressive strength curing at ambient temperature 23°C for 28 days (M. Sofi, 2007). Similar to OPC concrete, GPC has poor tensile and flexural strength, at approx. 10-20% of the compressive strength.

Table 1 Typical properties of geopolymer concrete mixes (B. Singh, 2015)

	Density (kg/m3)	**Molarity (M)**	**Slump (mm)**	**Compressive Strength (MPa)**	**Splitting Tensile Strength (MPa)**	**Flexural Strength (MPa)**	**Modulus of Elasticity (GPa)**	**Poisson's ratio**	**Activator/ binder ratio**	**Curing temperature and time**
(Djwantoro Hardjito, 2005)	2330–2430	10–16	60–215	30–80	3.74–6	5–12	23–31	0.12–0.16	0.35–0.4	60–80°C for 24 h
(A. Fernandez-Jimenez, 2005)	Not Reported	8 & 12.5	Not Reported	29–43.5	Not Reported	6.86	10.7–18.4	Not Reported	0.4 & 0.55	85°C for 20 h

(M. Sofi, 2007)	2147–2408	NR	Not Reported	47–56.5	2.8–4.1	4.9–6.2	23–39	0.23–0.26	0.45–0.59	35°C for 24 h then 23 C for 28 days
(E.I. Diaz-Loya, 2011)	1890–2371	14	100–150	10–80	Not Reported	2.24–6.41	1.9–42	0.08–0.22	0.4–0.94	60°C for 72 h
(Z. Pan, 2011)	1876–2555	8	Not Reported	65.1–77.9	2.8–5.1	Not Reported	11.2–41.2	0.15–0.19	0.4–0.65	60°C for 24 h

For the purposes of this research paper, the chosen type of geopolymer to be used as the base for the geopolymer concrete with partial aggregate substitution with natural fibre would be heat-cured fly-ash based geopolymer. Fly ash is chosen as MNEXT and the Scalda Concrete Laboratory has an existing local supply of fly ash. While heat-curing is inappropriate for on-site conditions, the MNEXT research group seeks to develop a geopolymer concrete tile, and temperature curing is possible for this purpose.

As Geopolymer concrete is a relatively new field, there lacks a European standard geopolymer concrete mix design procedure. As B. Singh reported (B. Singh, 2015), there are multiple different mixture designs that could achieve the required compressive and flexural strength by MNEXT. A literature review is performed in order to find an appropriate fly-ash-based GPC mix design to replicate, and the MNEXT research group have chosen the M30 geopolymer concrete mix design by S.V. Patankar et al (Patankar S. V. G. Y., 2015) as the reference baseline geopolymer concrete mixture for this research.

Table 2 Geopolymer concrete mixture (Patankar S. V. G. Y., 2015)

Geopolymer concrete M30	
Materials	kg/m^3
Fly ash (type F)	405,0
Sodium hydroxide solution ($NaOH$)	70,88
Sodium silicate solution (Na_2SiO_3)	70,88
Water content	108,35
Fine aggregate	683,13
Coarse aggregate	1268,66

The mixture had a water/fly ash ratio of 0,267. S. V. Pattankar proposed this as the optimal water/fly ash ratio for a high compressive strength and still maintaining a sufficient workability for the GPC.

The study by S.V. Patankar reported that after 1 day of curing at 60°C, the M30 GPC mixture had the following properties:

Table 3 Properties of M30 mixture (Patankar S. V. G. Y., 2015)

Properties	**Value**
Compression strength	37.22 MPa
Dry density	2601.48 kg/m^3
Workability	Medium

2.3. Natural fibre as partial replacement for aggregate

Fly-ash-based geopolymer concrete overall represent a promising more circular and sustainable alternative to Ordinary Portland-cement concrete, however any geopolymer concrete mixture by their nature as a concrete still require a large volume of aggregates. Natural aggregates commonly utilized in construction are crushed rocks, gravel and sand. Since 2016, the annual global demand from the construction industry for aggregates is more than 40 billion metric tons and growing, and their extraction has led to both depletion of local resource and damaging to the environment (Sutton, 2020; Marco Hernandez, 2021) especially the Middle East and South East Asia. Even if the global construction industry switches over to Geopolymer Concrete, this demand for coarse aggregates would remain, and thus there is a need for more renewable aggregate alternatives, such as recycled concrete aggregate (Syed Minhaj Saleem Kazmi, 2022), recycled tire rubber aggregate (Sachinthani Karunarathna, 2021) and natural fibre aggregate. Adding natural fibre reinforcement to the cementitious matrix is a well-established method to enhance the strength and other mechanical properties of concrete, and natural fibre has received much scientific attention as a potential biobased aggregate alternative or substitution for OPC concrete. Substantial research has been done into bio-based aggregate alternatives for Portland-cement concrete shredded coconut shell (Radha Tomar, 2021), palm oil shell (Mahmud, 2012), rice husks (Morgan Chabannes, 2014), hemp (Samer Ghosn, 2020) and other fibre as aggregate alternatives for OPC concrete. Particularly of note is Miscanthus fibre (Ezechiels, 2017; Acikel, 2011; Fan Wu, 2023; Y. X. Chen, 2020). Ezechiels (Ezechiels, 2017) experimented with substituting OPC concrete aggregate with different percentages by volume with short-strand Miscanthus fibre with additional water to saturate the natural fibre, and found that at 10% by volume substitution of aggregate with Miscanthus fibre resulted in OPC concrete that reached approximately 30 MPa.

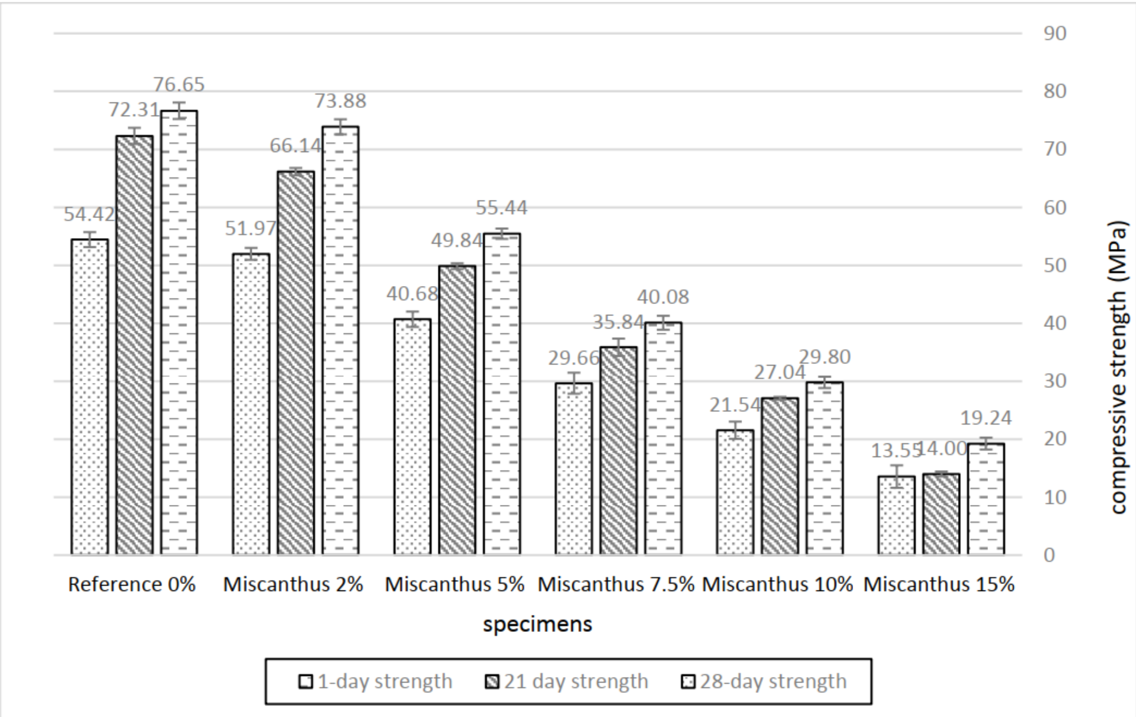

Figure 1 Compressive strength results of Miscanthus-Based Bio-Concrete: Mix series 3 (replace all aggregates by volume) (Soo Huey Teh, 2017)

For both using natural fibres as reinforcement or as renewable aggregate (full or partial) substitute, the strength of the resulting concrete or composite is dependent on the fibre content used, the mixture used as well as curing conditions of the concrete mixture, and the fibre properties such as the fibre strength, moisture absorption, and most importantly the interfacial bond between the fibre surface and the geopolymer matrix. According to Santos et al (Santos, 2015), natural fibres have several advantages over other alternative concrete aggregates: low density, high tensile strength and stiffness, better crack resistance, is bio-based and renewable as well as being widely available in area with agriculture industry. By utilizing agricultural wastes and by-products such as coconut shell, palm oil shell, hemp, flax and Miscanthus stems and pulp as natural fibre aggregate, the sustainability of concrete can be further increased. Biobased aggregates such as natural fibre also have lower density than mineral-based coarse aggregate like gravel and crushed concrete and can be used to create Light-weight Aggregate concrete (LWAC). By substituting partially or completely the coarse aggregate in a concrete mixture, the density of the mixture can be decreased. This can counteract the decrease in compressive strength by using biobased aggregates by increasing the relative Specific compressive strength.

However, untreated natural fibres also have many disadvantages: poorer mechanical properties, are hydrophilic, poor longevity in alkaline environment and more importantly have poor interfacial bond between the fibre and cementitious matrix. Hydrophilic natural fibre soaks in and lock up free water from a concrete mixture, thus affecting the water/binder (cement or fly ash) ratio and the workable water, negatively affecting both the strength and workability of a concrete mixture. In order to overcome this, additional water is used to saturate the natural fibre before adding to the concrete mixture, but this changes the water/binder ratio and have a negative effect on the concrete mix, potentially causing excessive shrinkage, bleeding and reduction in strength (Yu, 2014; Chen, 2008). The interfacial bond between the natural fibre and the cementitious matrix is the critical factor determining the strength of a concrete mixture with fibre additive. While alkaline pretreatment is commonly used to reduce the water absorption of natural fibre and increase the interfacial bond, prolonged exposure to an alkaline environment can damage the strength and durability of natural fibre, an important factor to consider due to OPC concrete’s alkaline nature and that GPC is made using alkaline activator and have a higher alkalinity than OPC concrete, potentially worsening the deleterious effect on the natural fibre aggregate. Natural fibres need to be pretreated before they can be used in producing geopolymer concrete. Natural fibres are typically treated using synthesized chemicals such as Sodium Hydroxide, Silane and Sodium Silicate, and researches into using bio-based chemicals such as Sodium Alginate to treat natural fibres such as coir, banana, sisal (Mani, 1990) and flax fibre (Maya, 2022) as reinforcement for plastic composites were promising but inconclusive.

Natural fibre’s hydrophilic nature can be countered by pretreatment and a suitable addition of water. While too much additional water can lead to bleeding, shrinkage and reduction in strength, a balance can be reached between workability and concrete strength loss (Yu, 2014; Chen, 2008). The addition of water can be done by adding water to saturate the fibre before mixing (Stephens, 1994; Mohr, 2005) or by adding water into the concrete mixture to counteract the amount that would be absorbed by the fibre (Marcos, 2015). For Miscanthus fibre and OPC concrete, Ezechiels (Ezechiels, 2017) determines that the appropriate amount is 2,5g of water per 1g of dry Miscanthus fibre, to reach a water/cement ratio of 0,5. As GPC require water only for maintaining workability, the water/fly-ash ratio to balance workability and strength loss for natural fibre is likely to be lower than 0,5.

While natural fibre as OPC concrete aggregate substitute has received much attention over the years, research into natural fibre as geopolymer concrete aggregate substitute remains scarce, and even more scarce is research into using Miscanthus fibre as aggregate substitute for both OPC and geopolymer concrete. Nonetheless, MNEXT recognizes using Miscanthus fibres as sustainable and biobased substitute filler in OPC and geopolymer concrete mixture is a highly promising avenue of research. Prior research by MNEXT (Adebiyi, 2022; Marianna Coelho, 2013) have proven that short-strand Miscanthus fibre is highly compatible as an aggregate substitute for OPC concrete, however the strength and durability of the fibre and concrete mixture is affected by the alkaline content of the mixture, with high alkalinity mixture displaying noticeably less compression and flexural strength and durability. Geopolymer concrete, being made from an alkaline activator and aluminosilicate precursor, typically has a higher alkalinity than OPC concrete. This in addition with the fundamentally different chemical hardening process between the 2 types of binders mean that a separate experiment is necessary to determine the effectiveness of Miscanthus fibre as an aggregate substitute for geopolymer concrete.

In the hope of increasing the sustainability of geopolymer concrete, MNEXT would like to design a GPC concrete mixture with 10% by total volume replaced with Miscanthus fibre and investigate into using Sodium Alginate as a pretreatment method for short-strand Miscanthus fibre aggregate.

2.4. Miscanthus x Giganteus fibre

Miscanthus is a genus of perennial rhizomatous grass (commonly called 'elephant grass') that grow across Africa, Eurasia, and the Pacific islands of great interest as a high-yield biomass crop (John Clifton-Brown, 2017). At the moment of all the species within the genus, the naturally occurring, sterile hybrid Miscanthus x giganteus is the most commonly studied and cultivated genotype in Europe due to its high dry matter yield, low water and fertilizer input needs, and the ability to be grown in barren marginal lands, the inability to become an invasive species due to its natural sterility as well as its adaptability to a wide range of European climates (Davey CL, 2017). In addition to being an ideal energy crop (Anderson, et al., 2014), Miscanthus x giganteus is increasingly being cultivated in Europe as an ornamental crop. Additionally, densely grown plots of Miscanthus grass have the potential to be used as a natural, green, sustainable and non-toxic bird control system (Game & Wildlife Conservation Trust, UK), and has been grown with this purpose in the Schiphol airport in the Netherlands (Schiphol Airport, 2021). Elephant grass (Miscanthus) and flax would be grown next to the runways in order to create low-cost, natural barriers to Western European bird species, and when the grass would exceed the allowed growth height they would be cut down and allowed to regrow until future harvest. The agriculture waste from this purpose represents a potential source of cheap biomass for construction materials.

Among natural fibre, Miscanthus fibre is a highly promising candidate as aggregate replacement for geopolymer concrete in the Netherlands due to its rising availability in Europe, low cost, and good mechanical properties of the fibre (K. Kaack, 2001). In recent years there have been a growing interest in utilizing miscanthus fibre as a biobased sustainable building materials, and there have been several researches into the material particularly as additive for biobased cement mortar (Fan Wu, 2023), as a potential bio-based reinforcement and aggregate substitute for OPC concrete (Ezechiels, 2017; Acikel, 2011; Y. X. Chen, 2020); however, there is very few studies on using natural fibre as aggregate substitute for geopolymer concrete, studies on the use of Miscanthus fibre for OPC concrete are still very scarce, and there is almost none for geopolymer concrete. Acikel reported that the use of ground Miscanthus fibre as an additive increases the strength of OPC concrete (Acikel, 2011), and Ezechiels reported that pretreated short-strand Miscanthus fibre had good compatibility with OPC concrete matrix (Ezechiels, 2017). MNEXT has made contributions to using Miscanthus fibre as aggregate replacement for Portland-cement concrete (Adebiyi, 2022; Marianna Coelho, 2013), and found that OPC with significant Miscanthus fibre aggregate replacement can achieve compressive strength of 30 MPa. In Ezechiels and Adebiyi's studies (Ezechiels, 2017; Adebiyi, 2022), untreated Miscanthus fibre still have a high water adsorption and poor interfacial bond with concrete, and similar to all natural fibre the pretreatment method for the fibre has a major influence on the strength and workability of the resultant concrete mixture.

2.5. Pretreatment of Natural Fibre

There are multiple factors affecting the mechanical properties and strength of a concrete mixture with lignocellulosic fibre (natural fibre) as reinforcement or aggregate replacement. The most important factors are the surface area, fibre density and lignin/cellulose content which directly affects the Ultimate Tensile Stress (UTS) of the fibre, the interfacial bond (surface bonding strength/debonding stress) between the fibre and the cementitious matrix which affects how much of the stress/load within the matrix can be transferred to the fibre, and moisture absorption how much water the fibre will absorb from the concrete slurry which affects the mixture's workability.

Pretreatment is the process of reducing the lignin/cellulose content of the natural fibre before the fibre can be added to the concrete mixture, and depending on the pretreatment method chosen can affect the surface cross section area, structure, the lignin/cellulose content, surface bonding strength and moisture absorption of the fibre in different ways. Natural fibres are typically pretreated using synthesized chemicals such as Sodium Hydroxide, Silane and Sodium Silicate. The pretreatment methods most commonly used in industry is Alkali pretreatment (also known as Mercerization treatment) using Sodium Hydroxide (NaOH). Sodium Hydroxide treatment remove a certain amount of lignin, wax and oils that would interfere with the fibre-cement matrix interface, disrupt the hydrogen bonds and depolymerize the cellulose fibre, breaking down fibre bundles into smaller fibre crystallites that increases the surface roughness; these modifications combined lead to an increase in debonding stress and fibre strength of the treated fibre. Not enough Sodium Hydroxide (NaOH) remove insufficient lignin and cellulose, but too much Sodium Hydroxide (NaOH) can destroy the fibre, and Geopolymer concrete has a very high concentration of NaOH as NaOH is the activator solution. It has been reported that soaking Miscanthus fibre in a 5 wt% NaOH solution over 30 minutes at room temperature effectively remove most of the lignin, pectin, wax and oils and increases the surface roughness of the treated Miscanthus fibre (Estefania Boix, 2016).

These synthesized chemicals however result in chemical waste, are not eco-friendly and can be toxic to human life and the environment, thus in recent years other more bio-based treatment methods are beginning to be looked into, however this remains a sparsely researched field (Mani, 1990). In this field, Sodium Alginate is one avenue worth exploring. Experiment by Mani et al (Mani, 1990) reported that by using 0,25% Sodium Alginate + CaCl2 solution to treat Coir, Banana and Sisal fibre resulted in a minor (~16%) increase in Ultimate Tensile Strength (UTS) and in debonding stress for Coir fibre, while resulting in a negative decrease in UTS and debonding stress for Banana and Sisal fibre. Mani reported that the Sodium Alginate + CaCl2 solution did not alter the chemical properties of the treated fibre (unlike with alkali treatment and silane treatment) but achieve this via applying a hydrophilic protective coating to the fibre surface that increase the surface roughness (while also increasing the moisture absorption of the fibre) and consequently debonding stress of the fibre. Another experiment by Maya Jacob John (Maya, 2022) reported that by using 2% and 4% Sodium Alginate solution to treat flax fibre as reinforcement for PLA and PHBV composites resulted in an increase in stiffness in treated composite over untreated composite, but did not report on any changes in Ultimate Tensile or Compressive Strength. There is still a knowledge gap regarding using Sodium Alginate as a treatment method for natural fibre as a whole and Miscanthus fibre in specific for usage as fibre reinforcement/aggregate replacement for Geopolymer concrete for which there is no research prior, and this is an avenue worth pursue into, especially taking into consideration the broader research goal of the MNEXT Geopolymer research group is producing bio-based Geopolymer concrete tiles using Sodium Alginate as binder replacement and using Miscanthus fibre treated with Sodium Alginate as aggregate replacement, by which can streamline the production process considerably. In the effort to make geopolymer concrete greener this paper would like to investigate if Sodium Alginate is a possible 'greener' treatment method for natural fibre, and how would this affect the strength of the resultant geopolymer concrete mixture.

Conventionally, any treated fibre will have to be rinsed clean and dried of excess humidity in order to prevent chemical contamination to any polymer or concrete mixture the fibre would be used in, or for the pretreatment chemical to continue to excessively damage the natural fibre. However, Sodium Alginate does not have any reaction with Sodium Hydroxide (NaOH) and does not have a chemical effect on Miscanthus fibre. Thus, theoretically fibre pretreated with Sodium Alginate can be used without requiring a thorough rinsing or drying, and the excess moisture absorbed by the Miscanthus fibre during the treatment process can be used as the additional water used to saturate the natural fibre.

Furthermore, there is very few research in using natural fibre as aggregate replacement for geopolymer concrete, and there is very few research into bio-based or Sodium Alginate pretreatment method for natural fibre overall and none for Miscanthus fibre in specific as of the writing of this report. This research would like to contribute to filling in this knowledge gap by investigating using Sodium Alginate to pretreat Miscanthus fibre used as aggregate substitute for geopolymer concrete, since Sodium Alginate is the material being investigated as admixture/binder replacement for Geopolymer Concrete tiles by the MNEXT Geopolymer research group.

2.6. Sodium Alginate

Alginic acid is a relatively low-cost, green and naturally-occurring polysaccharide with a linear copolymer layout consisting of a single main chain composed of consecutive and alternating blocks of β-D-mannuronic acid (M) and α-L-guluronic acid (G). Alginic acid salts with sodium and calcium are commonly called alginates. Alginate is extracted from brown algae by an alkaline treatment, typically using Sodium Hydroxide, and subsequently precipitated into salt, transformed into alginic acid and finally into water-soluble sodium alginate powder (US Patent No. 2036922, 1936; M., 2008). Thanks to its low toxicity and high biocompatibility, Alginate is extensively used and researched as a stabilizer/thickener in food technology, and Alginate polymer is a highly promising natural replacement for synthetic polymers, and show promises as an additive and bio-admixture that can potentially increase the mechanical strength of concrete (R. Ramasubramani, 2016). Like Geopolymer, Alginate also form polymers and is extracted using Sodium Hydroxide, and it is thus a novel and promising field of research into whether Alginate and Geopolymer can form a hybrid matrix with sufficient mechanical properties for applications in the civil engineering field or not, and whether Alginate can be used as an additive/admixture for Geopolymer Concrete. There has been researches into hybrid of porous geopolymer and sodium alginate to produce adsorbents that effectively remove Cu(II) (Yuanyuan Ge, 2017) and methylene blue (S. M. A. S. M. Nurddin, 2020), but as of this research project beginning there is no known researches into geopolymer/alginate hybrid for civil engineering application.
As stated above, there exists a few (Mani, 1990; Maya, 2022) researches into Sodium Alginate as a bio-based chemical treatment method for natural fibres, in which results were promising but inconclusive, having been only tested in Coir, Sisal and Banana fibre as standalone material and in Flax fibre as reinforcement for PLA and PHBV composite, and for Flax fibre there is no known result on treatment effects on UTS and surface bonding strength, the 2 decisive factors affecting the produced concrete's strength. There is also no known experiments on using Sodium Alginate to treat fibre for usage as either reinforcement or aggregate replacement for Portland-cement concrete or geopolymer concrete. Furthermore, Sodium Alginate acts as a natural coagulant, and this can affect the workability of any given geopolymer concrete mixture negatively, especially since geopolymer by their characteristic have a very low water content compared to Portland-cement concrete. This in combine with the fact that natural fibre is also a water absorbent means that any mixture with Sodium Alginate will require a higher water content in order to maintain workability. On the other hand, Sodium Alginate does not react with the Sodium Hydroxide (NaOH) activator solution in geopolymer concrete and does not have a deleterious effect on the Miscanthus fibre, and as a Sodium Alginate solution with water is being used to pre-treat natural fibres in this experiment, hypothetically the pretreatment solution absorbed by the fibre can be utilized as the extra water to saturate the fibre by adding directly to the mixture without rinsing and drying. While this would still affect the produced mixture's strength negatively due to causing more water to eventually evaporate from the mixture compared to a mixture with less water, the save in energy and water this could have makes this solution a worthwhile investigation.

3. Methodology

3.1. Research approach

To fulfil the main objective and goals of this research, this research paper follows the following research model:

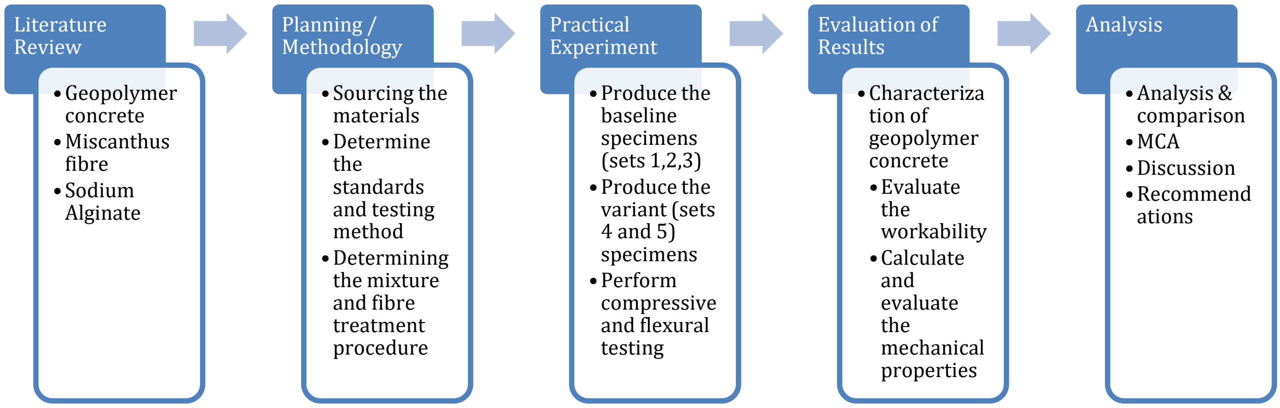

Figure 2 Research model

The research plan consists of 5 phases, roughly coinciding with the sections of this report:

Phase 1: Desk research, where a literature review of developments and characteristics regarding Geopolymer concrete, Miscanthus x Giganteus fibre, and Sodium Alginate is performed in order to establish the Theoretical Framework of this research. Identify a method for treating Miscanthus fibre with Sodium Alginate.

Phase 2: Sourcing the materials, and identifying the properties of the materials such as amount, density, particle size distribution, etc. Design the mixture with Miscanthus fibre as coarse aggregate partial substitute. Once the most applicable Eurocode standards for experimentation is identified and adapted for the conditions of the Joint Research Center Zeeland and Scalda Concrete Lab Vlissingen, begins the practical experiment phase.

Phase 3: Practical experiment phase, where the specimens are produced and tested as planned in phase 2, getting slump, self-weight, and maximum force at which the specimen cube fail.

Phase 4: The raw data from the testing is analysed and tabulated to get the mechanical properties of the concrete cubes.

Phase 5: Analyse and compare the different variants and baseline of geopolymer concrete using a Multi-Criteria Analysis model following the Analytical Hierarchy Process. Discuss and recommend the path forward for future follow-up research.

3.2. Experiment methodology

3.2.1. Slump test

The slump test is a method of measuring the consistency of freshly mixed concrete. The test can be used as an indicator for the uniformity and workability of the fresh concrete mixture. The test shall be carried out in accordance with the standards NEN EN 12350-2 and EN 206:2013+A2:2021: The testing cone is filled in 3 layers, each approximately one third of the height of the cone when compacted, each layer compacted with 25 strokes of the compacting rod. The cone is then raised vertically, and the slump *h* is determined as the difference between the height of the cone and the highest point of the slumped cone.

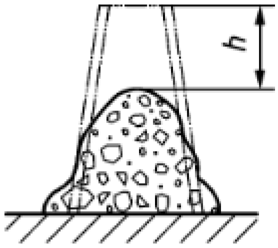

Figure 3 Slump measurement

Figure 4 Forms of slump

Table 4 Slump classes according to EN 12350-2

Class	Slump mm
S1	10 to 40
S2	50 to 90
S3	100 to 150
S4	160 to 210
S5	≥ 220

3.2.2. Compression test

In order to determine the effectiveness of the fibre aggregate substitution, an important factor to consider is the compressive strength of the geopolymer concrete mixture. As there is no existing European norm or standard for testing Geopolymer Concrete as of the writing of this report, this research will carry out testing in accordance with the standards NEN-EN 12390-3:2021 for testing hardened concrete. The compressive test is performed by a *RatioTEC RT 3000 2-D servo* machine at a Loading rate of 0.5 MPa/s (N/mm^2*s), and the maximum load at which the specimen fail will be recorded in kN. The produced cubes' dimensions and weight shall be measured before the test, and the specimen's failure mode will be recorded.

Figure 5 Cube in RatioTEC compression testing machine

According to NEN 12390-3:2021, the compressive strength is given by the formula:

$$f_c = \frac{F}{A_c}$$

Where:

f_c is the compressive strength, in MPa (N/mm^2)

F is the maximum load at failure as read from the test result, in N

A_c is the cross-sectional area of the specimen on which the compressive force acts, calculated from the designated size of the specimen (see EN 12390-1). $A_c = 22500\ mm^2$

The compressive strength shall be expressed to the nearest 0,1 MPa (N/mm^2).

From the Compressive Strength and Density (Mass/Volume), we can calculate the Specific compressive strength ($Pa * m^3/kg$) of each concrete cube:

$$Specific\ strength = \frac{f_c * V}{m}$$

Where:

$Specific\ strength$ is the specific compressive strength of the specimen, in $Pa * \frac{m^3}{kg} = kN * \frac{m}{kg}$

f_c is the compressive strength, in kN/m^2 (= 1/1000 * N/mm^2).

V is the Volume of the specimen, in m^3.

m is the Mass of the specimen, in kg.

This is an important value to determine for civil engineering applications since a building constructed out of a lighter material with the same strength can withstand a higher load. Furthermore, low self-weight benefits non-load bearing components of a structure (example: curtain walls, décor façade…).

3.2.3. Modulus of rupture and breaking strength of ceramic tiles

As there is no existing European norm or standard for testing Geopolymer Concrete tiles as of the writing of this report, this research will use the closest analogue being the NEN-ISO 10545:2019 standards for testing ceramic tiles for testing flexural strength due to geopolymer being a ceramic-like substance, using a three-point bending testing scheme as seen in figure 6.

The methodology of this experiment followed standard NEN-ISO 10545-4:2019 for testing ceramic tiles, however due to practical conditions not all requirements could be code-compliant. This determines the breaking load, breaking strength, and modulus of rupture of a tile by applying a force at a specified rate to the centre of the tile, the point of application being in contact with the proper surface of the tile.

The test was performed using a Tinius Olsen 50ST hydraulic press. The support and center loading members are arranged according to the 3-point layout, with the following values:

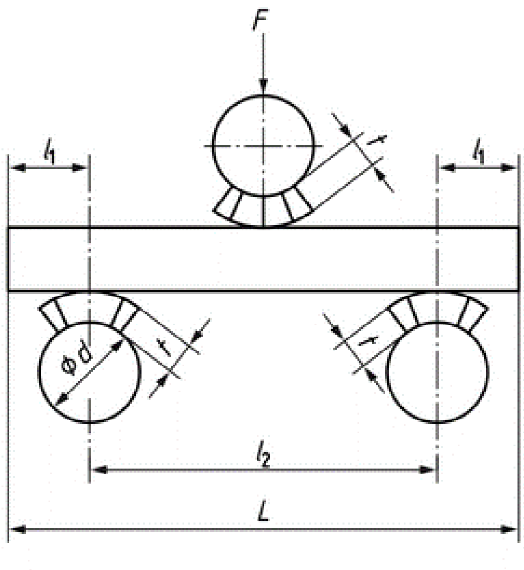

Figure 6 Layout Three-point Flexure test, NEN-ISO 10545-4:2019

Table 5 Diameter of rods, d, thickness of rubber, t, and overlap of tile beyond the edge support l_1

Long side of tile L	Diameter of rod d	Thickness of rubber t	Overlap of tile beyond the edge supports l_1
$18 \leq L < 48$	5 ± 1	1 ± 0,2	2 ± 1
$48 \leq L < 95$	10 ± 1	2,5 ± 0,5	5 ± 3
$L \geq 95$	20 ± 1	5 ± 1	10 ± 5

Where:

$d = 10\ mm$ is the diameter of rod.
$L = 120\ mm$ is the long side of tile.

$l_1 = 10\ mm$ is the overlap of tile beyond the edge supports.
$l_2 = 100\ mm$ is the span between the support rods.
$t = 2.5\ mm$ is the thickness of rubber.

The test was performed at a Loading rate of (1 ± 0,2) N/mm$_2$/s.

The test gives the following results:

- The Breaking Strength S, expressed in newtons, is calculated using formula:

$$S = \frac{F * l_2}{B} = \frac{F * 100}{B}$$

Where:
F is the breaking load, being the force necessary to cause the test specimen to break, expressed in N.
l_2 is the span between the support rods, in mm.
B is the short side of the test specimen under testing, in mm.

- The Modulus of Rupture R, expressed in newtons per square millimetre, is calculated using the formula:

$$R = \frac{3 * F * l_2}{2 * B * h^2} = \frac{3 * S}{2 * h^2}$$

Where:
F is the breaking load, being the force necessary to cause the test specimen to break, expressed in N.
l_2 is the span between the support rods, in mm.
B is the short side of the test specimen under testing, in mm.
h is the minimum thickness of the test specimen measured after the test along the broken edge, in mm.

The calculation of the modulus of rupture is based on a rectangular cross-section. In the case of tiles with variable thickness along the broken edge, only approximate results are produced. The shallower the relief, the more exact are the approximations.

The specimen population per tile variant is 1-2 specimens. This is below the minimum population n=5 required for acceptable test results, but as this is only a preliminary experiment to establish the viability for further in-house research, it is considered to be acceptable by MNEXT.

Furthermore, since the tile L = 120 mm > 95 mm, the diameter of rod d required was 20 mm, and the thickness of rubber pad required was 5 mm. The performed testing with d = 10 mm and t = 5 mm was not Eurocode-compliant due to insufficient testing apparatus and should be corrected when beyond the preliminary testing phase.

3.3. Test Specimens Design

3.3.1. Specimen design:

In order to test compressive strength, the cube specimens will be compliant to the NEN-EN 12390-1:2021 standards, being a cube with a nominal size of 150 mm x 150 mm x 150 mm.

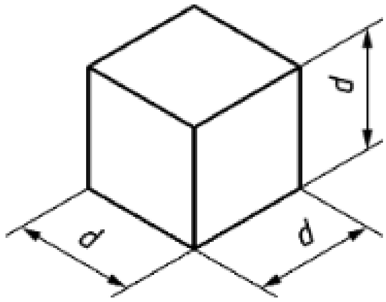

Figure 7 Cube design, NEN-EN 12390-1:2021

with

$d = 150$ mm

$A_c = 150 \text{ mm} * 150 \text{ mm} = 22500 \text{ mm}^2$

In order to test modulus of rupture, breaking load and breaking strength, the tile specimens will be as follows:

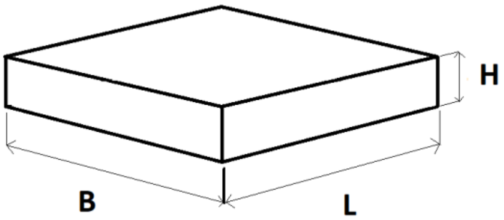

Figure 8 Tile design

with

$L = B = 120$ mm

$H = 20$ mm = thickness

3.3.2. Concrete mixture design

As Geopolymer concrete is a relatively new field, there is no standard geopolymer concrete mix design procedure. The most effective and repeatable solution is thus to replicate a working mix design by an existing study as baseline and modify the mixture in accordance with the research purpose. The mixture used by this project is based off the mix design utilized by S.V. Patankar et al (Patankar S. V. G. Y., 2015). Based off this mix design, this paper would thus substitute 10% of the total volume of the M30 mixture with Miscanthus fibre pretreated with Sodium Alginate.

The experiment will compose of 4 sets of materials, with 2 to 3 cubes for each sets as follows:

- Set 1: Geopolymer with no natural fibre added: control baseline mixture with no additives or modifications.
- Set 2: Geopolymer with 5 wt% Sodium Hydroxide-treated Miscanthus fibre: control with fibre substitution pretreatment using conventional Miscanthus fibre pretreatment method, dried fibre.
- Set 3: Geopolymer with 10 wt% Sodium Alginate-treated Miscanthus fibre: variant 1 with Miscanthus fibre pretreated using Sodium Alginate 10wt% solution, saturated fibre.

- Set 4: Geopolymer with 5 wt% Sodium Alginate-treated Miscanthus fibre: variant 2 with Miscanthus fibre pretreated using Sodium Alginate 5wt% solution, saturated fibre.

These mixture variant sets have the following proportion of materials:

Table 6 Geopolymer mixes variants

Set	Name	Fly ash kg/m³	Water content kg/m³	Fibre kg/m³	Coarse aggregate kg/m³	Fine aggregate kg/m³	NaOH kg/m³	Na_2SiO_3 kg/m³	Sodium Alginate kg/m³
1	Control baseline	405	78,79	0	1268,66	683,13	70,88	70,88	0
2	Control NaOH dry treated fibre	405	78,79	25,42	1268,66	683,13	70,88	70,88	0
3	Sodium Alginate 10wt% saturated fibre	405	169,58	25,42	1268,66	683,13	70,88	70,88	4,54
4	Sodium Alginate 5wt% saturated fibre	405	169,58	25,42	1268,66	683,13	70,88	70,88	9,08

Set 1 and 2 would serve as control variants for the experiment, with set 1 being an unaltered M30 concrete mixture by S.V. Patankar with no modifications or additives, set 2 being the M30 concrete mixture with 10% of the total volume of the mixture substituted with Miscanthus fibre filler pretreated using conventional 5wt% NaOH pretreatment methods. Set 3 and 4 would be the 2 variants proposed by this research paper, with 10% of the total volume of the mixture replaced with Miscanthus fibre filler pretreated using 10 and 5 percent by weight Sodium Alginate solution respectively without drying the fibre before being added to the mixture.

Set 2, using conventional natural fibre pretreatment method will not have any additional water content added to the geopolymer concrete mixture. Set 1 and 2 have a water/fly ash ratio of 0,195, 8% higher than the proposed optimal 0,18 water/fly ash ratio by D. Hardjito (Djwantoro Hardjito, 2005) for highest compressive strength and 27,3% lower than the proposed optimal water/fly ash ratio by S. V. Pattankar (Patankar S. V. G. Y., 2015) for the optimal balance between compression strength and workability for GPC; however for set 2 the actual water/fly ash ratio is lower due to the Miscanthus fibre absorbing and locking a portion of the water content from the mixture.

For sets 3 and 4, the Miscanthus fibre will not be dried out during the pretreatment process. The solution used for pretreatment process would be utilized as the excess water for saturation of the fibre in order to increase the workability of the fresh geopolymer concrete mixture. This additional water content will be 90,79 kg/m³ (approx. 215%) for set 3 and 4. Set 1 and 2 have a water/fly ash ratio of 0,419, 232,6% higher than the proposed optimal 0,18 water/fly ash ratio by D. Hardjito (Djwantoro Hardjito, 2005) for highest compressive strength for GPC and 156,5% higher than the proposed optimal water/fly ash ratio by S. V. Pattankar (Patankar S. V. G. Y., 2015) for the optimal balance between compression strength and workability for GPC, and 16,2% lower than the proposed 0,5 water/cement ratio for optimal compressive strength for OPC concrete with Miscanthus fibre aggregate substitution.

3.3.3. *Material procurement & Preparation*

All materials used in this research had been procured by MNEXT in accordance with discussion and agreement with Drs. Marianna Coelho and the rest of the research group.

❖ *Aggregates:*

Aggregates are sourced from De Hoop Terneuzen, Deutschlandweg 2, the Netherlands.

The following sieve analysis are taken separately on 1 kg of fine aggregate and 2kg of coarse aggregate, and is taken as representative for all fine and coarse aggregate used by this research. The sieve analysis is performed in accordance with NEN-EN 933-2 for coarse aggregates and NEN-EN 12620 for dry aggregates together with member of the MNEXT Geopolymer research group Kisnathas Shandran (Shandran, 2023), with results as follow:

Table 7 Fine aggregate sieve analysis (Shandran, 2023)

Sample: Fine aggregate				
Input mass dry total (M1) = 1000 gram				
Sieve size (mm)	**Mass residue** R1 (g)	**Percentage of sample mass** R1/M1*100%	**Cumulative percentages sieve residue**	**Cumulative percentage fall 100%-sieve residue**
8	0	0	0	100
5.6	37	3,7	3,7	96,3
4	61	6,1	9,8	90,2
2	187	18,7	28,5	71,5
1	685	68,5	97	3
0.5	10	1	98	2
0.25	2	0,2	98,2	1,8
0.125	0	0	98,2	1,8
Sum	**982**	**98,2**	**-**	**-**
Loss	**18**	**1,8**	**-**	**-**

Table 8 Coarse aggregate sieve analysis (Shandran, 2023)

Sample: Coarse aggregate				
Input mass dry total (M1) = 2000 gram				
Sieve size (mm)	**Mass residue** R1 (g)	**Percentage of sample mass** R1/M1*100%	**Cumulative percentages sieve residue**	**Cumulative percentage fall 100%-sieve residue**
31.5	0	0	0	100
22.4	0	0	0	100

16	57	2,85	2,85	97,15
11.2	718	35,9	38,75	61,25
8	731	36,55	75,3	24,7
5.6	379	18,95	94,25	5,75
4	95	4,75	99	1
2	8	0,4	99,4	0,6
1	2	0,1	99,5	0,5
Sum	**1990**	**99,5**	**-**	**-**
Loss	**10**	**0,5**	**-**	**-**

- *Fly ash:*

Fly ash is sourced from De Hoop Terneuzen, Deutschlandweg 2, the Netherlands.

- *Miscanthus Fibre:*

Chopped and dried short-strand Miscanthus fibre (0.5-2mm) is sourced from Vibers B.V.

After a literature review, the pretreatment method chosen is modified from Maya's biobased Sodium Alginate treatment for flax fibre for PLA and PHBV composites (Maya, 2022) which is modified from Mani's Sodium Alginate pretreatment for lignocellulosic fibres (Mani, 1990). A 5wt% and 10wt% concentration of Sodium Alginate was used to pretreat the Miscanthus fibre due to not using a coagulant. At 5wt% and 10wt%, the Sodium Alginate form a hydrogel in the treatment solution, and Miscanthus fibre is mixed to soak in these pretreatment solutions before being taken out and squeezed dry. As the Sodium Alginate solution only coat the fibre's exterior and does not have a deleterious effect on the fibre, the pretreatment solution is used as the saturation solution, reducing the energy and labour necessary for these pretreatment methods.

The pretreatment methods are described in the table 9 below:

Table 9 Fibre pretreatment method

Set	Name	Fibre pretreatment method
1	Control baseline	None
2	Control NaOH dry treated fibre	Chopped, dried Miscanthus fibre were left to soak in a 5 wt% NaOH solution for 30 minutes. The solution is then drained out and left to dry in air at room temperature for 15 minutes until no more liquid is leaking freely from the fibre. Miscanthus fibre is then baked in a drying rack in an oven at 100°C until completely dry.
3	Sodium Alginate 10wt% saturated fibre	Chopped, dried Miscanthus fibre were mixed to soak in a 10 wt% Sodium Alginate gel solution for 30 minutes. The solution is then drained out and left to dry at room temperature for 15 minutes until no more liquid is leaking freely from the fibre. Miscanthus fibre is added to the mixer without drying
4	Sodium Alginate 5wt% saturated fibre	Chopped, dried Miscanthus fibre were mixed to soak in a 5 wt% Sodium Alginate gel solution for 30 minutes. The solution is then drained out and left

		to dry at room temperature for 15 minutes until no more liquid is leaking freely from the fibre. Miscanthus fibre is added to the mixer without drying.

The above treatment methods for set 2 produces completely dried out Miscanthus fibre, while set 3 and 4 every 0,28 kg of fibre would be saturated with an extra 1 kg of water that was used to treat the fibre. This extra water content is meant to ensure the workability of the GPC mixture, and would impact the compressive and flexural strength of the resulting GPC mixture.

Unlike with Mani's pretreatment method using 0,25wt% Sodium Alginate and CaCl2 coagulant solution, the treatment methods for set 3 and 4 do not use coagulant, thus requiring a higher Sodium Alginate concentration to maintain treatment effectiveness.

- *Sodium Hydroxide (NaOH):*

Sodium Hydroxide (NaOH) 40 g/mol is sourced from Boom B.V. (Boomlab laboratory supplier), Rabroekenweg 20, 7942 JE Meppel, the Netherlands. ID Code: 24076051291.2500 10 PROD2300043 17240102 .

- *Sodium Silicate (Na2SiO3):*

Sodium Silicate (Na2SiO3) solution was sourced from Merck KGaA, 64271 Darmstadt, Germany, of Sigma-Aldrich/Millipore and Merck group. Batch number: K54485421 245.

- *Sodium Alginate:*

Food-grade Sodium Alginate powder was sourced from Special Ingredients Ltd. Foxwood Industrial Park, S41 9RN, the United Kingdom. Batch number: K54485421 245.

- *Mould:*

The moulds for the cubes used are 15 cm x 15 cm x 15 cm plastic moulds supplied by the Scalda Concrete Lab (Vlissingen, the Netherlands).

The moulds for the tiles used 12 cm x 12 cm x 2 cm, 3D printed by MNEXT.

3.3.4. Production method of specimens

3.3.4.1. Production of Geopolymer concrete block:

The production of geopolymer concrete blocks consists of 2 main phases: the mixing of the geopolymer concrete mixture, and the curing of the geopolymer concrete cubes. These 2 phases can be described in the following 7 steps:

- **Step 1:** Aggregate and fly ash is mixed in a dry state in the concrete mixer.
- **Step 2:** Prepare the activator solution: Sodium Hydroxide (NaOH) pellets is slowly added to the water for the mixture in an external plastic container under steady stirring. Due to the high concentration of NaOH, the exothermic reaction releases a lot of heat and fumes, and thus the solution requires cooling to keep the reaction speed under control. For the conditions of Scalda Concrete Lab, this cooling is performed via keeping the plastic container in cold tap water sink and changing the water when it gets unacceptably hot (>50℃). After the NaOH pellet is dissolved, Sodium Silicate is added to the solution and stirred until mixed.
- **Step 3:** The activator solution is added to the mixture in the concrete mixer. If the variant has natural fibre, the pretreated natural fibre is added.
- **Step 4:** Mix the ingredients together for around 3-4 minutes like normal concrete.

- **Step 5:** Perform the slump test with the fresh geopolymer concrete. Cast the geopolymer concrete mix to 150 x 150 x 150 mm^3 cube moulds and 120 x 120 x 20 mm^3 tile moulds, use vibration table to ensure all the air bubbles inside the moulds are filled out.
- **Step 6:** After 7 days, the cubes and tiles are demoulded. The surviving cubes and tiles are cured in an oven at 60°C for 24 hours.
- **Step 7:** The cubes and tiles are left to cure at room temperature for a further 20 days, to a total of 28 days before testing is carried out.

Originally, the geopolymer concrete cubes and tiles were meant to be demoulded after one day and oven cured for 24 hours, left to cure at ambient temperature for 7 more days before testing. However, delays with the oven and an unplanned breakdown of the compression testing machine led to changes to the plans as detailed above.

3.4. Multi-Criteria Analysis (MCA)

Multi-Criteria Analysis (MCA) is a method of appraising the effectiveness of one or more solutions to a problem by quantifying and comparing specific chosen criteria. Each solution/variant is given a quantified score based on a variety of chosen criteria and a weight factor representing the importance of that criteria in the decision-making process to give a final score; the final scores of all variants are then compared to choose a solution/variant. There are multiple methods to calculating MCA score, and the method chosen for this paper is the Analytical Hierarchy Process (AHP).

3.4.1. Analytical Hierarchy Process (AHP)

The method of MCA chosen for this paper is the Analytical Hierarchy Process (AHP), a structured technique for analysis and decision-making developed by W. R. Saaty. The method decomposes a decision problem into a hierarchy of criteria and alternatives from which is used to analyse the problem and reach a decision.

Steps of performing AHP: (Saaty, 1987; Rainer Haas)

1. Structure the problem: Identify the main objective/goal, criteria, and alternatives to a problem. Model the goal, criteria related to the problem and available alternatives into a hierarchical structure.
2. Determine the relative importance of each criterion: Calculate the relative weight and priorities of each element and criteria in the hierarchy by analysing the matrices of pairwise comparisons using matrix algebra and eigenvalue. (Saaty, 1987)
3. Pairwise comparisons: Set up matrices to compare each criterion with every other criterion regarding their relative importance or priority. These comparisons are made using a scale of relative importance ranging from 1 to 9, where 1 indicates equal importance, and 9 indicates extreme importance. Reciprocal means if activity *i* has one of the above numbers assigned to it when compared with activity *j*, then *j* has the reciprocal value when compared with *i*. (Saaty, 1987)

Table 10 Scale of relative importance

Scale	Numerical Rating	Reciprocal
Extremely Preferred	9	1/9
Very strong to extremely	8	1/8

Very strongly preferred	7	1/7
Strongly to very strongly	6	1/6
Strongly preferred	5	1/5
Moderately to strongly	4	1/4
Moderately preferred	3	1/3
Equally to moderately	2	1/2
Equally preferred	1	1

4. Check Consistency: The pairwise matrix in Step 2 and 3 is checked for consistency in order to ensure accuracy. Inconsistent judgments may require reassessment or adjustment. Consistency is calculated from the Consistency Index (CI), and the Consistency Ratio (CR) which is derived from the relationship between the Consistency Index (CI) and the Random consistency Index (RI). The matrices are considered consistent when CR<0,1:

$$CI = \frac{\lambda max - n}{n - 1}$$

$$CR = \frac{CI}{RI}$$

RI is a fixed value based on the number of criteria being evaluated:

Table 11 Random consistency index table (Saaty, 1987)

N	1	2	3	4	5	6	7	8	9	10
RI	0	0	0,58	0,9	1,12	1,24	1,32	1,41	1,45	1,49

5. Weigh and synthesize the results: The score of each variant per criteria and the weight of each criterion is then combined to get the overall ranking or priority of each variant.

3.4.2. Criteria

The variant sets 1-4 will be evaluated based on the following criteria:

Criteria	Definition
Compressive Strength	The main function of concrete in civil engineering is to withstand heavy compressive load, thus one of the most important factors to evaluate a (geopolymer) concrete is that mixture's compressive strength. The higher the compressive strength, the better the geopolymer concrete mixture.
Specific Compressive Strength	It is also important to consider the dead load of a concrete structure imposed by its self-weight, which is affected by the density of the concrete used to manufacture that structure. A column built from concrete with a lower density (less self-weight) can be built higher without collapsing or withstand a higher load.

	Specific strength, also known as strength-to-weight ratio, is one factor used to represent this relationship. A geopolymer concrete mixture with higher specific strength is better than a mixture with lower specific strength.
Workability	Workability refers to how easy a fresh concrete mixture can be mixed, placed, and consolidated with minimal segregation. Workability is a vital property of concrete, and is related with the strength of a concrete mixture. Furthermore, as can be observed later in section 4.5., workability's importance in the strength of concrete tile is further increased.
Modulus of Rupture	Modulus of rupture measures the highest stress of a material just before it yields in a tensile test. In this research Modulus of Rupture measure of the flexural strength of a concrete tile. As the client of this research is developing a geopolymer concrete tile, this is an important criterion to consider in evaluation.
Sustainability	The goal of this research is to develop a biobased geopolymer concrete using Miscanthus fibre pretreated using a biobased method, For this research, a mixture using more natural fibre as aggregate substitution is better, and the pretreatment method that utilize less material, energy and uses more biobased chemical is better.
Labor/Time	Another important criterion that affect sustainability, cost and practicality of a concrete mixture is the amount of time and labor a mixture require to create when including the pretreatment process for the natural fibre. The less time and labor consuming mixture cost less to produce and is better.

4. Results

4.1. Slump test

The slump test was performed for each batch of geopolymer concrete of every sets gives the following results:

Table 12 Slump test result

Set	Slump (mm)	Classification
1	10	Zero
2	5	Zero
3	0	Zero
4	28	S1

Numerically, geopolymer with miscanthus fibre pretreated conventionally (set 2) did not have a remarkable decrease in workability, but it was noticeable during observation. This was observed during the casting of the geopolymer concrete, where the mortar was noticeably dryer compared to the baseline control, being unable to fill out the moulds completely. This is likely due to the dry Miscanthus fibre absorbing the water content in the mixture, despite having been pretreated with NaOH, and thus lowering the workability. Conversely, the geopolymer concrete with miscanthus fibre pretreated using 5wt% Sodium alginate (set 4), has a noticeable increase in slump, almost triple the baseline control (set 1) and increasing the classification to S1. This is likely due to the increased water content (approx. 2,15 times) giving the mixture more workability and counteracting the decrease in workability due to the natural fibre absorption of water and the hydrophilic Sodium Alginate on the pretreated fibre. Though this is still undesirable, it is still an improvement. On the other hand, it can be said that the Sodium Alginate had increased the viscosity of the mixture despite the increase in water – for the water content for set 4 had increased 2,15 times while the slump only increased by 1,87 times. The viscosity increase effect of Sodium Alginate on the geopolymer concrete mixture can be seen more clearly with set 3 where the mixture had twice the amount of Sodium alginate in the pretreated fibre, where the slump was reduced practically to zero, even lower than in set 2 where there is still a low but noticeable amount of slump despite having 2,15 times the water content of set 1 and 2. While this effect may not be preferable for these 2 variants (due to still being below the desired S2-S3 workability), it will be an important factor for future consideration when trying to create a mixture with a higher percentage of Miscanthus fibre pretreated with Sodium Alginate without drying.

Furthermore, the 10wt% Sodium Alginate used to pretreat the Miscanthus fibre for set 3 resulted in a highly viscous slurry of hydrogel that made the pretreatment mixing difficult. Set 4's pretreatment mixture of 5wt% Sodium Alginate resulted in a much less viscous solution that mixed into Miscanthus fibre more easily.

4.2. Produced specimens (samples)

The experiment produced a total of 11 cubes and 7 tiles. The ID of each specimen was logged under the format "C/T – *set* – *number*" whereas C stands for Cube, T stands for Tile, *set* identifies the mixture set the specimen belong to in this particular research paper, and *number* being the production order of the produced specimen. The excel spreadsheet also keep track of the mixture set according to the broader research group by column "Mixture". The data from the specimens was logged using ColorNote software on Samsung Note S9 phone, exported and organized manually to MS Excel document titled 'Timeline planning.xlsx'.

- **Set 1: Geopolymer with no natural fibre added (control baseline).**

Figure 9 Specimens C01-01 and C01-02 (Shandran, 2023)

- **Set 2: Geopolymer with Sodium Hydroxide-treated, dried Miscanthus fibre (control standard treatment)**

All GPC specimens in this set displayed signs of fluorescent. This is potentially a sign of excessive water content in the GPC mixture.

Figure 10 Specimen C02-01

Figure 11 Specimen C02-02

Figure 12 Specimen C02-03

- **Set 3: Geopolymer with 10wt% Sodium Alginate-treated Miscanthus fibre (variant 1)**

All GPC specimens in this set displayed signs of fluorescent. This is potentially a sign of excessive water content in the GPC mixture.

Figure 13 Specimen C03-01

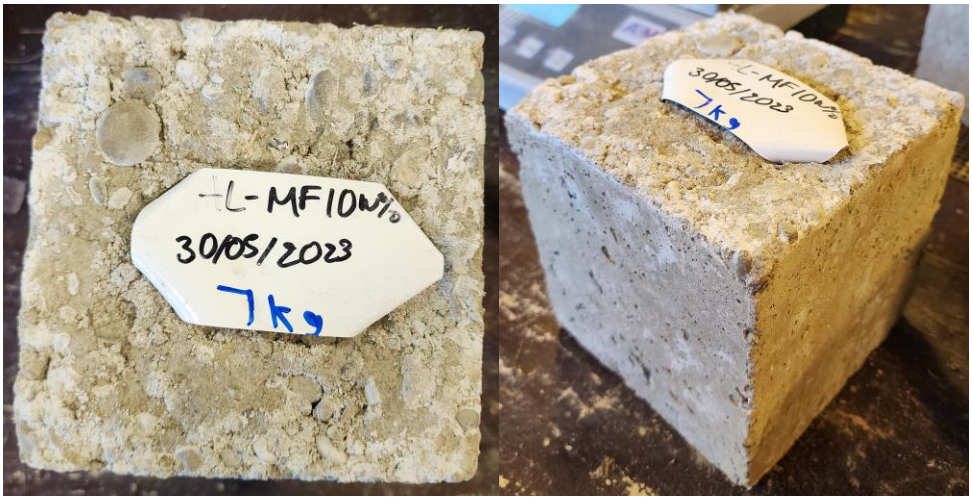

Figure 14 Specimen C03-02

Figure 15 Specimen C03-03

❖ **Set 4: Geopolymer with 5wt% Sodium Alginate-treated Miscanthus fibre (variant 2)**

All GPC specimens in this set displayed signs of fluorescent. This is potentially a sign of excessive water content in the GPC mixture.

Figure 16 Specimen C04-01

5

Figure 17 Specimen C04-02

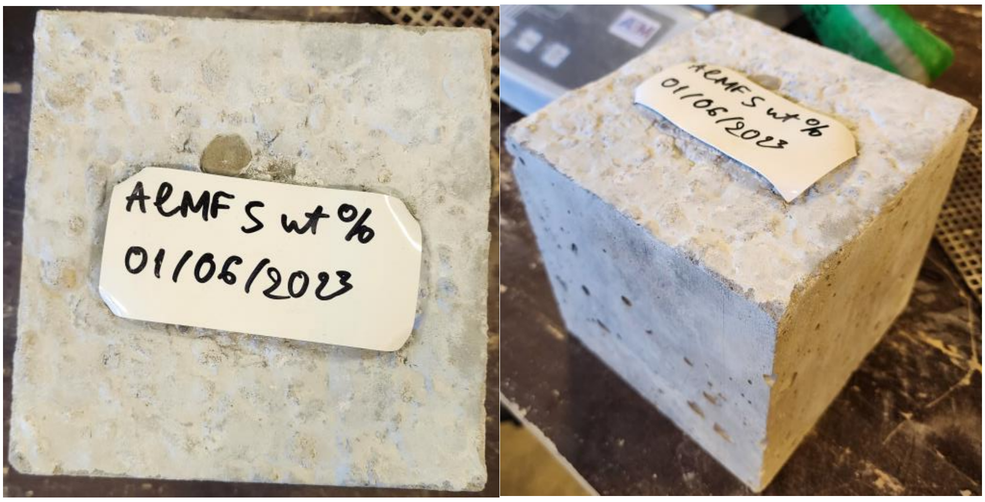

Figure 18 Specimen C04-03

4.3. Compressive test

The resultant data from the experiment is displayed in the following table 13. The density is the weight of the cube casted from the geopolymer concrete mixture after curing for 27 days + 1 days in the oven divided by the cube's dimensions. Density was rounded to the nearest 0, all other units were rounded to the nearest 0,1:

Table 13 Geopolymer concrete cube data (Sets 01-04)

ID	Set	Date produced	Weight (g)	Density (kg/m^3)	Max Force (kN)	Comp. strength (MPa)	Specific strength (kN*m/kg)
C01-01	1	23-05-23	7672	2273	967,5	43,0	18,9
C01-02	1	23-05-23	7797	2310	1080	48,0	20,8
C02-01	2	06-06-23	7349	2178	468,6	20,8	9,6
C02-02	2	06-06-23	7280	2157	571,0	25,4	11,8
C02-03	2	06-06-23	7200	2133	264,6	11,8	5,5
C03-01	3	30-05-23	6818	2020	326,1	14,5	7,2
C03-02	3	30-05-23	7029	2083	327,0	14,5	7,0
C03-03	3	30-05-23	7070	2095	336,1	14,9	7,1
C04-01	4	01-06-23	6923	2051	1318,1	58,6	28,6
C04-02	4	01-06-23	7113	2108	325,6	14,5	6,9
C04-03	4	01-06-23	7028	2082	256,8	11,4	5,5

Analysing table 14 gives the following results:

Table 14 Compressive force, Compressive strength, and Specific strength of all specimens and average of each set

Set ID	Max Comp Force (kN)	Comp. strength (MPa)	Density (kg/m3)	Specific Strength (kN*m/kg)
1	**1023,8**	**45,5**	**2292**	**19,8**
C01-01	967,5	43,0	2273	18,9

C01-02	1080,0	48,0	2310	20,8
2	**434,7**	**19,3**	**2156**	**8,9**
C02-01	468,6	20,8	2177	9,6
C02-02	571,0	25,4	2157	11,8
C02-03	264,6	11,8	2133	5,5
3	**329,7**	**14,7**	**2066**	**7,1**
C03-01	326,1	14,5	2020	7,2
C03-02	327,0	14,5	2083	7,0
C03-03	336,1	14,9	2095	7,1
4	**633,5**	**28,2**	**2080**	**13,6**
C04-01	1318,1	58,6	2051	28,6
C04-02	325,6	14,5	2108	6,9
C04-03	256,8	11,4	2082	5,5

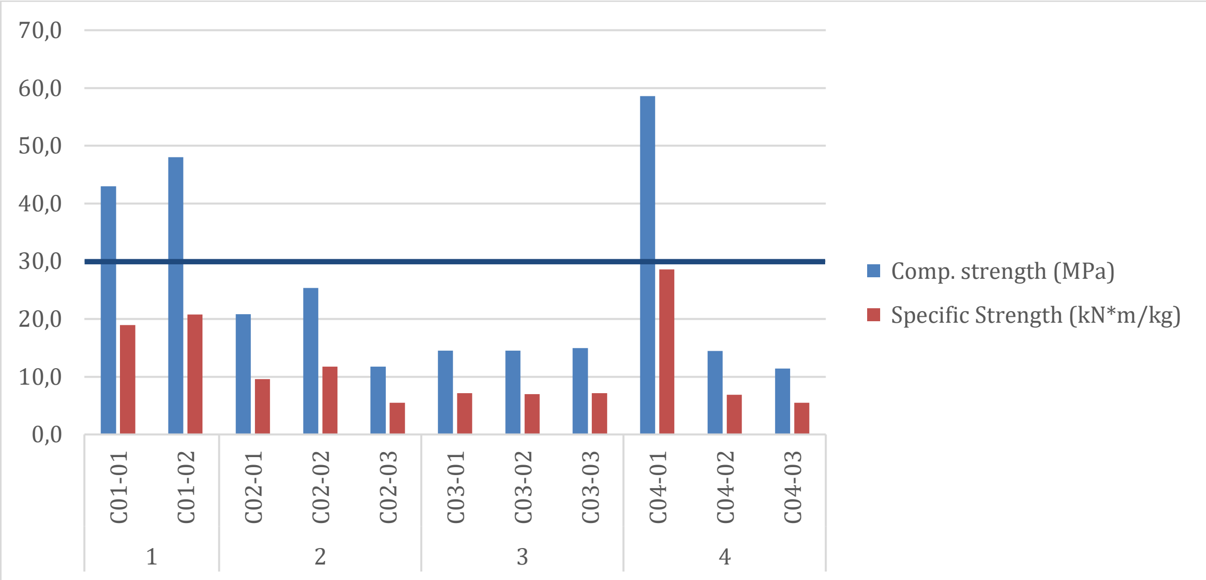

Figure 19 Compressive Strength and Specific Strength graph for all specimens

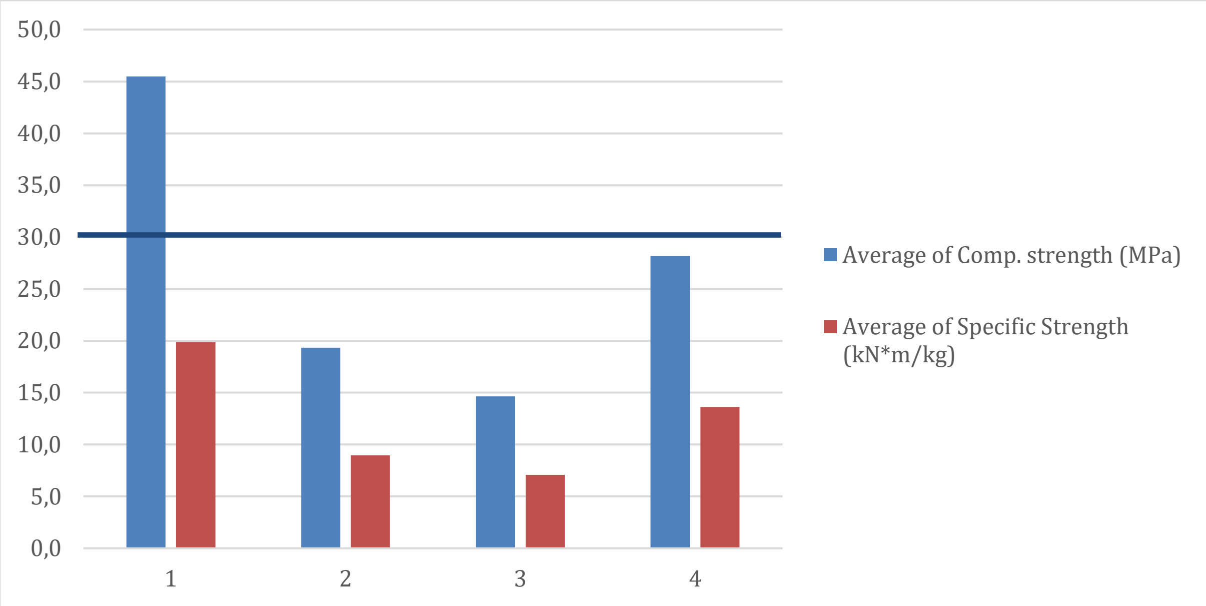

Figure 20 Average Compressive Strength and Specific Strength for each set

As can be seen in Table 14, the compressive strength of the M30 geopolymer concrete mixture were greatly reduced when 10% volume of the mixture was substituted with Miscanthus fibre, reducing by 57,5%, 67,8% and 38,1% respectively for sets 2, 3 and 4 compared to the control baseline of set 1. The aggregate substitution only resulted in set 2 reducing in density by 5,9%, while the increased water content that is then evaporated from the cured geopolymer concrete block from the saturated pretreated fibre of set 3 and 4 further reduced the density by 10,3% and 9,2% respectively comparing to the control baseline of set 1. This resulted in a Specific Compressive Strength decrease of 54,9%, 64,3% and 31,3% respectively for sets 2, 3 and 4 when compared to set 1.

Regardless, on average set 4 reached 93,8% of the Compressive Strength target goal 30 MPa, and set 3 reached 48,8% of the target 30 MPa. Furthermore, the Specific Compressive Strength of set 4 was much higher than the other 2 set where aggregate substitution happen – being 52,8% higher than of set 2 and almost twice (1,92 times) as high as set 3. However when analyzing per specimen, it can be seen that set 4 had a great variance of Compressive strength between specimens, with compressive strength differing by 5,2 times between the highest (C04-01) and lowest (C04-03) compressive strength for set 4. It can be inferred from the results that outlier specimen C04-01's very high compressive strength may have caused the whole set 4 to have its mean compressive strength be higher than the actual case. Without the outliers C04-01 and C04-03, the mean compressive strength and specific compressive strength of set 4 would be 14,5 MPa and 6,7 kN*m/kg, nearly equal to the mean 14,7 MPa and 7,1 kN*m/kg of set 3. However, as can be seen later in table 15 and 16 of section 4.5., mixture set 4 produces samples with consistently higher breaking strength and modulus of rupture than set 3.

Compared to the control pretreatment method set 2, set 3 had 23,8% less compressive strength. Due to set 3 having 4,2% less density than set 2, the resultant Specific strength of set 3 is only 20,2% less than set 2. The decrease in strength potentially corresponds to set 3 having 2,15 times more water than set 2. The data points of set 3 were also the most reliable and consistent of all 4 sets, with variance within ±0,2 of the chosen units – an order of magnitude smaller than the variance of the 2 control sets 1 and 2, and 2 order of magnitude smaller than the variance of set 3. This reliability is rather unexpected due to the set 3's low workability. Furthermore, all geopolymer concrete mixture tested have a density of >2000 kg/m3, making all specimens normal-weight concrete. All mixtures with natural fibre substitution are lighter than the baseline geopolymer concrete mixture without natural fibre substitution.

In conclusion: Of the 2 proposed variants set 3 and 4, set 4 Sodium Alginate 5wt% had better compressive and specific strength, but the result from set 3 Sodium Alginate 10wt% is much more reliable. Neither sets 3 or 4 reached the functional requirement of 30 MPa in Compressive Strength, but set 4 got very close when counting the outlier values. Further repeat experiments are necessary to conclude whether set 4 data is reliable.

4.4. Failure mode of cubes

All cube specimens displayed satisfactory failure mode according to NEN-EN 12390-3:2019 - all four exposed faces are cracked approximately equally, generally with little damage to faces in contact with the plates.

- **Set 1: Geopolymer with no natural fibre added (control baseline).**

No available image.

- **Set 2: Geopolymer with Sodium Hydroxide-treated, dried Miscanthus fibre (control standard treatment)**

Figure 21 Specimen C02-01 failure mode

Figure 22 Specimen C02-02 failure mode

Figure 23 Specimen C02-03 failure mode

- **Set 3: Geopolymer with 10wt% Sodium Alginate-treated Miscanthus fibre (variant 1)**

Figure 24 Specimen C03-01 failure mode

Figure 25 Specimen C03-02 failure mode

Figure 26 Specimen C03-03 failure mode

❖ **Set 4: Geopolymer with 5wt% Sodium Alginate-treated Miscanthus fibre (variant 2)**

Figure 27 Specimen C04-01 failure mode

Figure 28 Specimen C04-02 failure mode

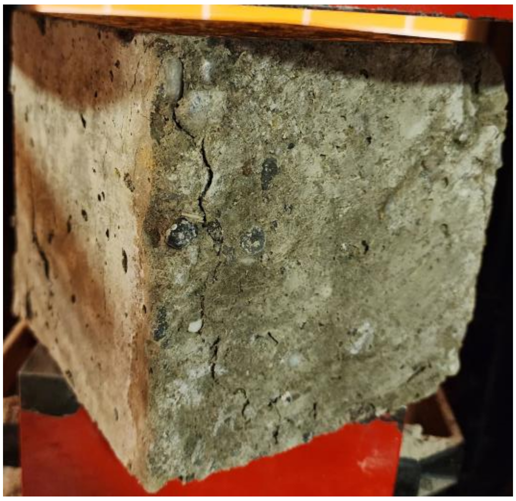

Figure 29 Specimen C04-03 failure mode

4.5. Modulus of rupture and Breaking strength test

The resultant data from the experiment is displayed in the following tables 15. Breaking loads were rounded to the nearest 0, all other units were rounded to the nearest 0,1:

Table 15 Geopolymer concrete tile data (Sets 01-04)

ID	Set	Date produced	Width (mm)	Height (mm)	Breaking load (kN)	Breaking strength (kN)	Modulus of rupture (N/mm2)
T01-01	1	23-05-23	119,8	22	972	811,4	2514,5
T01-02	1	23-05-23	119,7	23,2	1560	1303,3	3632,0
T02-01	2	09-05-23	120,4	23,2	443	367,9	1025,4
T02-02	2	06-06-23	119,5	20,75	855	715,5	2492,6
T03-01	3	30-05-23	121	20,2	471	389,3	1430,9
T04-01	4	01-06-23	119,8	21,5	958	799,7	2594,9
T04-02	4	01-06-23	119,8	21,5	1210	1010,0	3277,5

Analysing table 15 gives the following results:

Table 16 Breaking load, Breaking Strength, and Modulus of rupture of all specimens and average of each set

Set ID	Breaking load (kN)	Breaking strength (kN)	Modulus of rupture (N/mm2 = MPa)
1	**1266**	**1057,3**	**3073,3**
T01-01	972	811,4	2514,5
T01-02	1560	1303,3	3632,0
2	**649**	**541,7**	**1759,0**

T02-01	443	367,9	1025,4
T02-02	855	715,5	2492,6
3	**471**	**389,3**	**1430,9**
T03-01	471	389,3	1430,9
4	**1084**	**904,8**	**2936,2**
T04-01	958	799,7	2594,9
T04-02	1210	1010,0	3277,5

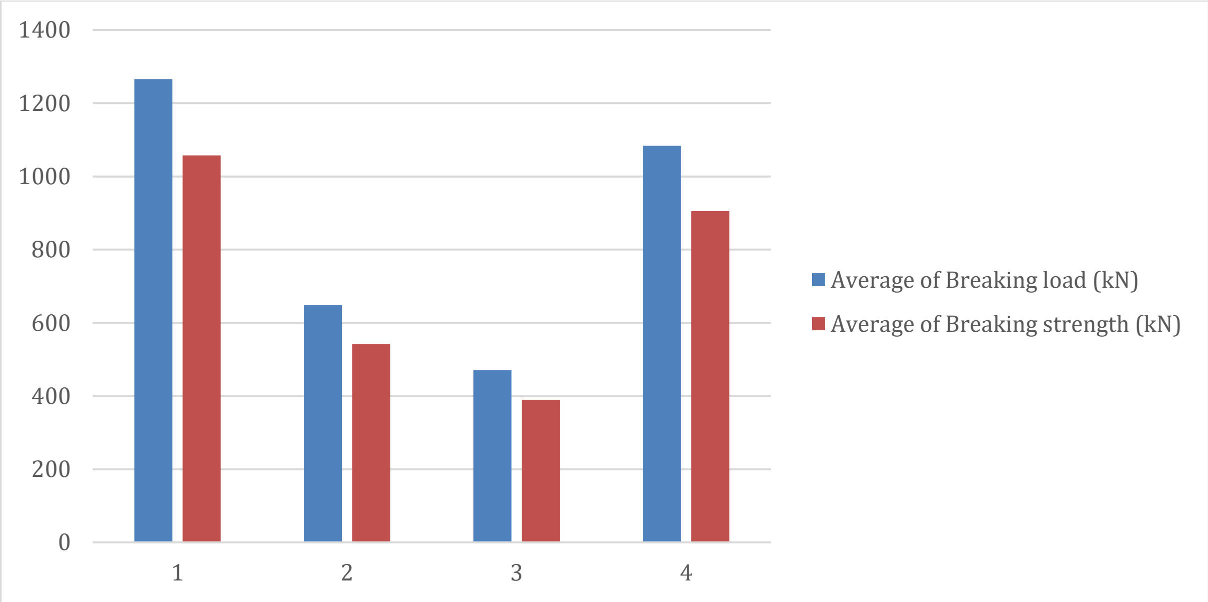

Figure 30 Average of Breaking load and Breaking Strength of each set

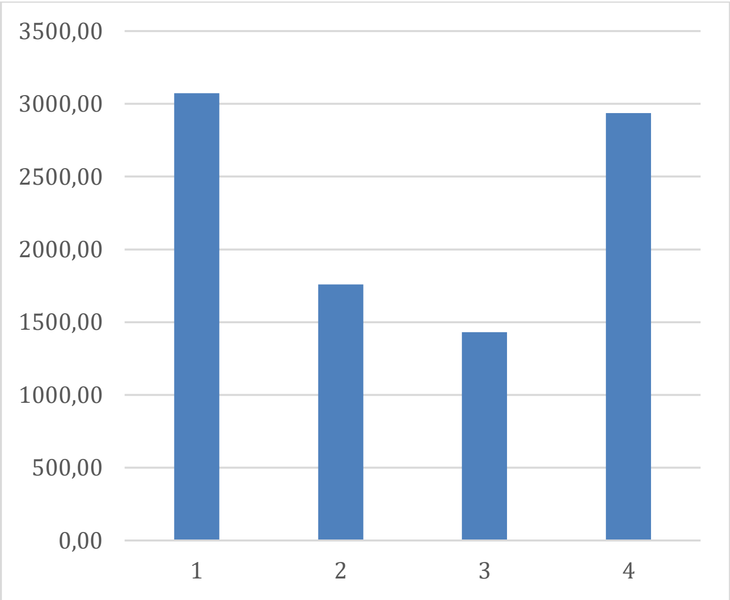

Figure 31 Average of Modulus of Rupture (N/mm2) of each set

Again, it can be seen that the breaking strength and modulus of rupture of the M30 geopolymer concrete mixture were greatly reduced when Miscanthus fibre substitution happen. The control baseline tile set 1 had its breaking load decrease by 48,7%, 62,8% and 14,4%, breaking strength decrease by 48,8%, 63,2% and 14,4%, and modulus of rupture decrease by 42,7%, 53,4% and 4,5% respectively for tile sets 2, 3 and 4.

Similar to the compressive test, tile set 4 had much better breaking strength and modulus of rupture than tile sets 2 and 3, with 67% higher breaking load, breaking strength and modulus of rupture than the control pretreatment method set 2, and 2,3 times higher breaking load and breaking strength and 2,05 times higher modulus of rupture than the tile set 3. Furthermore, the decrease in modulus of rupture due to aggregate substitution is not as great as the decrease in compressive and specific strength, with set 2 and 3 having approx. 10% less decrease in modulus of rupture compared to the decrease in compressive strength, while set 4 has significantly (34%) less decrease in rupture compared to the decrease in compressive strength. This is likely due to the innate property of the Miscanthus natural fibre aggregate replacement being more effective at resisting against tensile and flexural stress than against compressive stress.

The difference in flexural strength and modulus of rupture correlates with the workability observed during slump testing in section 4.1., where set 1 and 4 had the most workability (10mm and 28mm of slump) and consequently had the best modulus of rupture (3073,3 MPa and 2936,2 MPa respectively, a difference of less than 5%), set 3 had the worst workability (zero slump) and the lowest modulus of rupture (389,3 MPa), and set 4 had the second worst workability (5 mm of slump) and the second lowest modulus of rupture (541,7 MPa). The reason for this correlation potentially is due to the nature of the tile mould. Tile moulds are thin and difficult to fill out, so a more fluid and workable mixture may have allowed the tile mould to be filled out completely and give full strength to the mixture. This can be observed as all the tiles with >700 kN breaking strength (T01-01, T01-02, T04-01, T04-02 and T02-02) had smooth and filled out surface and even cross section, while tiles with <400 kN (T03-01 and T02-01) had much rougher surface and uneven cross section, potentially causing much higher local stress where the cross sections were thinner, leading to earlier failure. Regardless, even when disregarding the tile with uneven surface and area, tile set 4 had higher breaking strength and modulus of rupture than the control pretreatment method set 2 (the weaker tile T04-01 had 12% higher breaking load, 11,8% higher breaking strength and 4,1% higher modulus of rupture compared to tile T02-02) and still reached almost parity with the control baseline tile set 1 despite the much higher water content and substitution of aggregate with Miscanthus fibre.

Tile set 3 has 1 tile, T03-01 which has 27,4% less breaking load, 28,1% less breaking strength and 18,6% less modulus of rupture than the average of tile set 2, but has 6,3% higher breaking load, 5,8% higher breaking strength and 39,6% higher modulus of rupture when comparing to the tile T02-01 which also has uneven surface and cross section despite having more water.

In conclusion: From evidence, we can see that workability has a very high correlation with the strength and modulus of rupture of the tiles. For this reason, the best tile set is the tile set 4 which had almost parity with the control baseline tile set 1 despite the substitution of aggregate with Miscanthus fibre. Furthermore, it can be potentially posited that due to the flexure action of the geopolymer tile, natural fibre is particularly suitable for aggregate substitution as aggregate substation for geopolymer concrete tile (unlike with geopolymer concrete cubes) as long as the moisture absorption by the natural fibre can be controlled to ensure suitable workability. Further repeat experiments is necessary for tile set 2 and 3 in order to reach a conclusive result.

4.6. Failure mode of tiles

All tiles specimens displayed satisfactory failure mode.

- **Set 1: Geopolymer with no natural fibre added (control baseline).**

No available image.

- **Set 2: Geopolymer with Sodium Hydroxide-treated, dried Miscanthus fibre (control standard treatment)**

Figure 32 Specimen T02-01

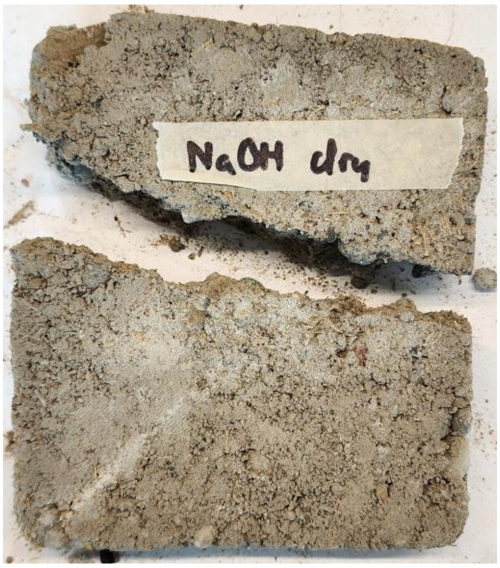

Figure 33 Specimen T02-02

- **Set 3: Geopolymer with 10wt% Sodium Alginate-treated Miscanthus fibre (variant 1)**

Figure 34 Specimen T03-01

- **Set 4: Geopolymer with 5wt% Sodium Alginate-treated Miscanthus fibre (variant 2)**

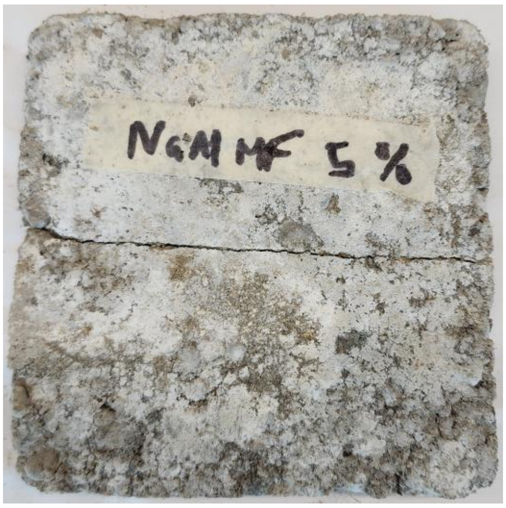

Figure 35 Specimen T04-01

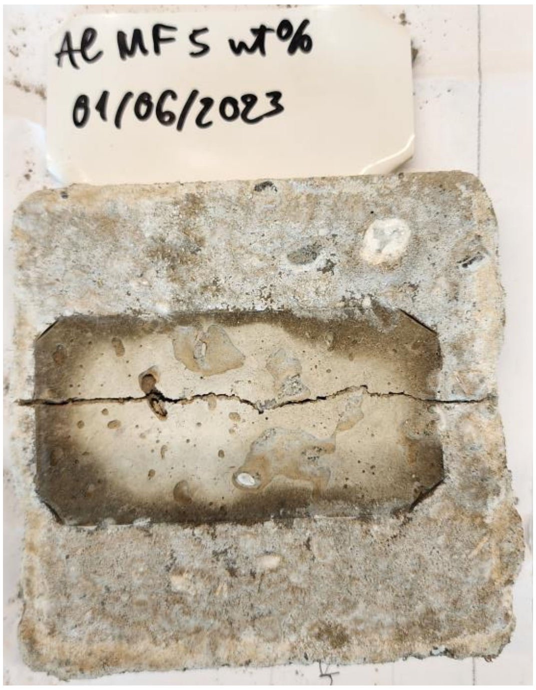

Figure 36 Specimen T04-02

4.7. Multi-Criteria Analysis (MCA)

Step 1: Hierarchy creation

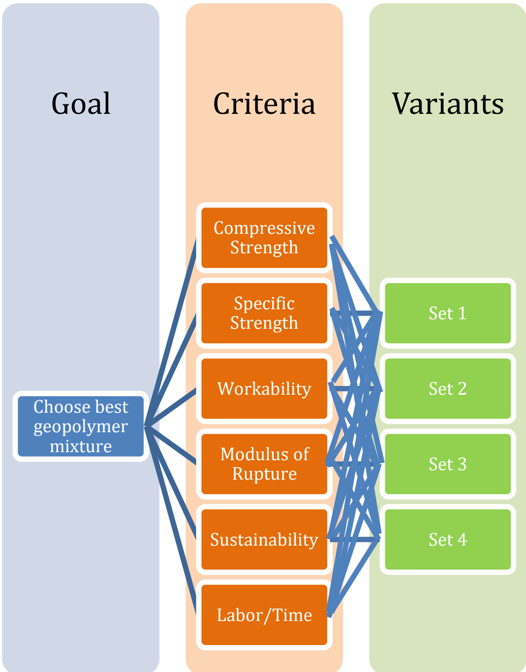

Figure 37 Hierarchy of MCA

Step 2: Determining the relative importance of criteria

The relative importance of each criterion is determined via pairwise comparison. The pairwise comparison matrix for criteria weight is shown in Table 17; the weight for one criterion compared to the other criteria is selected based on their importance to the research project. The values shown in Table 17 are then used to determine a weight. The weight matrix is calculated by taking the value of each matrix position in table 17 divided by the sum of that column, displayed in the matrix table 18. Normalized Weight of each criterion is then calculated by taking the arithmetic mean of each row. In this research, the most important criterion to evaluate the alternative geopolymer concrete mixture is the specific strength with a weight of 38% of the final score. The criteria of Sustainability and Time/Labour, achieve a combined weight of 15% of the final score.

Table 17 Pairwise comparison matrix for criteria weight

	Compressive Strength	Specific Strength	Workabi lity	Modulus of rupture	Sustainab ility	Labor/T ime
Compressive Strength	1	1/2	2	1	2	4
Specific Strength	2	1	4	2	4	8
Workability	1/2	1/4	1	1/2	1	2
Modulus of rupture	1	1/2	2	1	2	4
Sustainability	1/2	1/4	1	1/2	1	2
Labor/Time	1/4	1/8	1/2	1/4	1/2	1
Sum	5,25	2,63	10,50	5,25	10,50	21,00

Table 18 Calculated weights of criteria.

	Compressive Strength	Specific Strength	Worka bility	Modulus of rupture	Sustain ability	Labor/ Time	Normalized weight (w)
Compressive Strength	0,19	0,19	0,19	0,19	0,19	0,19	0,19
Specific Strength	0,38	0,38	0,38	0,38	0,38	0,38	0,38
Workability	0,10	0,10	0,10	0,10	0,10	0,10	0,10
Modulus of rupture	0,19	0,19	0,19	0,19	0,19	0,19	0,19
Sustainabilit y	0,10	0,10	0,10	0,10	0,10	0,10	0,10
Labor/Time	0	0,05	0,05	0,05	0,05	0,05	0,05
-	-	-	-	-	-	**Sum**	1,0

Step 3: Scoring pairwise comparison for each criterion.

The score of each mixture for each criterion is determined via pairwise comparison. The pairwise comparison score for each criterion are shown in Table 19. The score by each criterion is then aggregated into the matrix table 20.

For the criteria of Compressive strength, Specific Strength, Workability and Modulus of rupture are scored according to the quantifiable values from section 4. Results, with the better results giving a higher score.

For the criterion of Sustainability, baseline geopolymer concrete is considered to be the worst option because it had no biobased aggregate substitution. The biobased Sodium Alginate pretreatment (set 3 and 4) are scored to be more sustainable than the synthetic Sodium Hydroxide pretreatment (set 2) which require overnight oven baking (thus requiring more energy). Of set 3 and 4, set 3 used more Sodium Alginate in the pretreatment (10wt%) than set 4 (5wt%), so set 3 is scored as less sustainable than set 4.

For the criterion of Time/Labour, the baseline geopolymer (set 1) scored the highest because it required no extra steps to pretreat the natural fibre. The Sodium Hydroxide pretreatment require overnight oven baking to dry out so set 2 required more time to pretreat than set 3 and 4, scoring set 2 the lowest. Set 3 and 4 had equivalent pretreatment time, with negligible difference as the higher pretreatment solution concentration of Set 3 required more effort to evenly mix without clumping than set 4, but this is considered to be negligible so set 3 and 4 are scored the same.

Table 19 Pairwise matrix for each criterion

C. Strength	Set 1	Set 2	Set 3	Set 4
Set 1	1	5	6	1 1/2
Set 2	1/5	1	1 1/5	1/3
Set 3	1/6	5/6	1	1/4
Set 4	2/3	3 1/3	4	1
Sum	2,03	10,17	12,20	3,05

C. Strength	Set 1	Set 2	Set 3	Set 4	Score
Set 1	0,49	0,49	0,49	0,49	1,97
Set 2	0,10	0,10	0,10	0,10	0,39
Set 3	0,08	0,08	0,08	0,08	0,33
Set 4	0,33	0,33	0,33	0,33	1,31

S. Strength	Set 1	Set 2	Set 3	Set 4
Set 1	1	5	6	1 1/2
Set 2	1/5	1	1 1/5	1/3
Set 3	1/6	5/6	1	1/4
Set 4	2/3	3 1/3	4	1
Sum	2,03	10,17	12,20	3,05

S. Strength	Set 1	Set 2	Set 3	Set 4	Score
Set 1	0,49	0,49	0,49	0,49	1,97
Set 2	0,10	0,10	0,10	0,10	0,39
Set 3	0,08	0,08	0,08	0,08	0,33
Set 4	0,33	0,33	0,33	0,33	1,31

Workability	Set 1	Set 2	Set 3	Set 4
Set 1	1	2	3	1/3
Set 2	1/2	1	1 1/2	1/6
Set 3	1/3	2/3	1	1/9
Set 4	3	6	9	1
Sum	4,83	9,67	14,50	1,61

Workability	Set 1	Set 2	Set 3	Set 4	Score
Set 1	0,21	0,21	0,21	0,21	0,83
Set 2	0,10	0,10	0,10	0,10	0,41
Set 3	0,07	0,07	0,07	0,07	0,28
Set 4	0,62	0,62	0,62	0,62	2,48

Mod Rupture	Set 1	Set 2	Set 3	Set 4
Set 1	1	3	4	1
Set 2	1/3	1	1 1/3	1/3
Set 3	1/4	3/4	1	1/4
Set 4	1	3	4	1
Sum	2,58	7,75	10,33	2,58

Mod Rupture	Set 1	Set 2	Set 3	Set 4	Score
Set 1	0,39	0,39	0,39	0,39	1,55
Set 2	0,13	0,13	0,13	0,13	0,52
Set 3	0,10	0,10	0,10	0,10	0,39
Set 4	0,39	0,39	0,39	0,39	1,55

Sustainability	Set 1	Set 2	Set 3	Set 4

Sustainability	Set 1	Set 2	Set 3	Set 4	Score

Set 1	1	1/2	1/4	1/5
Set 2	2	1	1/2	2/5
Set 3	4	2	1	4/5
Set 4	5	2 1/2	1 1/4	1
Sum	12,00	6,00	3,00	2,40

Set 1	0,08	0,08	0,08	0,08	0,33
Set 2	0,17	0,17	0,17	0,17	0,67
Set 3	0,33	0,33	0,33	0,33	1,33
Set 4	0,42	0,42	0,42	0,42	1,67

Labor/T	Set 1	Set 2	Set 3	Set 4
Set 1	1	4	2	2
Set 2	1/4	1	1/2	1/2
Set 3	1/2	2	1	1
Set 4	1/2	2	1	1
Sum	2,25	9,00	4,50	4,50

Labor/T	Set 1	Set 2	Set 3	Set 4	Score
Set 1	0,44	0,44	0,44	0,44	1,78
Set 2	0,11	0,11	0,11	0,11	0,44
Set 3	0,22	0,22	0,22	0,22	0,89
Set 4	0,22	0,22	0,22	0,22	0,89

Table 20 Aggregated score matrix without weight

Goal	Compressive Strength	Specific Strength	Workability	Modulus of rupture	Sustainability	Labor/Time
Set 1	2	2	5/6	1 5/9	1/3	1 7/9
Set 2	2/5	2/5	2/5	1/2	2/3	4/9
Set 3	1/3	1/3	2/7	2/5	1 1/3	8/9
Set 4	1 1/3	1 1/3	2 1/2	1 5/9	1 2/3	8/9

Step 4: Consistency check

After the weights for each criterion had been determined, the consistency of each weight value is checked in order to confirm whether the choices made are rational (Saaty, 1987). The individual weight value of each matrix position in table 18 is multiplied with the normalized weight of table 18 to form the matrix table 21. The sum of each row is then divided by the Normalized Weight (w) in table 18 for the consistency check.

Table 21 Consistency check

	Compressive Strength	Specific Strength	Workability	Modulus of rupture	Sustainability	Labor/Time	Sum i	Normalized weight (w)	Sum i/w
Compressive Strength	0,04	0,07	0,02	0,04	0,02	0,01	0,19	0,19	1,00
Specific Strength	0,07	0,15	0,04	0,07	0,04	0,02	0,38	0,38	1,00
Workability	0,02	0,04	0,01	0,02	0,01	0,00	0,10	0,10	1,00
Modulus of rupture	0,04	0,07	0,02	0,04	0,02	0,01	0,19	0,19	1,00
Sustainability	0,02	0,04	0,01	0,02	0,01	0,00	0,10	0,10	1,00
Labor/Time	0,01	0,02	0,00	0,01	0,00	0,00	0,05	0,05	1,00

With the consistency check performed as follows:

$$\lambda max = \frac{\Sigma(\frac{Sum\ i}{w})}{n} = \frac{6}{6} = 1$$

Consistency index: $CI = \frac{\lambda max - n}{n-1} = \frac{1-6}{6-1} = -1$

Consistency rate: $CR = \frac{CI}{RI} = \frac{-1}{1,24} = -0,81 < 0,1$ => Check ok.

Step 5: Calculating the final score

The table below show the results of the MCA analysis comparing the variant mixture sets 1-4 and their final score based on the chosen criteria.

Table 22 MCA Results

Set	Compressive Strength	Specific Strength	Workability	Modulus of rupture	Sustainability	Labor / Time	Score
1	0,37	0,75	0,08	0,29	0,03	0,08	1,61
2	0,07	0,15	0,04	0,10	0,06	0,02	0,45
3	0,06	0,12	0,03	0,07	0,13	0,04	0,46
4	0,25	0,50	0,24	0,29	0,16	0,04	1,48

From the table, the alternative with the highest score was the control baseline set 1, which had a score of 1,61. This is due to the set having the highest compressive, specific strength and modulus of rupture. The 2nd proposed variant Set 4 had second highest score at 1,48, 0,13 points lower than the baseline control set 1, and approximately 3 times as high as the control pretreatment method set 2 and the alternative 1st proposed variant set 3. The score of the 1st proposed variant set 3 is 0,01 point higher than of the control pretreatment method of set 2 (0,46 vs 0,45).

5. Conclusions and Recommendations

5.1. Summary of main findings

The result showed that different different pretreatment methods for Miscanthus x giganteus short fibre influenced the workability, density, compressive strength and modulus of rupture of the geopolymer concrete, as well as whether additional water is used to saturate the natural fibre before adding to the concrete mixture. By substituting 10% of the geopolymer concrete mixture with pretreated, unsaturated fibre resulted a 50% reduction in the workability of the geopolymer concrete mixture. Furthermore, it was found that different Sodium Alginate concentrations also played a key role in determining the workability and strength of the geopolymer concrete mixture: 10wt% Sodium Alginate pretreatment solution with 90,79 kg/m^3 extra water for saturating the fibre caused the concrete mixture to lose all workability, while 5wt% Sodium Alginate pretreatment solution with 90,79 kg/m^3 extra water resulted in an increase of 187% in workability compared to the baseline geopolymer concrete mixture.

From the results of this experiment, it can be observed that baseline geopolymer concrete performed the best in compressive and flexural strength, and second best in workability. Any substitution of aggregate with natural fibre resulted in a decrease in compressive and flexural strength of the geopolymer concrete no matter the pretreatment method, and most pretreatment methods resulted in a decrease of workability of the geopolymer concrete. The best performing geopolymer concrete mixture is the mixture using

5wt% Sodium Alginate pretreatment for natural fibre, with a mean Compression Strength of 28,2 MPa, mean Specific Compression Strength of 13,6 kN*m/kg, Modulus of Rupture of 2936,2 MPa at 28 days, and workability of 28 mm slump. The worst performing geopolymer concrete mixture is the mixture using 10wt% Sodium Alginate pretreatment for natural fibre, with a mean Compression Strength of 14,7 MPa, a mean Specific Compression Strength of 7,1 kN*m/kg, Modulus of Rupture of 1430,9 MPa at 28 days, and workability of 0 mm slump. The baseline geopolymer concrete mixture also outperformed its design value, with a mean Compression Strength of 45,5 MPa, 50% higher than its design value of 30 MPa. However, these results are preliminary, and cannot be said to be representative of the actual performance of these pretreatment methods due to the limited number of specimens tested and the very high variance in the test result data.

5.2. Conclusion

From the results of this preliminary study, it can be concluded that Sodium Alginate is a highly promising alternative pretreatment method for Miscanthus fibre worth further research into to determine its viability. While no pretreatment method resulted in geopolymer concrete mixture that meets the required value of the client, the 5wt% Sodium Alginate pretreatment method outperformed the baseline conventional 5wt% NaOH pretreatment method in compressive strength, specific compressive strength, modulus of rupture and workability. When Labor/time and Sustainability factor is also taken into account, both pretreatment methods using Sodium Alginate solution outperformed the baseline NaOH treatment, and the 5wt% Sodium Alginate pretreatment mixture is the best performing mixture of all 4 mixtures in this research. The 5wt% Sodium Alginate pretreatment method also reached 94% of the required 30 MPa compression strength demanded by the client. Furthermore, the results in this experiments are very promising compared to Miscanthus fibre substitution for Ordinary Portland cement concrete: Ezechiels (Ezechiels, 2017) found that Portland cement concrete with 10% by volume of aggregate substituted with saturated Miscanthus fibre at 28 days achieved a mean Compressive strength of 29,8 MPa – only 5,87% higher than the mean Compressive strength of 28,2 MPa of the geopolymer concrete mixture with 10% by volume overall substituted with Miscanthus fibre pretreated using 5wt% Sodium Alginate solution.

The results confirmed that dry Miscanthus fibre have a detrimental effect on both the workability and compressive strength of the geopolymer concrete mixture by absorbing water from the concrete mixture, despite water not being part of the polymerization process and theoretically only having a role in ensuring workability of the geopolymer concrete. The results showed that the saturated fibre pretreatment method where the fibre is not oven dried after pretreatment is a viable way of introducing additional water to saturate the natural fibre without resulting in bleeding of the concrete mixture. However, it is also observed that the Sodium Alginate concentration of the pretreatment solution also play a key role in determining the workability of the concrete mixture because Sodium Alginate acts as a coagulant agent; with 5wt% Sodium Alginate resulting in an acceptable workability, while 10wt% Sodium Alginate resulted in a mixture with almost no workability despite the additional water for fibre saturation.

Furthermore, this research has not been able to determine the long-term durability of Miscanthus x giganteus fibre in the highly alkaline environment of geopolymer concrete. Further research is necessary to determine this durability before it is possible to determine the viability of geopolymer concrete with natural fibre substitution.

Overall, in can be concluded that the 5wt% Sodium Alginate pretreatment method is the best pretreatment method of the pretreatment methods in this experiment, and Sodium Alginate pretreatment is a highly promising alternative natural fibre pretreatment method for geopolymer concrete worth further investigation.

5.3. Recommendations

For research:

3. Sodium Alginate is a viable pretreatment method for Miscanthus fibre.
4. Pursue further research into using Sodium Alginate to pretreat Miscanthus fibre, specifically repeat experiment for mixture set 4 Geopolymer with Miscanthus fibre pretreated using 5wt% Sodium Alginate solution to validate the preliminary results from this research.
5. Investigate into the ideal additional water content to saturate the natural fibre while still maintaining an acceptable fly ash/water ratio.
6. Repeat experiments on saturated fibre NaOH pretreatment method using additional water to saturate fibre in order to eliminate the effects of different water content to establish a better comparison between the effectiveness NaOH and Sodium Alginate pretreatment methods.
7. Repeat experiments on baseline geopolymer without natural fibre using the original water content by S. V. Pattankar instead of the reduced water content mixture for the baseline geopolymer to ensure workability and accuracy.
8. For three-point bending test, use thicker d=20mm and t=5mm in the future, or adjust the tile size so future research is Eurocode-compliant.
9. Repeat experiments on substituting geopolymer concrete by volume of aggregate, not overall volume so that it is possible to directly compare the effectiveness of aggregate substitution and for easier comparisons with existing research.
10. Investigate into the addition of steel slag for ambient temperature curing geopolymer concrete. Sofi found that with the addition of steel slag in the mixture, GPC can cure at room temperature for 28 days and reach a strength range of 47-56,5 MPa. (M. Sofi, 2007)
11. Further investigate into modifying the Sodium Alginate percentage in pretreatment solutions, additives and coagulants to increase pretreatment effectiveness, etc.
12. Investigate more deeply into the interaction between Sodium Alginate and the fly-ash based geopolymer.
13. Use finer, smaller aggregates for the mixture for tile moulds. As of right now the aggregate cause the most difficulty when working with tile moulds.
14. Ensure correct ambient curing time in the future for all tiles and cubes.
15. Further research into the viability of fully substituting all aggregates in a geopolymer concrete to create Lightweight Biobased Aggregate Geopolymer Concrete for non-load-bearing structures.

Practical recommendations:

1. Increase oven capacity to prevent delays with oven curing.
2. Get better tile moulds, since current ones break after one use causing waste and has difficulty demoulding without breaking the specimens contained that have not been oven cured.
3. Workability is very important for ensuring the strength of geopolymer concrete tiles.
4. Use the entire water content to dissolve NaOH pellets, instead of splitting the water 50/50 between the NaOH and Na_2SiO_3.

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