Fly Ash Based Geopolymer Concrete

Chemistry

Class: Science and Technology

To what extent do different sodium silicate (Na_2SiO_3) to sodium hydroxide (NaOH) ratios in alkaline solutions influence the durability of fly ash based geopolymer concrete measured through a sorptivity test?

Contents:

1. Introduction

As countries are becoming more developed, demand for resources in the construction industry is increasing. These resources include Ordinary Portland Cement (OPC), being one of the most used man-made binder in the preparation of concrete, and a common building material[20].

OPC production is a highly energy-intensive process requiring large amounts of natural cementitious materials containing calcium, silicon, aluminium and iron[18]. Moreover, during its manufacturing, the cementitious materials are placed in a rotary kiln heated at elevated temperatures of around 1510°C for a chemical process known as calcination to occur[18]. Significant contributions to the global CO_2 emissions occur during these steps, from both its calcination, and the heating of the kiln. Approximately 1 kg of cement produces a total of 0.83 kg of CO_2, contributing to 8% of global CO_2 emissions[20][3].

As demand for cement increases, it is crucial for research in new binders and materials as an alternative to OPC, to be carried out. Promising new materials include geopolymer cement. This could reduce the CO_2 emission caused by cement industries from 80 to 90%[5].

1.1. Aim

The aim of this investigation is to compare the water absorption of alkali activated fly ash concrete, as the ratio between sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) used as an activator is changed. This is done in order to determine the relationship between the activator's solution ratio and the sorptivity of concrete samples, linked to its durability.

2. Background Research

1.2. Geopolymer concrete

Geopolymers are an innovative alternative to traditional OPC used to make geopolymer concrete[20]. They are characterised by their use of lower amounts of energy and raw materials whilst having durable chemical and physical properties, making them an important new technology[20]. Geopolymers are amorphous aluminosilicate based cementitious materials synthesised through a polycondensation reaction known as geopolymerisation[21].

They are commonly made by activating source materials containing silica and alumina using *alkali solutions*[20].

1.3. Source materials

Source materials, or geopolymeric precursors, used in the production of geopolymer concrete contain aluminosilicates (minerals composed of aluminium, silicon, oxygen, and countercation) and can be derived from by-products of agricultural and industrial processes [2][20]. These include power generation, iron and steel manufacturing and mining facilities [14]. Materials such as kaolinite, clays, fly ash and silica fumes can therefore be utilised due to their high composition of aluminosilicates necessary for a geopolymerisation reaction to occur[20]. The use of these materials limits the potential problems linked with their disposal[14]. Because of variations in structure and composition, individual materials have a significant effect on the geopolymer concrete properties[15].

In this investigation, fly ash was used as a precursor material because of its extensive use in geopolymer concrete, showing better physical and chemical properties compared to OPC[20]. A precursor is a substance that, following a reaction, becomes an intrinsic part of a chemical product[20]. Concretes made with fly ash are also associated with higher life expectancy and an increase in durability[15]. The spherical shape of the fly ash molecules also reduces its permeability[15].

1.4. Alkali solutions

For a geopolymerisation reaction to occur, an alkali activator solution must be present to activate the source material[21]. Alkali activators have a number of important roles during the geopolymerisation process, affecting the geopolymers yielded. A mixture of aqueous potassium hydroxide (KOH) or sodium hydroxide (NaOH), and sodium silicate (Na_2SiO_3) or potassium silicate (K_2SiO_3) is most common[15]. Both the type and concentration of the solutions play an important role in the formation of geopolymers[15][20].

Potassium hydroxide or sodium hydroxide react with the source material, extracting silica and alumina through dissolution, whilst roughening the material's particles[15]. In this
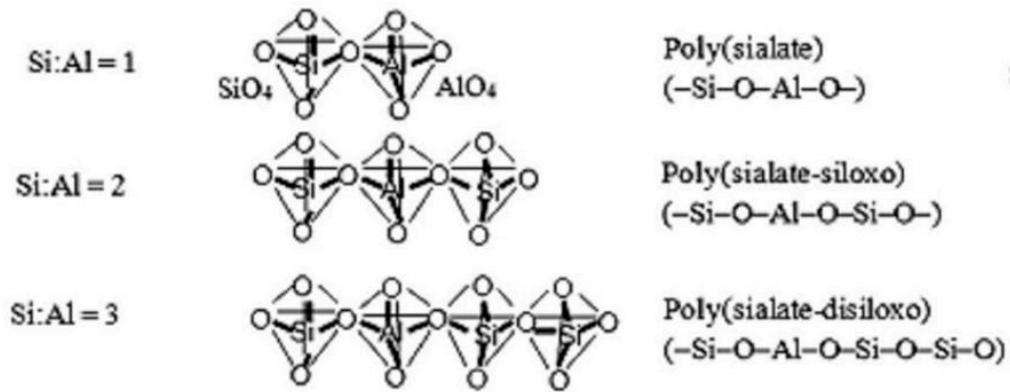
investigation, a 10 mol dm^{-3} sodium hydroxide solution was used due to its stronger dissolution property compared to potassium hydroxide[15].

Secondly, sodium silicate was chosen. When large amounts of silicon are already present in the solution from the sodium silicate, geopolymer processes occur rapidly, as silicon atoms are readily available for polymerisation to occur[20]. This leads to an enhancement in the reaction between the source materials and the activator[15].

1.5. Geopolymerisation reaction

Geopolymerisation occurs when inorganic aluminosilicate-based materials react with alkaline solutions[21]. This leads to a polycondensation reaction of geopolymeric precursor or source material and alkali polysilicates [21].

During geopolymerisation, three main processes can be observed:

1. Dissolution of aluminosilicate materials into silicon and aluminium atoms
2. Transportation/orientation of the dissolved species into monomers
3. Polymerisation of monomers, forming polymeric structures.

Figure 1: *A schematic formation of geopolymer materials* [6].

As seen in figure 1, the end result is a three-dimensional polymeric chain of Si-O-Al-O containing cross-linked SiO$_4$ and AlO$_4^-$ tetrahedral species linked with covalent bonds called polysialate [21] [6][16]. This can be characterised by the formula:

$$M_n \left[(SiO_2)_z - AlO_2-\right]_n$$

Where M is the element of the alkali cation (calcium, potassium, sodium), n is the degree of polycondensation, and z is 1, 2, 3 or higher. The alkali cations are required to balance the negative charges from the Al^{3+} in *IV-fold coordination structure* [6][16].

Figure 2: *The structure and the nomenclature of three basic forms of sialates*[6].

As the Si to Al ratio values change, a variety of polysialate structures are produced (figure 2), forming different geopolymer structures. As a result, the Si to Al ratio present in the polysialate can be used to determine the application of geopolymers[15]. Generally, a low Si-Al ratio of 1 to 3 leads to a rigid three-dimensional structure, used in bricks, concretes, foundry equipment, and heat resistant components[15]. Other applications involve automobile, aerospace and civil engineering[16].

1.6. Activation

For the dissolution mechanism (stage 1 of the reaction process) to occur an alkali solution must be present, acting as an activator to form the geopolymer precursor (stage 2 of the reaction process)[15]. The surface of the raw fly ash materials is attacked by the activator, dissolving Si and Al until the fly ash particles are completely or almost consumed[15]. The highly alkaline solution then yields amorphous structures consisting of Si-O-Al and Si-O-Si structures. Further Al atoms are penetrated into the Si-O-Si structures during their breakdown, finally forming geopolymer precursors[13]. During this process, the free oxygen atoms originating from water in the solutions help to link the Si and Al atoms together.

1.7. Sorptivity test

As water corrodes concrete, its durability is in parts determined by its resistance to water[9]. Other elements in reinforced concretes such as steel rods may also corrode if the concrete absorption is high, further lowering the durability[17].

The durability of concrete can be found through a water sorptivity test which is defined as a measure of the capacity of a medium to absorb or desorb liquid by capillarity (the transport of liquids in porous solids due to the surface tension of capillaries)[9]. Insufficient compaction of concrete during geopolymerisation as well as the viscosity, density and surface tension of the liquid used during testing may affect sorptivity values[17].

1.8. Hypothesis

Numerous studies have shown that the durability of geopolymer concrete is linked with the sodium silicate to sodium hydroxide ratio used in the activator[10][7][11].

According to Lavanya and Jegan (2015), augmenting the proportion of sodium silicate increases the silica content present in the mixture, enabling the formation of higher quantities of geopolymer precursors, whilst providing interparticle bonding. Additionally, higher amounts of silica can be used to occupy the void spaces between particles, resulting in lower water absorption[10]. Further amounts of Na can also be observed as the ratio increases, playing an important role in the formation of geopolymers as more can be used to act as charge balancing ions[7].

The ratios used in this investigation ranged from 0.400 to 2.500. As a result, samples prepared using a 2.500 ratio are expected to show lower sorptivity values linked to higher durability compared to samples containing a 0.400 ratio.

This leads to the formation of the hypothesis: *An increase in sodium silicate to sodium hydroxide ratio present in the alkali activator solution will increase the durability of concrete, lowering its sorptivity values.*

2. Methods

2.1. Independent Variable

The independent variable was the sodium silicate to sodium hydroxide solution ratio used as an alkali activator. The chosen ratios were: 0.400, 0.925, 1.450, 1.975 and 2.500. The range of ratio from 0.400 to 2.500 was chosen to mimic alkali activator solutions used to commonly make alkali activated concrete. Moreover, it was found by numerous academic papers that an alkali activator ratio ranging from 0.400 to 2.500 was optimum in yielding high durability and strengths of concrete[15].

The ratios were prepared by mixing different masses of the chemical compounds sodium silicate (Na_2SiO_3) and sodium hydroxide (10M NaOH). Both compounds contain different concentrations of H_2O, Na_2O and SiO_2, changing the chemical properties of the geopolymer reactions. Therefore, this will further indicate which chemical composition present in the alkali activator produces the highest durability of concrete.

2.2. Dependent variable

Due to problems linked with the use of absorption tests and permeability tests which measure the water intake of materials through direct pressure of liquids on concrete, a sorptivity test was used to assess the durability of the geopolymer concrete[17].

A mass method can be used to measure the sorptivity of concrete[9]. Water is penetrated in the concrete in a unidirectional pathway, where only 1 surface of a concrete sample is exposed to water[9]. This prevents evaporation and supplementary absorption from adjacent surfaces[9].

Sorptivity, S (mm/min$^{1/2}$) can be calculated by finding the gradient of a straight line defined by the formula[9]:

$$I = S * t^{\frac{1}{2}}$$

Where I is the mass in grams of absorbed water per unit cross section (mm^2), S is the sorptivity coefficient (mm/min$^{1/2}$), and t is the time the samples are exposed to water (min) [22].

3. Preliminary experiments

I decided to conduct preliminary experiments to make sure that the chosen methodology worked.

Originally, when designing the experiment, I intended to use a water/solid (w/s) ratio. According to numerous research papers, a w/s ratio of 0.29 enabled a consistent workability to be achieved[1][11]. For fly ash geopolymer concrete, the quantity of water in the mix is the sum of water contained in the sodium silicate and sodium hydroxide solution and added water, whilst the quantity of solid is the mass of fly ash and solid contained in the activator solution (Na_2O and SiO_2). Unfortunately, the chemical suppliers were unable to give a precise account of the constituents in the sodium silicate solutions. After extensive research, a sample solution of sodium silicate was sent to a private lab to conduct an ICP-AES/SFMS, AFS test. Having received the test results, I was unable to use the findings as it contained significant uncertainties (see appendix 1). Thereafter, I decided not to use a w/s ratio, but instead used a constant volume of added water. As a result, preliminary tests were conducted to evaluate the best added water volume to obtain consistent workability. 25 ml of added water was chosen to be used.

The size and type of aggregates used affect the strength and characteristics of concrete[21]. A variety of fine and coarse aggregate sizes were therefore tested. I chose to use a 0-4 mm fine aggregate size as it matched common industrial uses. Secondly, a 8-11 mm coarse aggregate size was used, as it would not significantly impact the samples total volume composition, significantly affecting sorptivity values.

Finally, curing methods were also investigated during preliminary trials. When concrete samples were cured at room temperature, some had not completely finished their hardening process, chipped away and could not be demoulded correctly. On the other hand, all samples which were heat cured in an oven hardened completely, so a 24-hour heat curing at 70°C was chosen.

Control variables

Surface area of cubes submerged in water:

- The rate of absorption of water per unit time by concrete samples is largely dependent on the surface area submerged in water. A large area could contain higher amounts of capillary pores and space for water to penetrate in compared to smaller surface area. As a result, more water would be absorbed in a shorter time period, influencing the sorptivity values. Therefore, all samples were moulded in 50x50x50 mm cubes. To continue, to decrease risks of water being absorbed from adjacent sides, a concrete paint was applied. This covered all 4 concrete sides at close proximity to the water's level, providing a more controlled absorption.

Concentration of sodium silicate and sodium hydroxide:

- Additionally, the concentration of the sodium silicate and sodium hydroxide solutions were kept constant. This ensured that both solutions contained identical amounts of solids (Na_2O, and SiO_2) before being mixed in different ratios.

Size and quantity of aggregates:

- The size and quantity of aggregates used affect the strength and characteristics of concretes [14]. Hence, the amount of fine and coarse aggregate was controlled by using 450 g and 900 g of aggregate respectively for each independent variable. Moreover, their size was kept constant, being 0-4 mm for the fine aggregate and 8-11 mm for the coarse aggregate. Moreover, different concrete grades can be made by varying aggregate types and ratios. A M-20 grade concrete with a (fly ash) : (fine aggregate) : (coarse aggregate) ratio of (1) : (1.5) : (3) was chosen due to its common application. This also ensured that each sample contained approximately the same constituent and a constant aggregate composition.

Curing conditions:

- Shortly after being moulded, all concrete samples were placed in an oven at 70°C for 24-hours. Heat curing time and temperatures increase the rate of geopolymerisation[1]. Additionally, the samples were tested 7 days after being moulded. This ensured that the time of development and extent of geopolymerisaton was kept constant.

4. Materials List

4.1. Materials and chemicals

	Quantity:
35% Sodium silicate solution	1000 ml
10M Sodium hydroxide solution	200 ml
Type F fly ash	1500 g
Fine aggregate (0-4 mm)	2250 g
Coarse aggregate (8-11 mm)	4500 g

4.2. Apparatus

	Quantity:
0.01 g Scale	1
250 ml beaker	5
200 ml Conical flask	5
Glass funnel	2
Plastic bottle	1
10 ml Pasteur Pipette	5
Magnetic stirrer	1
400 ml Erlenmeyer flask	5
Parafilm sealing film	1
Fume cupboard	1
10 litre bucket	2
50 ml graduate cylinder	5
10 ml Graduated pipette	5
50x50x50mm cube moulds	25

Spoon	2
Oven	1
Non-absorbent concrete paint	
Metal rod	2
Rectangular container	2
Cloth	2

5. Procedures

5.1. Alkali Activator solutions:

1. 80.00 g of solid NaOH crystals was weighed on the scale using a 250 ml beaker and transferred to a 200 ml conical flask using a glass funnel. The conical flask was filled up to the 200ml mark with distilled water using the plastic bottle and the pipette to produce a 10 mol dm^{-3} solution. It was stirred using a magnetic stirrer until the solids dissolved completely.

2. A 250 ml beaker was placed on the scale and a pipette was used to add the desired masses of the sodium silicate solution and the 10M sodium hydroxide solution to produce the required ratios. The solutions were added to the 400 ml Erlenmeyer flasks and orbitally shaken.

3. Step 2 was repeated 5 to produce each alkali activator ratio solutions.

4. The Erlenmeyer flasks were sealed using a parafilm sealing film and were left to sit for 24 hours.

5. All the steps were carried out in a fume cupboard.

5.2. Producing the concrete mixtures

1. 300.00g, 450.00g and 900.00g of type F fly ash, 4mm fine aggregate and 10mm coarse aggregate was weighed on the scale respectively. The solids were then placed in a 10-litre bucket and mixed thoroughly until a homogenous mixture was achieved.

2. Using a 50 ml graduated cylinder, 30 ml of water was measured by adding 25 ml of water to the graduated cylinder, and adding the remaining 5 ml using a graduated pipette.

3. The alkali activator and the 30 ml of distilled water were mixed in the 10-litre bucket together with the type F fly ash and aggregates using a mixing apparatus until a homogenous mixture was achieved.

4. Steps 1-3 were repeated 5 times for each alkali activator solution.

5.3. Producing concrete blocks

1. The concrete mixtures were transferred using a spoon to 5 cube moulds measuring 50x50x50mm made from wood (see appendix 2). After being filled, the moulds were vibrated for 1-2 minutes.

2. The moulds were oven dried for 24 hours at a temperature of 70°C, and left at room temperature until tested.

5.4. Measurements

1. All concrete blocks were sealed on 4 sides to achieve unidirectional flow from the bottom of the blocks. The sealant used was locally available non-absorbent concrete paint. Weights of the specimens after sealing were taken as initial weight.

2. The samples were placed on the metal rods in the rectangular containers and immersed at a depth of 2-5 mm in the water. After selected times (1, 2, 3, 4, 5, 9, 12, 16, 20, 25, 30, 45, 65, 75, 100 minutes), the samples were removed from the water. Excess water at the surface was removed using a damp cloth, and the cubes were weighed on a 0.01g accuracy scale.

3. Steps 2 was repeated for each 25 samples.

6. Data processing

6.1. Significance tests

A one-way ANOVA significance test was performed to assess whether the mean sorptivity values were significantly significant[8]. This tested whether the null hypothesis (H_0) which states that there is no difference among group means was accepted or rejected. If a statistically significant difference is seen from the overall mean, an alternate hypothesis (H_A) can be accepted [8].

6.2. Processing Raw Data

Raw data collected (see appendix 3) consisted of weight measurements for each sample per unit time. These measurements were transformed in order to obtain sorptivity values. The mass (m) of absorbed water per unit cross section, I, was calculated using the following formula:

$$I = \frac{m_t - m_{(t=0)}}{cross\ sectional\ area}$$

The average water absorption ($I_{average}$) of each sample per unit time was then calculated and plotted against the square root of time (min$^{1/2}$) (see figure 3). The result should be a straight line, who's gradient defines the sorptivity coefficient[12].
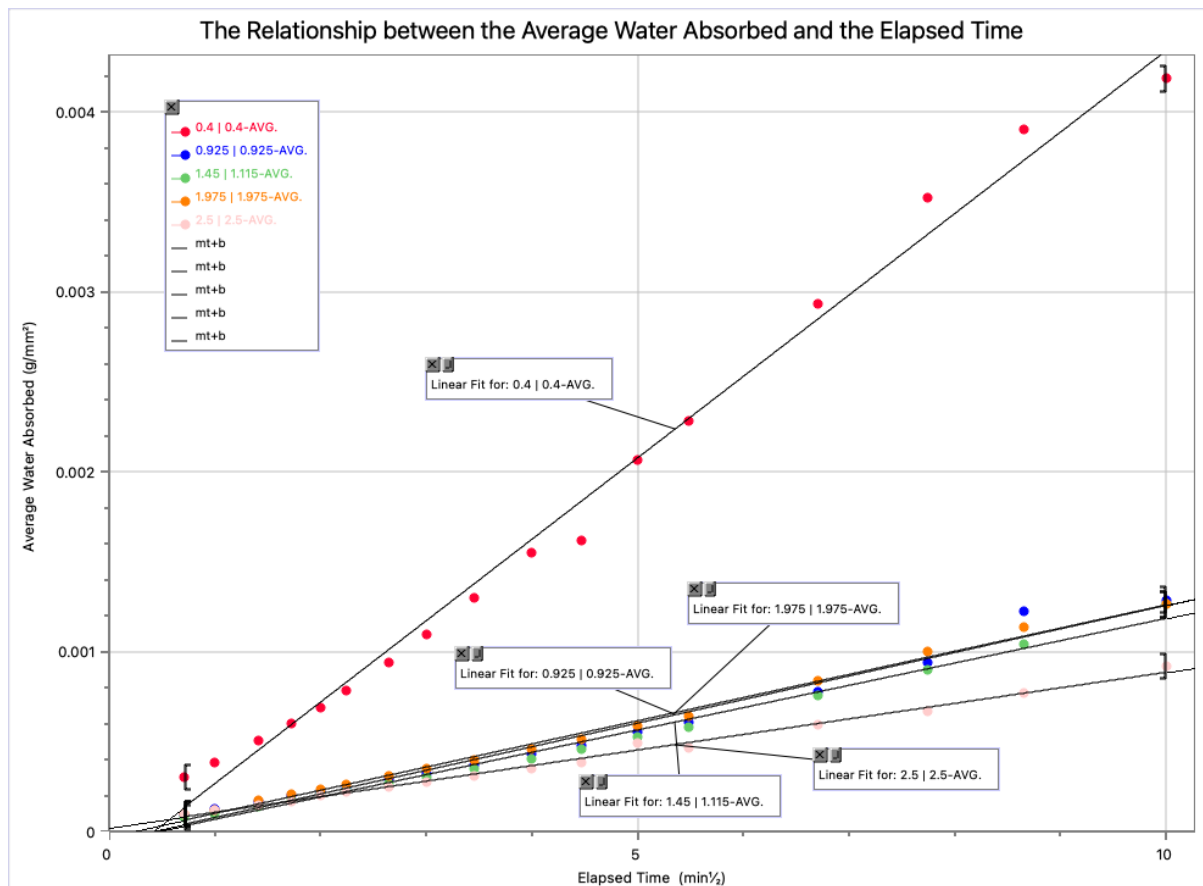
Figure 3: *Average absorption per unit area over time.*

6.3. Sorptivity values

The gradient of the lines on figure 3 was then calculated to obtain the average sorptivity

values for each ratio.

The Relationship between the Average Sorptivity and the Sodium Silicate to Sodium Hydroxide Ratio

Auto Fit for: Data Set | Sorptivity
S = A*x^B
A: 0.4391 +/- 0.04556
B: -1.190 +/- 0.2153
Correlation: 0.9641
RMSE: 0.04629 mm/√(min)

Sorptivity (mm/√(min))

Na₂SiO₃ : NaOH Ratio

Figure 4: *Graph showing the average sorptivity values as the sodium silicate to sodium*

hydroxide ratio increases.

Statistical significance:

SUMMARY

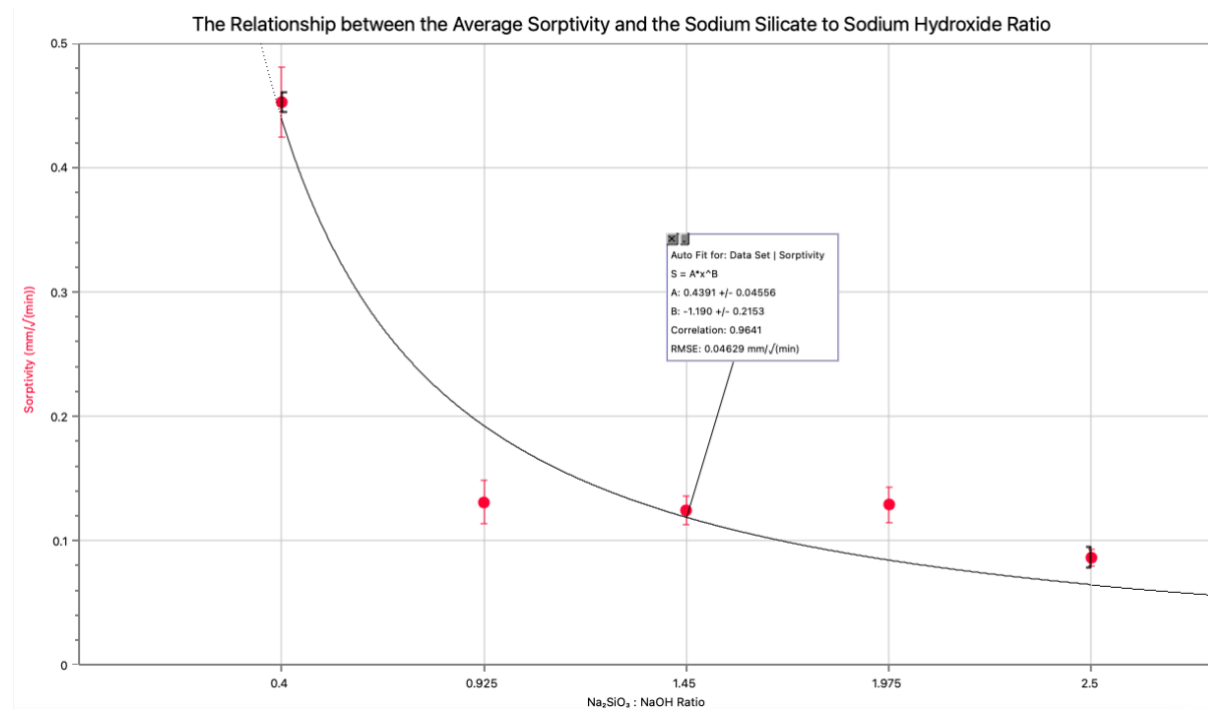| Groups (Na$_2$SiO$_3$:NaOH Ratio)	Count	Sum	Average	Variance
0.400	5	2.2633	0.45266	0.00081099
0.925	5	0.6553	0.13106	0.00030786
1.450	5	0.6205	0.1241	0.00012922
1.975	5	0.6439	0.12878	0.00020174
2.500	5	0.43242	0.086484	4.6524E-05

ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	0.45562783	4	0.11390696	380.620517	**1.45996E-18**	2.8660814
Within Groups	0.00598533	20	0.00029927			
Total	0.46161316	24				

A significance level of 0.05 was chosen, indicating a 5% risk of concluding that a difference exists between the means when there are no actual differences[8]. The P-value (p = 1.45996 x 10^{-18}) was less than the significance level, so H$_0$ is rejected, whilst H$_A$ is accepted.

7. Conclusions

According to figure 2, a negative relationship between sodium silicate to sodium hydroxide ratios and sorptivity can be observed. This can be proven by the strong negative correlation coefficient of -0.7696. This suggests that as the mixture's ratio increases, sorptivity values decrease, increasing durability. There is a large reduction in sorptivity as the ratio increases from 0.400 to 0.925. Smaller changes in sorptivity values can be seen from a ratio of 0.925 to a ratio of 2.500.

Mixtures with a sodium silicate to sodium hydroxide ratio of 0.400 had the highest sorptivity values, representing a high average rate of water absorption per unit time. The lowest sorptivity values were obtained from mixtures with a sodium silicate to sodium hydroxide ratio of 2.500, linking to higher durability. However, 1.450 and 1.975 ratios do not fit the trend as their sorptivity values are opposite of what they should have been.

8. Discussions

8.1. Credibility and Reliability

When comparing the results with other scientific journals, similar trends to the one observed in figure 4 were obtained as the sodium silicate to sodium hydroxide ratios increased. According to Law et al. (2009), high reductions in sorptivity values were observed as the ratio increased from 0.90 to 1.58, whilst a smaller reduction in sorptivity was recorded as the ratio increased to 2.60, implying the presence of a negative exponential trend. This indicates that my results match the general trends recorded by literature, furthering its credibility. Secondly, the ANOVA test has shown a statistical significance, implying that the results are not due to random change and that some of the group means are different, furthering the validity of the results.

From a sodium silicate to sodium hydroxide ratio of 0.400 to 0.925, a reduction in average sorptivity value can be explained due to an increase in sodium silicate in the alkaline solution. The presence of silicon atoms would therefore increase, which would favour the polymerisation process and higher mechanical strength due to the production of more stable structures[7]. A lower sorptivity is therefore seen as smaller interconnected pores would be created[7]. On the other hand, from a ratio of 0.925 to a ratio of 2.500, sorptivity values show little variation. A possible explanation for this trend could be that excessive alkali contents could be present in the mixture[7]. This retards the geopolymerisation reaction, decreasing its structural strength and durability[7].

Nevertheless, the experimental method is questioned, as uncertainties in the collected data are present, limiting the reliability of the conclusion.

Firstly, as shown on figure 4, high standard deviation relative to the differences in average sorptivity values result in overlaps in deviations. This is proven with concrete samples of ratio 0.925 to 1.975. This implies that average sorptivity values may vary significantly to one another, changing the observable trend. This may be caused due to changes in composition of each sample. More specifically, samples may contain more or less aggregate. Although each samples were mixed until a homogeneous consistency was achieved, the samples may

contain different combinations of fine and coarse aggregate. As aggregates cannot absorb water, their quantity and spatial location limit the potential amount of water which can be absorbed per unit area. This could have caused differences in recorded sorptivity values.

Additionally, fluctuations in sorptivity values which may have led to changes in average values could be caused due to oven temperature inconsistencies. During heat curing, samples were placed in a convection fan oven set at a constant temperature. Nonetheless, after monitoring of the oven temperature at different levels with an electronic thermometer, deviations from the specified temperatures were measured. According to Adam (2009), curing temperatures affect the rate of reaction and the extent of geopolymerisation the samples experience. Therefore, samples cured in areas prone to higher temperatures could have experienced faster chemical processes, increasing the sample's durability as less pores would be created[1]. This would further contradict the recorded sorptivity values, and could explain irregularities of the trend line.

Finally, limitations in the application of the results are present. Various grades of sodium silicates are determined by their solid SiO_2 to Na_2O weight ratio[4]. These ratios can vary from 2:1 to 3.75:1, being alkali or neutral[4]. Correspondingly, their chemical properties may vary, and different grades are used for different purposes[15]. Linking to its use in concrete, the concrete's strength and durability is significantly dependent on the ratio. Therefore, when designing geopolymer concrete, it is important to take into consideration the proportion of solids (Na_2O and SiO_2) already present in the solutions to adjust quantities such as water and fly ash. Without this information, incorrect characteristics and properties may be achieved depending on the concrete's use[15]. Although the same sodium silicate solution was used throughout this investigation, this would limit the real-life application of the conclusion obtained, as the sodium silicate solution used contains an unknown composition.

9. Evaluation

<u>9.1. Error analysis</u>

Source of error	Type	Significance	Improvement
Preconditioning for sorptivity test	Systematic	Significant	Carry sorptivity test in accordance to standard test methods
Vibration of samples after being moulded.	Systematic	Medium	Use a concrete vibrator for 10 seconds on each sample after being moulded for consistent treatment.
Oven temperature (during heat curing)	Random	Medium	Heat cure all samples placed at the same level in a convection fan oven to attain constant curing temperature
Wiping of samples	Systematic	Minor	Wipe samples using a damp cloth, changing it every 10 measurements to prevent over accumulation of water on cloth.

<u>9.2. Linking back to the Hypothesis:</u>

Overall, the results of this investigation indicate an influence of the sodium silicate to sodium hydroxide ratio on the sorptivity values of geopolymer concrete. These results can be linked to the concrete's durability. Although uncertainties in the methodology and limitations linked to the application of the result to real life situations are present, the hypothesis is accepted. When the sodium silicate to sodium hydroxide ratio increases, sorptivity values decrease, showing an overall negative trend between both variable. In addition to be negative, the trend is exponential.

9.3. Further exploration

One pertinent issue with the experimental data was that it only tested the durability of concrete after 7 days of being produced and moulded. Although heat curing was used to accelerate geopolymerisation, the durability of concrete may develop further over time. In order to accurately determine the sorptivity values, tests must be carried out over a longer time period at which complete geopolymerisation has been achieved.

Therefore, if I were to repeat this experiment, I would increase the range of testing days, by conducting sorptivity tests on the same samples on day 7, day 28 and day 32 to more accurately observe the development of the concrete samples, therefore determining a more reliable average sorptivity reading.

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11. Appendices

11.1. Appendix 1

ICP-AES/SFM, AFS test results for 35% sodium silicate solution.

Results for sample 2020-05795-001

Sample marking	35 % Na2SiO3
Sampled Date	13.aug.2020
Sample type	Other

Parameter	Results	Unit	PQL Lower	PQL Upper	Uncertainty Rel | Abs
Single element, ICP			*Method/standard:*	I-1-32	
Sodium, Na	100000	mg/l	0,2	200000	15% | ±0,2
Silicon, Si	200000	mg/l	0,015	10000	20% | ±0,05

Explanation: PQL = Practical Quantification limit, may differ due to pre-treatment of the sample. # = The analysis is performed by sub contractor
.
The uncertainty is expressed at 95% confidence level. If both a relative and an absolute uncertainty argument is stated, it is the argument that represents the highest uncertainty that applies.

Method reference

Parameter	Unit	PQL Lower	PQL Upper	Uncertainty Rel | Abs	Method	Standard
Single element, ICP					I-1-32	
Sodium, Na	mg/l	0,2	200000	15% | ±0,2		
Silicon, Si	mg/l	0,015	10000	20% | ±0,05		

Explanation: PQL = Practical Quantification limit, may differ due to pre-treatment of the sample. # = The analysis is performed by different laboratorium.
The uncertainty is expressed at 95% confidence level. If both a relative and a absolute uncertainty argument is stated, it is the argument that represents the highest uncertainty that applies.

11.2. Appendix 2

Wooden moulds made to create concrete blocks with dimensions of 50x50x50 mm.

11.3. Appendix 3

Raw data of mass of samples per unit time for each sodium silicate to sodium hydroxide ratio.

Time (±0.167 min)	0.400 ratio (Sodium silicate to sodium hydroxide)				
	Sample 1 (±0.01 g)	Sample 2 (±0.01 g)	Sample 3 (±0.01 g)	Sample 4 (±0.01 g)	Sample 5 (±0.01 g)
0	309.22	321.02	314.57	312.69	311.58
0.5	309.88	321.76	315.42	313.56	312.26
1	310.07	321.98	315.6	313.75	312.52
2	310.34	322.26	315.91	314.08	312.78
3	310.6	322.54	316.1	314.35	313.03
4	310.81	322.78	316.28	314.57	313.26
5	311.01	323.03	316.45	314.78	313.57
7	311.38	323.49	316.76	315.23	313.99
9	311.73	323.9	317.08	315.64	314.39
12	312.2	324.49	317.54	316.19	314.92
16	312.75	325.16	318.16	316.83	315.55
20	313.26	323.8	318.67	317.46	316.08
25	313.82	326.48	319.33	318.18	317.08
30	314.34	327.1	319.93	318.83	317.45
45	315.73	328.86	321.58	320.58	318.98
60	317.34	330.65	322.95	321.94	320.24
75	318.4	331.45	324.02	322.87	321.15
100	319.23	332.5	324.66	323.37	321.63

Time (±0.167 min)	0.925 ratio (Sodium silicate to sodium hydroxide)				
	Sample 1 (±0.01 g)	Sample 2 (±0.01 g)	Sample 3 (±0.01 g)	Sample 4 (±0.01 g)	Sample 5 (±0.01 g)
0	317.06	311.5	326.32	318.24	321.83
0.5	317.33	311.71	326.52	318.52	322.05
1	317.4	311.85	326.57	318.6	322.11
2	317.59	311.91	326.65	318.72	322.2
3	317.63	311.98	326.73	318.8	322.3
4	317.7	312.02	326.78	318.87	322.33
5	317.76	312.08	326.85	318.92	322.39
7	317.87	312.18	326.93	319.04	322.51
9	317.93	312.3	327.01	319.13	322.61
12	318.05	312.42	327.11	319.25	322.72
16	318.18	312.58	327.26	319.46	322.89
20	318.31	312.74	327.36	319.56	322.95
25	318.51	312.94	327.51	319.73	323.14
30	318.64	313.07	327.66	319.89	323.31
45	318.99	313.53	328.05	320.29	323.78
60	319.39	313.93	328.49	320.72	324.16
75	319.71	316.28	328.83	321	324.45
100	320.23	314.86	329.37	321.59	325

Time (±0.167 min)	1.450 ratio (Sodium silicate to sodium hydroxide)				
	Sample 1 (±0.01 g)	Sample 2 (±0.01 g)	Sample 3 (±0.01 g)	Sample 4 (±0.01 g)	Sample 5 (±0.01 g)
0	324.05	319.58	324.53	316.98	319.12
0.5	324.27	319.74	324.74	317.17	319.32
1	324.32	319.82	324.8	317.19	319.36
2	324.43	319.9	324.87	317.31	319.51
3	324.51	319.99	324.95	317.37	319.6
4	324.58	320.04	325.2	317.42	319.68
5	324.64	320.1	325.26	317.45	319.73
7	324.74	320.18	325.31	317.5	319.86
9	324.83	320.27	325.4	317.61	319.96
12	324.96	320.36	325.5	317.71	320.09
16	325.11	320.53	325.64	317.82	320.23
20	325.25	320.68	325.77	317.91	320.38
25	325.42	320.85	325.93	318.06	320.56
30	325.56	321	326.05	318.2	320.73
45	325.99	321.44	326.45	318.59	321.21
60	326.31	321.79	326.79	318.94	321.67
75	326.62	322.16	327.12	319.29	322.06
100	327.15	322.76	327.69	319.78	322.68

	1.975 ratio (Sodium silicate to sodium hydroxide)				
Time (±0.167 min)	Sample 1 (±0.01 g)	Sample 2 (±0.01 g)	Sample 3 (±0.01 g)	Sample 4 (±0.01 g)	Sample 5 (±0.01 g)
0	319.61	317.89	322.44	321.23	318.95
0.5	319.86	318.12	322.71	321.47	319.2
1	319.9	318.2	322.71	321.55	319.25
2	320.06	318.31	322.88	321.67	319.41
3	320.11	318.4	322.95	321.75	319.48
4	320.16	318.53	323.04	321.82	319.52
5	320.26	318.57	323.09	321.88	319.57
7	320.38	318.69	323.28	321.99	319.68
9	320.48	318.8	323.34	322.07	319.77
12	320.62	318.94	323.48	322.19	319.86
16	320.8	319.1	323.62	322.34	320.01
20	320.95	319.25	323.76	322.46	320.13
25	321.15	319.41	323.91	322.62	320.35
30	321.35	319.58	324.04	322.76	320.44
45	321.91	320.03	324.5	323.24	320.88
60	322.36	320.45	324.87	323.61	321.32
75	322.79	320.75	325.18	323.88	321.69
100	323.47	320.85	325.28	324.05	322.26

	2.500 ratio (Sodium silicate to sodium hydroxide)				
Time (±0.167 min)	Sample 1 (±0.01 g)	Sample 2 (±0.01 g)	Sample 3 (±0.01 g)	Sample 4 (±0.01 g)	Sample 5 (±0.01 g)
0	321.22	312.76	320.06	318.06	319.32
0.5	321.43	312.99	320.32	318.33	319.59
1	321.49	313.01	320.39	318.37	319.65
2	321.56	313.11	320.47	318.45	319.7
3	321.64	313.15	320.5	318.46	319.76
4	321.7	313.22	320.59	318.57	319.83
5	321.74	313.26	320.67	318.61	319.87
7	321.8	313.34	320.75	318.69	319.96
9	321.89	313.4	320.84	318.75	320.02
12	321.98	313.48	320.86	318.83	320.12
16	322.07	313.55	321.05	318.95	320.2
20	322.17	313.63	321.14	319.03	320.28
25	322.67	314.12	321.26	319.16	320.39
30	322.35	313.8	321.38	319.24	320.48
45	322.65	314.1	321.72	319.58	320.78
60	322.89	314.29	321.92	319.74	320.95
75	323.17	314.54	322.21	319.96	321.19
100	323.53	314.85	322.64	320.37	321.55