# Development of sustainable construction material from fly ash class C

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## Abstract

Purpose – Environmental issues caused by the production of Portland cement have led to it being replaced by waste materials such as fly ash, which is more economical and safer for the environment. Also, fly ash is a material with sustainable properties. Therefore, this paper aims to focus on the development of sustainable construction materials using 100% high-calcium fly ash and potassium hydroxide (KOH)-based alkaline solution and study the engineering properties of the resulting fly ash-based geopolymer concrete. Laboratory tests were conducted to determine the mechanical properties of the geopolymer concrete such as compressive strength, flexural strength, curing time and slump. In phase I of the study, carbon nanotubes (CNTs) were added to determine their effect on the strength of the geopolymer mortar. The results derived from the experiments indicate that mortar and concrete made with 100% fly ash C require an alkaline solution to produce similar (comparable) strength characteristics as Portland cement concrete. However, it was determined that increasing the amount of KOH generates a considerable amount of heat causing the concrete to cure too quickly; therefore, it is notable to forming a proper bond was unable to form a stronger bond. This study also determined that the addition of CNTs to the mix makes the geopolymer concrete tougher than the traditional concrete without CNT.

**Design/methodology/approach** – Tests were conducted to determine properties of the geopolymer concrete such as compressive strength, flexural strength, curing time and slump. In Phase I of the study, CNTs were studied to determine their effect on the strength of the geopolymer mortar.

Findings – The results derived from the experiments indicate that mortar and concrete made with 100% fly ash C require an alkaline solution to produce the same strength characteristics as Portland cement concrete. However, it was determined that increasing the amount of KOH generates too much heat causing the concrete to cure too quickly; therefore, it is notable to forming a proper bond. This study also determined that the addition of CNTs to the mix makes the concrete tougher than concrete without CNT.

Originality/value – This study was conducted at the construction engineering and management concrete laboratory at North Dakota State University in Fargo, North Dakota. All the experiments were conducted and analyzed by the authors.

Keywords Fly ash, Carbon nanotubes, Geopolymer, Compressive and flexural strength, Slump

Paper type Research paper

## Introduction

The development of green concrete mix designs is an important concern in the construction industry. Various methods have been used throughout the world to achieve concrete mixes that are able to sustain any load and address the challenges associated with concrete such as workability, bleeding and segregation. In addition to dealing with these challenges, another major concern of the concrete industry is the use of Portland cement, because it is considered to significantly contribute to global warming. Harmful gasses, such as carbon dioxide (CO<sub>2</sub>),



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nitrogen dioxide, sulfur dioxide and specks of dust are discharged into the atmosphere during the production of Portland cement because of the calcination of limestone and the combustion of fossil fuel [\(Hardjito, 2005](#page-24-0)). The  $CO<sub>2</sub>$  produced in this process is approximately one ton for every ton of Portland cement produced [\(Hardjito, 2005\)](#page-24-0). Portland cement production also requires a considerable amount of energy, following steel and aluminum production [\(Hardjito, 2005](#page-24-0)). Consequently, it is important to reduce the cement content in concrete while maintaining the indispensable characteristics of concrete, such as strength, workability and durability.

In the latter half of the twentieth century, geopolymer technology was developed to reduce the use of Portland cement in concrete [\(Davidovits, 1994\)](#page-23-0). As part of the sustainability movement in the concrete industry, the new technology has led researchers to discover green concrete, which may be used as a substitute for traditional concrete. Among the different types of geopolymer materials, fly ash is the most widely used material, and it is a viable replacement for Portland cement. Fly ash is considered to be the world's fifthlargest raw material resource [\(Ahmaruzzaman, 2009](#page-23-1)). Fly ash is an industrial byproduct produced in coal-fueled power plants and mainly categorized into two types, namely, classes C and F. Approximately, 500 million tons of fly ash is produced per year throughout the world ([Ahmaruzzaman, 2009\)](#page-23-1). The current amounts of fly ash used are limited to 10-30 per cent of its total production ([Wang](#page-24-1) *et al.*, 2008). Fly ash particles are highly contaminated because of their enrichment in potentially toxic trace elements, which condense from the flue gas; therefore, significant amounts of fly ash have to be disposed of in retention ponds. However, the disposal of fly ash is becoming more of a problem for fired power plants [\(Ahmaruzzaman, 2009](#page-23-1)).

On the other hand, the negative impact associated with the disposal of fly ash has generated some beneficial uses, mainly in concrete applications. The recycling of this abundant material is also minimizing the environmental impact associated with the production of Portland cement. Geopolymer technology could reduce up to 80 per cent of  $CO<sub>2</sub>$  emissions caused by the cement industry (Raijiwala *et al.*, 2011). Nevertheless, to understand fly ash's capacity for improving the performance of concrete and address the issue concerning its disposal, various research projects have been conducted to study replacing Portland cement with fly ash.

Prominent characteristics, such as high strength, have been discovered in the use of fly ash in concrete, which improves the performance of a wide range of concrete applications such as the construction of roads, embankments and structural fill ([Ahmaruzzaman, 2009\)](#page-23-1). This is because the properties of fly ash are similar to Portland cement. The pozzolanic properties of fly ash, including its lime binding capacity, make it useful for manufacturing cement, concrete building materials and concrete-admixed products [\(Ahmaruzzaman, 2009\)](#page-23-1). When used as a partial replacement for Portland cement, in the presence of water and ambient temperatures, fly ash reacts with calcium hydroxide during the hydration process of Portland cement to form the calcium silicate hydrate (C-S-H) gel ([Hardjito](#page-24-2) et al., 2005). In the process of making concrete more resistant to excessive loads, reinforcing materials such as steel have been incorporated into concrete.

In recent years, nanotechnology products are being investigated to use them to replace steel in concrete. Fibers, in the form of nanomaterials, such as carbon nanotubes (CNTs) have been used or investigated as a replacement for reinforcing steel. These nanomaterials have been found to have very high mechanical properties with high strength (i.e. 100 times more than steel), but yet are six times lighter ([Chaipanich](#page-23-2) *et al.*, 2009).

Furthermore, CNTs exhibit unique thermal, chemical and electrical properties, which increase the compressive strength of structural concrete (Shah *et al.*[, 2010\)](#page-24-3). Due to their

unique properties and characteristics, CNTs have been used in several concrete designs to reinforce the matrix [\(Chaipanich](#page-23-2) et al., 2009). The incorporation of such nanomaterials in concrete helps control the matrix cracks at the nanoscale level and possibly create crack-free concrete materials (Shah *et al.*[, 2010](#page-24-3)). The study of nanomaterials in concrete represents a relatively new research area [\(Chaipanich](#page-23-2) et al., 2009). Additional research and many investigations are required to learn more about the possibilities for the use of nanomaterials as key constituents in enhancing the strength of concrete [\(Chaipanich](#page-23-2) et al., 2009).

This study was conducted to investigate the possibility of using fly ash to replace Portland cement in construction applications. This study has the following objectives:

- to design concrete mixes to replace Portland cement (100 per cent) with class C fly ash based cementitious material with adequate durability, compressive strength and flexural strength;
- to test the resulting concrete product characteristics such as slump, setting time, compressive strength and flexural strength properties using the standard ASTM concrete tests; and
- to analyze the ability/possibility of CNTs to increase the durability of concrete and to eliminate cracking.

Class C fly ash was used as a 100 per cent replacement for Portland cement to develop geopolymer concrete. In addition to the class C fly ash, CNTs were also added to the mixture during Phase I of the investigation. The technology, and the equipment, currently used to produce Portland cement concrete, were used throughout the experiments. The concrete properties studied included compressive and flexural strength along with early features/ properties of fresh concrete, such as slump and setting time.

#### Literature review

The construction industry has used fly ash as a key binder in developing concrete-based construction materials for many decades, as a partial replacement for Portland cement. Concrete is the most commonly used construction material in the world. It is required globally for the construction of buildings, bridges, roads, runways, sidewalks, dams and others [\(Rubenstein, 2012\)](#page-24-4). Moreover, its availability has made it one of the most popular critical binder in developing cementitious construction materials ([Rubenstein, 2012\)](#page-24-4). The use of concrete has aided in the development of civilization, enhanced economic progress and improved the quality of life of humanity [\(Rubenstein, 2012\)](#page-24-4). However, some of its disadvantages, especially its environmental impacts, are challenging to mitigate. The major challenge is dealing with the  $CO<sub>2</sub>$  emissions associated with cement clinker production and its impact on climate.

According to [Rubenstein \(2012\),](#page-24-4) the cement production industry is responsible for 5 per cent of global  $CO<sub>2</sub>$  emissions. Concrete is the second most prolific substance on earth after water ([Rubenstein, 2012\)](#page-24-4). On average, three tons of concrete are created per capita each year. Cement production is growing by 2.5 per cent annually. According to [Rubenstein \(2012\)](#page-24-4), it was 2.55 billion tons in 2006, and its production is expected to rise to 3.7-4.4 billion tons by 2050. Also, cement production is highly energy-intensive because of the extreme heat required in the process. Producing a ton of cement requires 4.7 million BTU of energy, which is equivalent to about 400 pounds of coal, and it generates approximately a ton of  $CO<sub>2</sub>$ . This high amount of  $CO<sub>2</sub>$  emissions has a direct impact on the environment. [Roscoe](#page-24-5) *et al.* (2011) indicated that traditional cement is responsible for the generation of 7 per cent of the world's greenhouse gasses. Chen *et al.* (2010) reported that the Portland cement manufacturing

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industries are under scrutiny these days because of the production of large volumes of  $CO<sub>2</sub>$ , representing 5-7 per cent of the total  $CO<sub>2</sub>$  anthropogenic emissions. The resulting climate change due to these emissions has global dimming [\(Hardjito, 2005\)](#page-24-0). **IEDT** 

#### Fly ash overview

Fly ash is a byproduct of burning finely ground coal in electricity generating plants. Fly ash is "the finely divided residue resulting from the combustion of ground or powdered coal, which is transported from the firebox through the boiler by flue gases" ([ACI Committee 116,](#page-23-3) [2000](#page-23-3)). Physically, fly ash is a fine and powdery material, and it is light tan to dark gray depending on its chemical constituents. Fly ash with high lime content is tan and light in color, whereas fly ash that is dark gray has an increased amount of unburned carbon. Fly ash particles are usually spherical, and finer than Portland cement and lime. Its diameter ranges from less than 1  $\mu$ m to no more than 150  $\mu$ m. Generally, it is captured from the flue gasses by using electrostatic precipitators or other filtration equipment, before it is discharged into the atmosphere. Fly ash is a pozzolan, siliceous material that reacts with an alkaline activator at ordinary temperature to produce cementitious compounds. Due to its spherical shape and pozzolanic properties, it can be used to replace Portland cement in concrete.

Fly ash is composed of the oxides of silica, aluminum, iron and calcium. It also contains different essential elements, including both macronutrients (Phosphorus – P; Potassium – K; Calcium – Ca; and Magnesium – Mg) and micronutrients  $(Zinc - Zn; Iron - Fe; Copper - Cu;$ Manganese – Mn; Boron – B; and Molybdenum – Mo). The chemical composition of fly ash varies according to the type of coal used. Anthracite and bituminous coal produce fly ash classified as Class F. It contains aluminosilicate glass and has less than 10 per cent of calcium oxide (CaO). Class C, or high calcium fly ash, is produced by burning lignite or subbituminous coal, and it typically contains more than 20 per cent of CaO. Aside from its chemical composition, the other characteristics of fly ash considered are a loss on ignition (LOI), fineness and uniformity. LOI is a measurement of unburnt carbon remaining in the ash. The fineness of the fly ash depends on the operating conditions of the coal crushers and the grinding process of the coal. Finer gradation results in more reactive ash that contains less carbon.

#### Fly ash usage

Fly ash works well when used in applications that normally require cement. Using it in durable construction materials benefits the environment. [Ram and Masto \(2014\)](#page-24-6) provided an overview of the potential applications of fly ash for soil amelioration. Fly ash improves the physical, chemical and biological qualities of soil. The application of fly ash along with various organic and inorganic amendments such as lime, gypsum, red mud, farm manure, animal manure, sewage sludge, composts and press mud, helps improve soil quality and leads to higher plant biomass production. In addition to using fly ash in amending soils, a common recommendation is difficult due to the heterogeneity in fly ash characteristics, soil types and agro-climatic conditions.

Di et al. (2012) reported that the utilization of fly ash has economic significance and environmental value. Fly ash has the same physicochemical properties as limestone mineral powder, which makes it a possible replacement for mineral powder. It was further determined that fly ash can improve the high-temperature stability of concrete, bituminous mixtures by completely replacing the limestone mineral powder.

[Ahmaruzzaman \(2009\)](#page-23-1) reported that fly ash could be used in construction, as a low-cost adsorbent for the removal of organic compounds, flue gas and metals, lightweight

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aggregate, mine backfill, road sub-base and zeolite synthesis. Research has also been conducted on the utilization of fly ash for the adsorption of  $NO<sub>x</sub>$ ,  $SO<sub>x</sub>$ , organic compounds and mercury in air, dyes and other organic compounds in waters [\(Ahmaruzzaman, 2009\)](#page-23-1). Fly ash has been used as an adsorbent for the removal of various pollutants [\(Ahmaruzzaman, 2009](#page-23-1)) because the unburned carbon content in fly ash improves its adsorption capacity. The adsorption capacity of fly ash is increased after chemical and physical activation.

The conversion of fly ash into zeolites has many applications such as ion exchange, molecular sieves and adsorbents converting waste material into a marketable commodity [\(Ahmaruzzaman, 2009](#page-23-1)). Basu *et al.* [\(2008\)](#page-23-4) reported that fly ash is used in agriculture applications to modify soils and also improve crop performance. The high concentration of the essential plant nutrients such as macronutrients (K, Ca, Mg, P and S) and micronutrients (Fe, Mn, Zn, Cu, Co, B and Mo) in fly ash increases the yield of many crops. Fly ash has also been used to stabilize erosion-prone soils. There is an expectation that using fly ash instead of lime in agriculture applications will reduce  $CO<sub>2</sub>$  emission, thus reducing the impact on global warming (Basu et al.[, 2008](#page-23-4)). [Wang](#page-24-1) et al. (2008) indicated that fly ash-based adsorbents are being proposed as an alternative to more expensive adsorbents such as activated carbon for the removal of heavy metals from industrial wastewater. This process was developed by synthesizing the two pure forms of zeolites (A and X) from fly ash to remove heavy metal (e.g. copper and zinc) ions. The removal mechanism of metal ions followed adsorption and ion exchange processes. The authors also attempted to recover heavy metal ions and regenerate adsorbents. From experiments, [Wang](#page-24-1) et al. (2008) were able to obtain removal efficiencies in the range of 81.45 to 99.73 per cent. [Rao and Rao \(2005\)](#page-24-7) asserted that textile effluents contain highly toxic, large numbers of complex metal dyes and may cause many waterborne diseases and increases the biological oxygen demand in water. Therefore, in the [Rao and Rao \(2005\)](#page-24-7) research, adsorption studies were conducted by treating the textile dye solutions of methylene blue (M-B) and Congo red (CR) with fly ash. The authors concluded from experimental observations that about 90-100 per cent removal of M-B and CR is possible when a lower concentration of fly ash is administered, implying fly ash may be effectively used as an adsorbent.

## Advantages of using fly ash

The advantages of using fly ash in concrete are improved workability, sulfate resistance, increased resistance to freezing, thawing, cohesiveness, improved long-term strength, reduced the water content of the mix, reduced the heat of hydration, decrease in permeability and increased resistance to alkali-aggregate reactions. Even after determining the cementitious characteristics in fly ash, its widespread acceptance there is still research being conducted into when using 100 per cent fly ash concrete. One of the techniques used for producing environmentally safer concrete is to replace Portland cement with fly ash.

Hardjito et at. (2005) stated that a significant improvement in the use of fly ash in concrete occurred with the development of high-volume fly ash (HVFA) concrete that successfully replaced ordinary Portland cement (OPC) in concrete up to 60 per cent. HVFA concrete is more durable and resource-efficient than OPC concrete. [Yazici](#page-24-8) et al. (2004) concluded that high-volume class C fly ash is suitable for use in the construction of products such as cast-in-place and precast products. However, external factors such as steam curing and superplasticizer were required to develop the required compressive strength.

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In today's environmentally conscious society, geopolymer concrete is a technology that is generating considerable attention in the concrete construction industry. Unlike Portland cement, geopolymer concrete depends on minimally processed natural materials or industrial byproducts to produce binding agents that rival Portland cement ([Hardjito, 2005\)](#page-24-0). The chemical composition of geopolymers is similar to zeolites with an amorphous microstructure instead of crystalline. Vijai *et al.* [\(2010\)](#page-24-9) reported that hardened geopolymer concrete has an amorphous microstructure, which is similar to ancient structures such as the Egyptian pyramids and Roman amphitheaters.

Geopolymer, which was pioneered by Joseph Davidovits, is an inorganic aluminosilicate polymer synthesized from predominantly silicon (Si) and aluminum (Al) materials of the geological origin or byproduct materials such as fly ash, metakaolin and granulated blast furnace slag. The polymerization process involves a fast-chemical reaction under alkaline conditions on Si-Al minerals that result in a three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds. [Davidovits \(1994\)](#page-23-0) and [Rangan \(2010\)](#page-23-0) coined the term Geopolymer for these binders. Geopolymers have several advantages, such as reducing the production of  $CO<sub>2</sub>$ , more economical, improved mechanical properties and eco-friendly.

Although the cementing properties of geopolymer materials have been known for more than three decades and have been used as the building materials in other ancient construction, still there are areas for further research. Continual awareness of environmental sustainability has encouraged researchers to conduct more research on alkali-activated concrete using industrial byproducts such as fly ash and blast furnace slag. [Barsoum \(2006\)](#page-23-5), in a paper, indicated that geopolymers have several advantages when compared to Portland cement. Some of the structures built using fly ash have survived for nearly five millennia. Unlike OPC, the production of geopolymers does not consume more energy and generates 90 per cent less  $CO<sub>2</sub>$  with minimal pre-processing and a simple mixing process. The raw materials used to produce geopolymer cement are commonly found in the Earth's crust and are almost indistinguishable from natural stone materials, immobilization of hazardous metals and preparation of nanometer-sized crystallites. The production technique is based on the polycondensation process rather than the hydration process. This technique helps in solidifying industrial, mining and urban waste discharged in the form of dry and wet powders into monoliths as recycling materials. Geopolymers have two key components, namely, the source materials and the alkaline liquids. The source materials for geopolymers based on alumina-silicate should be rich in Si and Al. These could be natural minerals such as kaolinite, clays and others. Alternatively, byproduct materials such as fly ash, silica fume, slag, rice husk ash, red mud and others could be used as source materials. The alkaline liquids are soluble alkali metals that are usually sodium or potassium based. The most common alkaline liquid used in geopolymerization is a combination of sodium hydroxide or potassium hydroxide (KOH) and sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) or potassium silicate ( $K_2SiO_3$ ). Alkaline liquid plays a significant role in the polymerization process. Reactions occur at a high rate when the alkaline liquid contains soluble silicate, either  $Na<sub>2</sub>SiO<sub>3</sub>$  or  $K<sub>2</sub>SiO<sub>3</sub>$ , compared to the use of only alkaline hydroxides ([Barsoum, 2006\)](#page-23-5).

[Rovnanik](#page-24-10) et al. (2016) revealed in their paper that fly ash geopolymer has a limitation that arises from increased shrinkage associated with deterioration of fracture properties. In this regard, the authors researched using a varied amount of multi-walled CNTs (MWCNTs) to improving the fracture properties of fly ash geopolymers. In conclusion, the authors suggested that MWCNTs tend to increase elastic modulus and compressive strength of fly ash geopolymer. Consequently, the fracture toughness and energy are increased with a higher amount of MWCNTs. [Sethi \(2014\)](#page-24-11) studied the strength and durability of concrete by

adding CNTs to a mix of cement and fly ash, where the 20 per cent by weight was the amount of cement replaced with fly ash. With the constant ratio of the fly ash and cement and varied amount of CNTs, Sethi (2014) asserted that the mix with higher amount of CNTs exhibited an increase in compressive strength, splitting tensile strength and Young's modulus as compared with mixes of concrete without CNTs such as only cement and fly ash. Besides, the author emphasized the established low permeability to chloride coupled with the concrete mix with CNTs achieving the desired durability properties. In a recent development, [Dvorak and Gazdic \(2019\)](#page-24-12) exploited CNTs in concrete to improve the electrical properties needed in smart concretes and structures. The study was completed with fly ash geopolymer mortars, and the consideration was based on electrical and mechanical properties (compressive and flexural strength). [Dvorak and Gazdic \(2019\)](#page-24-12) proposed that the assessment of the mortar conductivity, resistance and capacitance depends on the concentration of CNTs.

### Methodology

The methods used for the experiments in this study are discussed in this section. [Figure 1](#page-7-0) is a diagram of the research process.

This investigation used 100 per cent high calcium (ASTM Class C) fly ash and adopted the technologies similar to those used by [Hardjito](#page-24-2) *et al.* (2005) for Portland cement mix design and testing (ASTM C496, ASTM C192, ASTM C143, ASTM C109, ASTM C78 and ASTM C39). There were two phases to this investigation. Phase I consisted of experiments conducted to formulate a mix design to produce fly ash-based geopolymer concrete with proper proportioning of the different components of the fly ash concrete mix to achieve the specified properties. Phase II was focused on determining the best mix, based on compressive strength, developed in Phase I for testing the various properties of the concrete, such as slump, setting time, compressive strength and flexural strength tests. For all the experiments, 100 per cent class C fly ash (according to ASTM C 618) was used. The properties of the fly ash are shown in [Tables I](#page-8-0) and  $\Pi$  and [Figure 2](#page-8-2). The coarse and fine aggregate selection was based on ASTM C33, with a maximum diameter of 19 mm (3/4 inches) for coarse aggregate. The specific gravity was  $2.65 \text{ kg/m}^3$  and  $2.63 \text{ kg/m}^3$ , respectively. The aggregate was oven-dried at  $200^{\circ}$ C (392<sup>°</sup> F) for 24 h. [Table III](#page-8-3) shows the results of the sieve analysis completed for both coarse and fine aggregate. Strong alkali activator KOH was used instead of a sodium base activator for strengthening the surface area of the resulting specimens [\(Hardjito and Fung, 2010](#page-24-13)). The surfactant was adopted as a water reducer to improve the workability of the concrete mixes. The water used for the investigation was municipal water.

In addition to the materials mentioned previously, citric acid was used as the modifying agent in a solid (crystalline powder) state without any odor but with the taste of strong acid. The KOH generates heat and makes the mix set faster. Therefore, citric acid was used as a retarder to slow down the setting process. It is worth noting that no reaction occurs between citric acid, a weak acid, and KOH. Thus, it only works as a retarder. In one mix during the first phase, 20 Mule Team Borax  $\mathcal D$  (decahydrate borax) in dry powder form, which is a laundry detergent, was used to see its effect in retarding the setting time.

In Phase I of the investigation, MWCNT were added to some of the mixes. MWCNTS are rolled with a diameter ranging from 10 to 80 nm. The Young's modulus of an individual nanotube was approximately 1 TPa, and its density is approximately 1.33  $g/cm<sup>3</sup>$  ([Shah](#page-24-3) *et al.*, [2010\)](#page-24-3). Molecular mechanical simulations indicated that the CNTs' fracture strains were between 10 and 15 per cent, with corresponding tensile stresses on the order of 65 to 93 GPa

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<span id="page-8-3"></span><span id="page-8-2"></span><span id="page-8-1"></span><span id="page-8-0"></span>Figure 2. Fly ash, coarse aggregates, fine aggregates and surfactant



(Shah et al.[, 2010\)](#page-24-3). Their aspect ratios are generally beyond 1000. The major challenge associated with the incorporation of CNTs in cement-based materials is its poor dispersion (Shah et al.[, 2010](#page-24-3)). Poor dispersion of CNTs leads to the formation of many defect sites in the nanocomposite and limits the efficiency of the CNTs in the matrix. The use of CNTs requires ultrasonic energy to achieve effective dispersion (Shah *et al.*[, 2010](#page-24-3)). The equipment used included different sizes of beakers, flasks, buckets and test tubes medium-sized pans, medium-sized spatulas, a mortar mixer, concrete mixer, cube  $(2 \times 2 \times 2)$  inches), cylinders  $(3 \times 6 \text{ in and } 4 \times 8 \text{ inches})$ , beam  $(6 \times 6 \times 20 \text{ inches})$  molds, a probe sonicator, slump cones, rods and bases, and an oven for the curing mortar cubes. Additionally, the strength of the specimens was determined using a Forney concrete compressive and tensile strength machine. Other equipment used were a mall pump blower, laboratory spoons, a water jar and demolding equipment.

#### Mix design

The mix designs for this study were based on ASTM C109 and ASTM C192. The watercement (w/c) ratio is the controlling factor for most of the desirable properties of concrete, such as strength, durability, shrinkage potential, and permeability. Durable concrete mixes usually need a w/c ratio of 0.50 or less (Sparkman, 2006). However, in this investigation, a w/c ratio of 0.40 was used. The ratio of fly ash-sand-coarse aggregates is the primary factor that influences the properties of mix designs. The guiding principle of mix design is to pack as much as possible aggregate into the mix to make the mixes economic and reduce the required paste volume (Sparkman, 2006). The fly ash, sand and coarse aggregate were used in 1:2:2 ratio for the cylinder and beam specimens, and fly ash and sand were used in 1:2.75 ratio for cube specimens ([Tables VI](#page-10-0) and [VII\)](#page-11-0) per ASTM C192 and ASTM C109. It is worth noting that water reducers offer the most benefits during the mix design process (Concrete Network, 2006). A water reducer is used to reduce the amount of water required to generate the concrete mix. In Phase I, different quantities of water reducer were used for the experiment, especially for the experiments using CNTs ([Table VIII\)](#page-11-1). In Phase II, 0.08 per cent of the total weight of the fly ash-sand-coarse aggregate was used. The alkaline solution is used to increase the strength and durability of geopolymer concrete.

Consequently, KOH was used to strengthen the mortar and concrete, whereas citric acid was used to retard the setting time of the mix. As alkaline activators are one of the key elements of geopolymer concrete, in Phase I, different amounts were used, and the effects were observed by performing compressive strength tests. The KOH solution was prepared by dissolving KOH pellets in water. The mass of the KOH solids in a solution was varied depending on the concentration of the solution expressed in terms of molarity, M. For instance, KOH solution with a concentration of 3M consisted of  $3 \times 56 = 168$  g of KOH solids per liter of the solution, where 56 is the molecular weight of KOH. In one of the mixes, borax was used as a retarder, and the setting time was observed ([Table VI\)](#page-10-0).

In Phase II, the best proportion of KOH and citric acid, which exhibited the highest compressive strength in Phase I, was used. CNTs were used in Phase I of the investigation. Five dispersions were prepared by mixing CNT with alkaline surfactant solution (100 g) at surfactant - CNTs weight ratios of 4, 6.25, 7.5, 9 and 2.9 for the cubes (Shah *et al.*[, 2010](#page-24-3)). In five out of the six mixes, a constant amount of CNTs, 0.56 g, was used, whereas, in the last mix, the amount of CNTs was increased to 1.2 g to observe any changes in the characteristic of the cubes. As CNTs tend to stick to each other, they need ultrasonic energy to achieve adequate dispersion in a solution. Therefore, a probe sonicator was used to disperse CNTs in the alkaline solution. The dispersions were sonicated at room temperature at the power of

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35W-49 W for 20 min with a medium intensity tip of diameter 0.25 inches (6.4 mm). The frequency used for the dispersion was 5 kHz.

#### Mixture proportion

To accomplish the aims of this study, experiments were divided into two phases. This section shows the exact proportion of the materials used for the mixes of both phases. [Table V](#page-10-1) indicates the number of mix designs prepared in Phases I and II. Several mix designs were prepared in Phase I to observe the results. In Phase II, only one set of mix designs, based on the best result achieved in Phase I, was prepared. CNT mixes were not repeated in Phase II, whereas, one mix design was prepared for the beam [\(Tables V\)](#page-10-1). [Table VI](#page-10-0) shows the proportion of materials used to prepare mix designs for cylinders and cubes in phase I ([Table IV\)](#page-10-2).

[Tables VI, VII](#page-10-0) and [VIII](#page-11-1) show the proportions of materials derived from Phase I and used to prepare mix designs for cylinders, beams and cubes in Phase II. [Table IX](#page-12-0) provides the number and sizes of the specimens, curing temperatures, quantities and the type of test used in Phase I and Phase II, along with the total amount of concrete prepared used in the research [\(Table V](#page-10-1)).

Table IV. Proportions of sonicated solution Proportions of sonicated solution Alkaline solution (g)  $\begin{array}{cccc} 97.2 & 95.9 & 95.2 & 94.4 & 95.3 \\ 95.9 & 2.24 & 3.5 & 4.2 & 5.04 & 3.5 \end{array}$ Surfactant (g)  $2.24$   $3.5$   $4.2$   $5.04$   $3.5$   $5.04$   $1.2$   $1.2$   $5.04$   $1.2$  $\text{CNTs}$  (g) 0.56 0.56 0.56 0.56 0.56 1.2 SFC/CNT 4 6.25 7.5 9 2.9





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<span id="page-10-2"></span><span id="page-10-1"></span><span id="page-10-0"></span>

To analyze the behavior of the fresh mortar, slump tests were performed. This test was based on ASTM C143. After casting, the specimens were kept at room temperature and demolded after  $24 \pm 4$  h and marked with the date of mixing and casting (preparation). The specimens in Phase I was cured at room temperature. In Phase II, cube specimens were cured in the oven using temperatures similar to room temperature, and 37.8° C (100° F), 65.6° C (150° F), 93.3° C (200° F), 121.1° C (250° F), 148.9° C (300° F), 176.7° C (350° F) and 204.4° C (400° F), whereas cylinders and beams were cured only at room temperature. The setting of concrete is a gradual transition from a liquid to a solid. The final setting of concrete relates to the point where stresses and stiffness start to develop in freshly placed concrete. The initial set time is crucial because it provides an estimate of when the concrete has reached the point where it has stiffened to such an extent that it can no longer be vibrated without damaging it. The setting time of the fresh concrete mortar was analyzed after placing it into the mold. After each mixture had been placed, specimens were observed for several hours. It was found that the specimens with higher amounts of KOH had shorter setting time than those with comparatively lesser amounts of KOH. [Figure 3](#page-13-0) shows the slump test process and the cubes, cylinders and beam specimens generated ([Tables VI, VII, VII](#page-10-0) and [IX](#page-12-0)). **IEDT** 18,6 1626

### Compressive strength tests

<span id="page-11-0"></span>Phase  $II^*$ 

molds

The compressive strength test (ASTM C39) is the most common method used to measure the strength and durability of concrete. Compressive strength was selected as the benchmark parameter because it is the most important parameter considered in the structural design of concrete structures. It is reported in units of pound-force per square inch (psi) in US units or pascals (Pa) in SI units. The results of this test method are used as the basis for quality control of concrete proportioning, mixing and placing operations, determination of compliance with specifications and control for evaluating the effectiveness



<span id="page-11-1"></span>

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Table IX. Phase I and II sizes, numbers, specimen and quantities

of admixtures (ASTM C39/C39M  $-11a$ ). In Phase I, the compressive strength tests were performed on the cylinders and cubes in the Structural Laboratory of the Civil Engineering Department at North Dakota State University. In phase II, the tests were conducted in the Construction Management and Engineering (CME) laboratory on the cylinders and cubes. The compressive strength tests were conducted at 24 h and 3, 7, 14, 21 and 28 days. ASTM C39 and ASTM C109 were followed for the compression test for both the cylinders and cubes. **IEDT** 18,6

## Flexural strength test

The three-point bending tests were conducted on a loading frame to calculate, according to ASTM C78, the flexural tensile strength on standard beam specimens of size 0.1524 m  $\times$  $0.1524 \text{ m} \times 0.508 \text{ m}$  (6  $\times$  6  $\times$  20 inches) by using the flexural strength machine in the CME laboratory. The loads were gradually increased at the loading rate of about 45.36 kg/s (100 lb/s). The test was carried out on the 28th day. The following formula calculated the modulus of rupture for the flexural strength of the beam:

$$
R = PL/bd^2,
$$
 (1)

where:

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- $R =$  Flexural strength (n/m<sup>2</sup> or psi);
- $P =$  the maximum applied load indicated by the machine at failure (kg or lb);
- b = Average width of the specimen (m or inch); and
- $d =$  Average depth of the specimen (m or inch).

Equation (1) is used if the fracture initiates in the tension surface within the middle third of the span length.

## Results and discussion

This section provides an analysis of the materials used, the experimental design and the results of the experiments. The results were evaluated to compare the relative compressive and flexural strength tests along with the characteristic tests such as a slump and setting time of all the mixtures. All the tests were performed using ASTM standards, compressive strength (ASTM C39), flexural strength (ASTM C78) and slump test (ASTM C143). Developing fly ash products and maintaining the desired properties of strength and workability were some of the goals of this study. Therefore, the study focused on the concrete characteristic tests such as a slump, setting time, compressive strength and flexural strength. Different materials such as KOH, citric acid, borax, CNT and surfactants were added to the fly ash C mixes, and their effects on the properties of the mixes were observed.

#### Slump

The results of the slump tests performed in Phase II are shown in [Figure 4.](#page-14-0) Three slump tests, slump 1, 2 and 3 were performed to compare the values obtained. Slump tests were repeated at four intervals of time, immediately, and at 7, 12 and 16 min after mixing to

<span id="page-13-0"></span>Figure 3. Slump test process, cubes, cylinders and beam specimens





<span id="page-14-0"></span>observe the workability of the mix. The results indicated that the workability span for all the mixes was short, i.e. the mixes tended to set faster; therefore, the slump value decreased as the time of mixing increased ([Hardjito](#page-24-2) *et al.*, 2005). This is because of the presence of high lime and the activation of the alkaline solution [\(Nicholson](#page-24-14) *et al.*, 2005). In [Figure 4](#page-14-0), as the mixing time was increased, the slump value dropped considerably. Comparing test results, appropriate workability was achieved at the 12 min test with a slump value from 0.1524 to 0.1778 m (6 to 7 inches).

#### Setting time

The setting times for the mixes were observed throughout the investigation. It was discovered that the specimens with a higher molar value of KOH had shorter setting times compared to those with lesser values. The overall setting time observed was not longer than 30 min. This is because fly ash C tends to set faster than other geopolymers due to the presence of high amounts of lime. Also, the activation of alkaline solutions generates heat, which causes the mixes to set faster. [Figure 5](#page-14-1) shows a graph of setting time with the mixes' molar values. [Figure 5](#page-14-1) indicates that as the molar value of the mixes increases, the setting time becomes shorter. The lowest molar value of 1.5 had a setting time of 30 min, whereas the highest molar value of 4 had a setting time of 10 min.



<span id="page-14-1"></span>Figure 5.

Compressive strength test **IEDT** 

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Compressive strength is considered to be one of the most important properties of hardened concrete. It is generally the principle property value used to investigate the quality of concrete according to ASTM C39. Consequently, it is important to evaluate whether changes in the mix composition affects the early and late compressive strength of concrete. Compressive stress results for cylinders and cubes at 1, 3, 7, 14, 21 and 28 days were investigated.

## Phase I results

[Figure 6](#page-15-0) shows the increase in strength as the age of the specimen progressed, with slight inconsistencies. It was noted that the alkali activators had a crucial role in strengthening the concrete materials by comparing the result from each mix. The mixes without KOH (Mix 1) resulted in lower strength than the mixes with KOH. The highest strength in Mix 1 is 1,498 psi, whereas the other mixes with KOH exhibited higher strengths. Moreover, there was a significant increase in strength in Mixes 2 through 5, as the molarity of KOH increased. [Figure 6](#page-15-0) shows the lines constantly increasing with slight variations for Mix 5. However, as the molarity of KOH was further increased in Mix 6, no gain in strength was observed; instead, it exhibited a slight decline. The strength dropped lower than Mix 5, and this could be attributed to the fact that the optimum strength was reached at Mix 5. Thus, an increase in the amount of KOH produced excessive heat causing the mix to set faster without adequately bonding. The crack patterns of the cylinders were compared, with the given patterns in ASTM C39, notably the Type 3 fracture pattern, which is columnar vertical cracking through both ends, no well-formed cones [\(Figure 7](#page-16-0)).

## Cubes

Comparable to the results obtained for the cylinders, the compressive strength of the cubes also improved with the increasing amount of KOH. However, there were some inconsistencies. The strength of Mix F1 increased with age. Mix F2 had a decline in strength at 21 days and then improved slightly at 28 days. Similarly, Mix F3A had a decrease in strength after 21 days. The strength for Mix F3 declined after 14 days until the 28th day. The drops in strength are attributed to the molarity of KOH being increased. The borax added to Mix F3A increased the setting time slightly and lowered the use of water when





<span id="page-15-0"></span>Figure 6. Phase I: compressive strength of the cylinders for various mixes





<span id="page-16-1"></span><span id="page-16-0"></span>Figure 8. Phase I: compressive strength of the cubes for various mixes

compared to the other cubes. [Figure 8](#page-16-1) shows a graph of the compressive strength gain and losses for the cubes.

## Carbon nanotubes

[Figures 9](#page-17-0) and [10](#page-17-1) show the compressive strengths of the cubes with CNT and non-CNT, respectively. The non-CNT cubes were only able to reach the second-highest strength obtained by the CNT cubes. However, the early strength of the non-CNT cubes was similar to the CNT cubes. The other observable difference found between the mixes with and without CNT was rigidness. Comparably, CNT cubes were strong enough to resist the compressive force. The CNT concrete mix withstood the compressive force until they were utterly crushed, whereas the non-CNT cubes were easily crushed once cracks formed in the concrete. Generally, the cubes with CNT exhibited increases in compressive strength with age, whereas the non-CNT cubes exhibited inconsistencies, as shown in [Figures 9](#page-17-0) and [10](#page-17-1).

<span id="page-17-0"></span>

### <span id="page-17-1"></span>Phase II results

## **Cylinders**

The results shown in [Figure 11](#page-18-0) demonstrates that the compressive strength increased gradually from the first day and peaked after 14 days. There was a sharp drop at 21 days and then an increase in strength at 28 days. The significant change in strength could have been due to the presence of excessive moisture from the considerable amount of rain experienced in Fargo during the laboratory experiments. The crack patterns were similar to crack types in the ASTM C39, especially type 1. The vertical cracks in the upper side indicate that there were no well-defined cones formed. [Figure 12](#page-18-1) shows the compressive strength test used on the cylinders and the crack pattern type 1 from ASTM C39.

#### Cubes

[Figure 13](#page-18-2) shows the compressive strength of the various cubes, which were oven-dried, and [Figure 14](#page-19-0) shows the comparison of the compressive strength of the cubes at different age and different curing temperatures. The effect of age and temperature on the strength is shown in [Figures 15](#page-20-0) and [16.](#page-21-0) of the cubes. The curves are different concerning age and

<span id="page-18-1"></span><span id="page-18-0"></span>

<span id="page-18-2"></span>temperature changes. Generally, compressive strength decreased with an increase in temperature yet increased with age ([Figure 13](#page-18-2)).

On day one, the strength was lower at higher temperatures, as shown in [Figure 14](#page-19-0). The reason could be the absence of a strong mortar bond. Therefore, as the temperature was increased, the bond started to deteriorate. On day three, the strength remained constant with increases and decreases. However, there was a moderate rise in the strength of the cube

cured at  $204.4^{\circ}$ C (400 $^{\circ}$  F). There were slight increases and decreases as the curing temperature increased on the seventh day. Significant changes in strength occurred on the 14th and 21st days. There was a decrease in strength as the temperature increased after the 28th day. After reaching its peak strength at room temperature, the cube started to lose strength. Even though many fluctuations in strength occurred, the strength of the cube improved with age. Considerable increases in strength were observed from day 1 to 28. The highest strengths were achieved at room temperature and 37.8° C (100° F). This could be due to the formation of a stronger bond with age. However, the strength did not increase substantially with temperature as the specimen aged. The results are as listed in [Table X.](#page-21-1)

[Figures 16](#page-21-0) shows the compressive strength gained at each temperature. Except for a slight strength loss on the third day, the room temperature cured cubes gained strength gradually with age. The strength achieved on the 28th day was the highest. Steady strength gain was observed along with age at 37.8° C (100° F) [\(Figure 16\)](#page-21-0), except on the 3rd and 21st day tests. Similar to the room temperature, concrete strength achieved on the 28th day was the highest of the ages. From the third day test, curing at  $65.6^{\circ}C(150^{\circ}F)$  showed a steady increase in the strength of the cubes ([Figure 16](#page-21-0)). At  $93.3^{\circ}$ C (200 $^{\circ}$  F), the steady increase in strength was disrupted at 21 days with a slight decrease in the rate of strength. Strength gain also occurred after 28 days. At 121.1° C (250° F), the compressive strength increased steadily with age until the 14th day. After that, not much strength was gained. At  $148.9^{\circ}$ C (300° F), a sudden decrease in strength occurred after the 14th day test. There was a steady increase in strength for 176.7° C (350° F) cure temperature, with a slight decrease in the 28th day test. At  $204^{\circ}C$  (400 $^{\circ}$  F), the gain in strength was disrupted on the 14th and 28th days. The results determined that changing the temperature during curing does not affect the strength. However, with additional experiments, it might be possible to develop mixes for geopolymer concrete with higher strength.

## Flexural strength tests

One of the measures of the strength of an unreinforced concrete beam is to resist failure in bending which is one of the properties used to observe the quality of concrete, according to ASTM C78 and expressed as the modulus of rupture in psi. According to the ASTM C78, this test is used in the formulation of the proportioning, mixing and placement process. It is used to test the concrete in slabs and pavements (ASTM C78). The flexural strength test for a beam cured at room temperature was performed on the 28th day. The flexural strength of

<span id="page-19-0"></span>





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<span id="page-20-0"></span>the beam on the 28th day was 339 psi, however, cracks were found to have formed at the middle third of the span length.

#### Conclusion and recommendations

The main purpose of this study was to design concrete mixes using green alternatives to replace Portland cement. Fly ash C was used as a green cement alternative in the experiments. The reason for using fly ash C was its self-cementing properties, which are similar to Portland cement ([Ahmaruzzaman, 2009\)](#page-23-1). Along with chemicals such as KOH and citric acid, fibers in the form of nanomaterial CNTs were used as a reinforcing material to increase the strength of the concrete. CNT was used to determine the improved mechanical properties of fly ash mortars.

A review of current trends in using fly ash provided direction to the research in formulating the mix designs and experiments. In Phase I, several experiments were



<span id="page-21-1"></span><span id="page-21-0"></span>

performed to test the mixture proportioning of fly ash-based geopolymer concrete to generate five mixes for cylinders and four mixes for cubes were developed. In Phase I, CNTs were used only for the mix design of cubes. To compare the designs, non-CNT cubes were also prepared using the same mix proportions. With the mix designs, compressive strength tests were performed at 1, 3, 7, 14, 21 and 28 days.

Only one set of mix designs were prepared for the cylinders, cubes, and beam specimens in Phase II. The Phase II investigation was based on the results achieved in Phase I. In addition to compressive and flexural strength tests, slump tests and setting time tests were also performed on the concrete. An analysis of the results was performed after completing the experiments.

For testing the fresh mortar properties, slump and setting time tests were conducted along with compressive and flexural strength tests to study the hardened concrete properties. The slump tests indicated that the mixes tend to set faster with lower slump values. This could be as a result of the presence of a high amount of lime and the activation of the alkaline solution [\(Nicholson](#page-24-14) *et al.*, 2005). The slump values decreased substantially as the mixing time was increased.

The setting time in all the experiments indicated that the specimens with a higher molar value of KOH had shorter setting times compared with those with lower molar values of KOH. The overall setting times were not more than 30 min because fly ash C tends to set faster in the presence of high amounts of lime. The activation of the alkaline solution generates heat, which also causes the mix to set faster. In Phase I, the results for the compressive strength test of cylinders showed that as the age progresses, strength gains were observed in each mix with slight inconsistencies.

From the results, it was determined that alkali activators play a key role in strengthening the concrete materials. The higher the amount of the KOH, the higher the strength achieved. Similar results were obtained for the cube mixes. The CNTs cubes did not exhibit any significant increase in strength, but the strength was similar to the non-CNT cubes. In Phase II, the cylinders did not exhibit any significant strength gains. The strength gains for the cubes occurred with lots of fluctuations. The strength of the cubes improved as the age progressed. However, the strength did not increase significantly with temperature. The expected flexural strength of the beams was not achieved in Phase II because of different reasons, which included the moisture content in the mixes and the laboratory.

In conclusion, the study was conducted to design a concrete mix using fly ash C as a green cement alternative to Portland cement. The conclusions based on the experimental work done in this study are as follows:

- The use of fly ash C as a full replacement for Portland cement needs an alkaline activator to produce the same strength as Portland cement. Therefore, the use of an alkaline activator has a significant effect on the strength of the concrete. In Phase I, the results indicated that, as the molarity of KOH increased, the fly ash concrete gained strength and become sturdier. However, the problem with the increased molar value of KOH is the generation of heat, resulting in a shorter setting time of the specimens and a decrease in strength.
- The highest strength of 10,142 psi was obtained using Mix 5 in Phase I.
- Although the concrete mix design with the highest strength achieved in Phase I was used in Phase II, the strength achieved was much less than the Phase I.
- It was determined that curing does not have any effect on strength. From comparing the strength gains, it is concluded that the key factor for increasing strength is the

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<span id="page-23-5"></span><span id="page-23-4"></span><span id="page-23-3"></span><span id="page-23-2"></span><span id="page-23-1"></span><span id="page-23-0"></span>Davidovits, J. (1994), "Properties of geopolymer cements, alkaline cements and concretes, Kiev Ukraine", First International Conference on Alkaline Cements and Concretes, Scientific Research Institute on Binders and Materials, Kiev State Technical University, Kiev, Ukraine, pp. 131-149.

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