INVESTIGATION OF THE TENSILE AND FRACTURING BEHAVIOUR OF GEOPOLYMER CONCRETE MADE BY SODIUM HYDROXIDE ACTIVATION OF GOLD MINE TAILINGS

by

Ammad Khan
A thesis submitted to the Faculty and the Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Master of Science (Underground Construction and Tunnel Engineering).

Golden, Colorado

Date ____________________

Signed: _______________________

Ammad Khan

Signed: _______________________

Dr. Ahmadreza Hedayat
Thesis Advisor

Golden, Colorado

Date ____________________

Signed: _______________________

Dr. Michael A. Mooney
Program Director
Underground Construction and Tunnel Engineering
ABSTRACT

Geopolymerization is the process of alkali activation of alumino-silicates in raw materials to make construction materials. Industrial wastes like fly ash and blast furnace slag and mining industry wastes such as mine tailings in form of crushed rocks are commonly used raw material for production of geopolymers. Geopolymerized mine tailings have a wide application in building and construction industry. The compressive strength of geopolymer has been extensively studied by many researchers; however, the tensile strength remains largely unexplored. Understanding of tensile behavior and fracture mechanics of geopolymerized tailings is critical and established techniques by the rock mechanics community such as the direct and indirect tensile tests can be utilized for evaluation of the material’s behavior. Different indirect tensile test methods such as splitting tensile strength, three- and four- point bending, and semi-circular bend test have been used.

The research objectives of this study were to evaluate the splitting tensile strength and flexural strength of the geopolymer concrete, identify critical strain levels for crack initiation, determine the mode I fracture toughness of the material, and ascertain the critical strain energy release rate. In this study, the splitting tensile strength of alkali activated geopolymerized mine tailings was investigated using the Brazilian indirect tensile method and the flexural strength has been investigated using semi-circular bend (SCB) test along with the digital image correlation (DIC) technique. A novel experimental set up was developed to perform the tests for this new type of material. For specimen preparation, sodium hydroxide (NaOH) was used as the alkali activator with molar concentrations of 8M, 10M and 12M. Initial water/tailing ratio was kept as 20% and specimens were tested after 7, 14 and 28 days of curing. All the specimens were kept in
oven at 75°C for an initial 24 hours and then sealed and cured at room temperature. Prior to the testing, the specimen was placed in the oven at 75°C for 24 hours to reach the dry condition. For SCB test, the semi-circular specimen were prepared with notch as well as without notch. SCB specimen without notch was used to ascertain the flexural strength of the specimen and SCB specimen with notch were used to characterize the fracturing behavior of the material. The length of the notch to the radius (a/R) of the specimen varied from 0.2 to 0.3.

The results indicate that splitting tensile strength of the specimen increased with the increase in molar concentration of NaOH. For 12M specimens the splitting strength was 1.8MPa. Generally, the tensile strength remained within the range of 10 to 12 % of the compressive strength of the geopolymer. The average flexural strength of the specimen was 1.7MPa for 10 M concentration of the specimen. The average mode I fracture toughness of the material was $0.2 \text{ MPa}\sqrt{m}$. The critical energy release rate, the measure of decrease in potential energy due to the increase in fracture surface area was estimated to be 32.2 N/m. The results from DIC analysis were compared with the extensometer physically attached at the bottom of the specimen to measure crack mouth opening displacement and both results were comparable.
TABLE OF CONTENTS

ABSTRACT ................................................................................................................................... iii

LIST OF FIGURES ..................................................................................................................... viii

LIST OF TABLES ......................................................................................................................... xi

ACKNOWLEDGEMENTS .......................................................................................................... xii

LIST OF ABBREVIATIONS ...................................................................................................... xiv

CHAPTER 1 INTRODUCTION ..................................................................................................1

1.1 Introduction and Research Motivation ..............................................................................1

1.2 Research Objectives ..........................................................................................................5

1.3 Thesis Outline ...................................................................................................................6

CHAPTER 2 REVIEW OF THE RELEVANT LITERATURE ..................................................8

2.1 Geopolymerization/Alkali Activation of the Materials .....................................................8

2.1.1 Use of Industrial Wastes for Geopolymerization ........................................................ 10

2.1.2 Effect of the Curing Environment and Temperature ................................................ 12

2.1.3 Effect of Molar Concentration of the Alkali Activation Agent ................................ 13

2.1.4 Use of Mine Tailings for Geopolymerization .............................................................. 14

2.2 Brazilian Indirect Tensile Strength Test ......................................................................... 17

2.3 Semi Circular Bend (SCB) Test ...................................................................................... 22

2.4 Review of Relevant Concepts from Fracture Mechanics .............................................. 24
2.4.1 Energy Balanced Approach ................................................................. 24
2.4.2 Stress Intensity Factor Approach .......................................................... 26
2.4.3 Relationship between Stress Intensity Factor and Energy Release Rate .... 28
2.4.4 Effect of Plane Stress and Plane Strain Conditions on Stress Intensity Factor ..... 28
2.4.5 Calculating Stress Intensity Factor using Williams’ Series ......................... 29
2.4.6 Cohesive Zone Model ............................................................................. 30
2.4.7 Crack Band Model .................................................................................. 31
2.5 Digital Image Correlation (DIC) Technique ................................................... 32
   2.5.1 Historical Development of DIC Technique ............................................. 32
   2.5.2 Fundamental Principles of DIC Technique ............................................. 33
   2.5.3 Experimental Considerations for Application of DIC Technique ............. 38
CHAPTER 3 EXPERIMENTAL PROGRAM .......................................................... 40
   3.1 Raw Material .......................................................................................... 40
   3.2 Specimen Preparation .............................................................................. 42
      3.2.1 Specimen Preparation for Brazilian Indirect Tensile Test .................. 43
      3.2.2 Specimen Preparation for Semi-circular Bend Test ............................ 47
   3.3 Experimental Setup ................................................................................ 50
      3.3.1 Experimental Setup for Brazilian Indirect Tensile Strength Test ........... 50
      3.3.2 Experimental Setup for Semi-circular Bend Test ............................... 53
CHAPTER 4  BRAZILIAN INDIRECT TENSILE STRENGTH TEST .........................55

4.1  Splitting tensile strength of the specimen .......................................................55

4.2  DIC results .............................................................................................................58

4.3  Determining the elastic properties using DIC results ....................................66

CHAPTER 5  SEMI-CIRCULAR BEND (SCB) TEST .............................................71

5.1  Results from Semi-circular Bend Test without Notch ......................................71

5.2  Calculation of Fracture Toughness using SCB Test with a Notched Specimen ......72

5.3  Correlation between Fracture Toughness and Splitting Tensile Strength ..........74

5.4  Estimation of the Critical Energy Release Rate .................................................74

5.5  Crack Mouth Opening Displacement .................................................................75

5.6  Full Field Tensile Strain in Horizontal Direction from DIC Analysis .............76

5.7  Width of the Fracture Process Zone .................................................................82

CHAPTER 6  CONCLUSION AND FUTURE WORK .........................................84

6.1  Conclusion and Future Work .............................................................................84

6.1.1  Research Tasks ...............................................................................................84

6.1.2  Research Findings ..........................................................................................86

6.2  Recommendations for Future Work .................................................................88

REFERENCES ..............................................................................................................90
LIST OF FIGURES

Figure 3-1  Grain size distribution of the raw mine tailings used in this study ......................... 40

Figure 3-2  XRD pattern along with percentage of each mineral in raw tailings (Q: quartz; M: muscovite; C: calcite) ........................................................................................................ 42

Figure 3-3  Procedure for preparation and testing for Brazilian indirect tensile testing............ 46

Figure 3-4  Pictorial view of preparation process through different stages .............................. 47

Figure 3-5  Schematic of the specimen for semi-circular bend test........................................... 49

Figure 3-6  Preparation of semi-circular specimen for SCB test............................................... 49

Figure 3-7  Experimental Setup for Brazilian indirect tensile test........................................... 51

Figure 3-8  Location of virtual strain gauges on the specimen for Brazilian indirect tensile strength test ....................................................................................................................... 53

Figure 3-9  Experimental setup for semi-circular bend test....................................................... 54

Figure 4-1  Representative load-displacement curves for geopolymerized samples based on molar concentration and curing age (M: molar concentration of NaOH and D: curing age in days) ........................................................................................................ 55

Figure 4-2  Tensile strength of the specimen for different molar concentrations and curing age (M: molar concentration of NaOH and D: curing age in days) ........................................ 56

Figure 4-3  Histogram representing the grayscale intensity of the speckle pattern on the surface of the specimen for Brazilian indirect tensile test ................................................. 58
Figure 4-4  Location of the virtual strain gauges (labeled as S-1 to S-9) .......................... 59

Figure 4-5  Variation of strain and load over the time for 8 M-28 days specimen.......... 60

Figure 4-6  Full field strain profile for selected points (a, b, c and d) along the curve for specimens-curing age of (1) 8 M-28 days, (2) 8 M-7 days, (3) 10 M-28 days and (4) 10 M-7 days .......................................................... 62

Figure 4-7  Variation of strain and load over the time for 8M-7 days specimen .......... 65

Figure 4-8  Variation of strain and load over the time for 10M-28 days specimen .......... 65

Figure 4-9  Variation of strain and load over the time for 10 M - 7 days specimen .......... 66

Figure 4-10 Schematic for calculation of elastic properties from DIC analysis ............. 68

Figure 4-11 Horizontal stress at the center of the specimen based on DIC analysis ....... 69

Figure 4-12 Full field horizontal stress at the surface of the specimen near to the failure .... 70

Figure 5-1  Load displacement curve for SCB specimen without notch ....................... 71

Figure 5-2  Mode I Fracture toughness from SCB notched tests ................................. 73

Figure 5-3  Crack mouth opening displacement based on extensometer and DIC analysis .... 75

Figure 5-4  Location of virtual strain gauges for SCB Specimen ................................. 76

Figure 5-5  Evaluation of horizontal strain and force throughout the experiment for SCB specimen with a/R = 0.2 .......................................................... 77
Figure 5-6  Evaluation of horizontal strain and force throughout the experiment for SCB specimen with \( \frac{a}{R} = 0.25 \) ................................................................. 78

Figure 5-7  Evaluation of horizontal strain and force throughout the experiment for SCB specimen with \( \frac{a}{R} = 0.3 \) ................................................................. 79

Figure 5-8  Full field strain profile for selected points (a, b, c and d) along the curve for SCB specimens (1) \( \frac{a}{R} = 0.2 \), (2) \( \frac{a}{R} = 0.25 \) and (3) \( \frac{a}{R} = 0.3 \) ........................................ 81

Figure 5-9  Six Horizontal Lines marked on the specimen to identify the width of fracture process zone (origin of the coordinates system is transformed at the notch tip) .. 82

Figure 5-10  Horizontal displacement of specimen at different heights from the notch tip ...... 83
LIST OF TABLES

Table 3-1  Geotechnical properties of the raw mine tailings .................................................. 41
Table 3-2  Preparation of different types of specimen for Brazilian tensile test ................. 46
Table 3-3  Preparation of different types of specimen for semi-circular bend test .......... 48
Table 4-1  Summary of Splitting tensile test ........................................................................ 57
Table 6-1  Strength requirements of construction material for different construction purposes ................................................................................................................ 87
ACKNOWLEDGEMENTS

I would like to thank my advisor Dr. Reza Hedayat for his support throughout my research. He was always available to help me out and gave wonderful suggestions and mentorship. His care for his student’s career progression is commendable. I would also like to extend my thank you notes to the respected members of my thesis committee; Dr. Jamal Rostami and Dr. Linda Ann Figueroa; they provided great suggestions in review of my thesis.

I also owe a thank you note to Dr. Nan Zhang who guided me throughout my research work and gave insightful comments on my work.

I owe my success in life to my parents, family and friends. I would not be where I am today without them. My son, Muhammad Umar Khan and adorable daughter, Ezzah Ammad always provided me refreshing energy after my work time.

The financial support provided by the Universidad Nacional de San Agustín (UNSA) through the joint Center for Mining Sustainability with the Colorado School of Mines is highly acknowledged.
To my beloved wife, adorable kids and my parents.......
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AASHTO</td>
<td>American Association of State Highway and Transportation Officials</td>
</tr>
<tr>
<td>AOI</td>
<td>Area of interest</td>
</tr>
<tr>
<td>ACI</td>
<td>American Concrete Institute</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>a/R</td>
<td>Notch length to the radius of the specimen ratio</td>
</tr>
<tr>
<td>C&lt;sub&gt;CC&lt;/sub&gt;</td>
<td>Cross correlation coefficient</td>
</tr>
<tr>
<td>C&lt;sub&gt;SSD&lt;/sub&gt;</td>
<td>Sum of squared differences correlation coefficient</td>
</tr>
<tr>
<td>CCD</td>
<td>Charged-coupled devices (CCD)</td>
</tr>
<tr>
<td>CDOT</td>
<td>Colorado Department of Transportation</td>
</tr>
<tr>
<td>CMOD</td>
<td>Crack mouth opening displacement</td>
</tr>
<tr>
<td>CMOS</td>
<td>Complementary metal oxide semiconductor</td>
</tr>
<tr>
<td>DIC</td>
<td>Digital image correlation</td>
</tr>
<tr>
<td>EPA</td>
<td>Environmental Protection Agency</td>
</tr>
<tr>
<td>FDOT</td>
<td>Florida Department of Transportation</td>
</tr>
<tr>
<td>FHWA</td>
<td>Federal Highway Administration</td>
</tr>
<tr>
<td>Acronym</td>
<td>Description</td>
</tr>
<tr>
<td>---------</td>
<td>-------------</td>
</tr>
<tr>
<td>ISRM</td>
<td>International Society for Rock Mechanics and Rock Engineering</td>
</tr>
<tr>
<td>LEFM</td>
<td>Linear Elastic Fracture Mechanics</td>
</tr>
<tr>
<td>NCC</td>
<td>Normalized cross correlation</td>
</tr>
<tr>
<td>MT</td>
<td>Mine tailings</td>
</tr>
<tr>
<td>MTS</td>
<td>Material Testing Service</td>
</tr>
<tr>
<td>NaOH</td>
<td>Sodium hydroxide</td>
</tr>
<tr>
<td>NSSD</td>
<td>Normalized sum of squared difference</td>
</tr>
<tr>
<td>SCB test</td>
<td>Semi-circular bend test</td>
</tr>
<tr>
<td>SIF</td>
<td>Stress intensity factor</td>
</tr>
<tr>
<td>UCS</td>
<td>Unconfined compressive strength</td>
</tr>
<tr>
<td>USCS</td>
<td>Unified Soil Classification System</td>
</tr>
<tr>
<td>ZNCC</td>
<td>Zero normalized cross correlation</td>
</tr>
<tr>
<td>ZNSSD</td>
<td>Zero normalized sum of squared difference (ZNSSD)</td>
</tr>
</tbody>
</table>
CHAPTER 1
INTRODUCTION

1.1 Introduction and Research Motivation

Geopolymerization is the process of alkali activation of alumino-silicates in raw materials to make construction materials. Industrial wastes like fly-ash and blast furnace slag and mining industry wastes such as mine tailings in form of crushed rocks are among the commonly used raw material for production of geopolymers. Mine tailings are the waste product of mining industry in the form of the residual material after extracting the valuable minerals from the ore. Mine tailings may consist of crushed rock, water, trace quantities of metals and additives used in mineral processing. Geopolymerized mine tailings can have a wide range of applications in the building and construction industry as they can partially or entirely replace the Portland Cement Concrete. Use of mine tailings for production of geopolymerized concrete will not only provide an eco-friendly construction material to the construction industry but it will also help the mining industry to dispose of the waste material in a cost-effective manner. Cement industry shares 8% of Carbon dioxide emission with a global production of 4.1 billion metric tons per annum, which has huge environmental consequences (Andrew, 2019; U.S. Geological Survey, 2020). Therefore, the use of geopolymerized materials as alternate cementitious material will help in reducing the negative environmental impacts.

Mine tailings are stored in mine tailings storage facilities/tailing dams near the quarry site/mine site. These tailing dams are similar to the hydro dams with the difference of storing both water and solid material. In the past, many tailing dams experienced failures, resulting in significant property damage, environmental contamination, and even fatalities. The frequency of
these failure incidents has been much higher in North America as compared to the rest of the world (Lyu et al., 2019). As an alternative approach to the storage, the mining industry can utilize innovative measures to extract more efficiently the targeted valuable minerals and minimize the production of mine tailings. Alternatively, another favorable approach will be the reuse of mine tailings in construction industry. The least favorable option is the storage of the tailings in a dam (Lottermoser, 2011). For its use in the construction industry, the material should be able to provide sufficient strength to withstand different types of loadings. Geopolymerization of mine tailings can enhance the strength of these mine tailings and convert them into cementitious materials.

Glukhovsky was the first to come up with an idea of alkali activated cements or “Soil Cements” back in 1957 (Krivenko, 2017). The term “Geopolymer” was first used by Davidovits during “Fourth Annual Pacific Technical Conference and Technical Display” conference organized by “Society of Plastic Engineers” in 1979 (Davidovits 1979; Davidovits 1991). Since the development of the idea, extensive research has been done on different types of geopolymers. Industrial wastes like fly ash and ground granulated blast-furnace Slag (GGBS) have been investigated to produce alkali activated cementitious materials (Palomo, Grutzeck, & Blanco, 1999; Ryu, Lee, Koh, & Chung, 2013). In literature alkali activation of mine tailings from different mine sources can be found (Ahmari, Parameswaran, & Zhang, 2015; Manjarrez & Zhang, 2018; Huang, Feng, An, & Zhang, 2020).

The process of geopolymerization is typically based on reaction between alumino-silicates and concentrated alkali solution at room or higher temperature, which leads to formation
of “Geopolymers” or “inorganic polymers.” The mechanical strength of these materials is highly dependent upon the Silicon/ Aluminum ratio (Si/Al ratio) present in the material (Duxson, et al. 2007).

The characterization of the mechanical behavior of the geopolymerized mine tailings is important for their use as an alternative construction material. The compressive strength of geopolymer has been extensively studied by many researchers; however, the tensile strength remains largely unexplored. Understanding of tensile behavior and fracture mechanics of geopolymerized tailings is critical and established techniques by the rock mechanics community such as the direct and indirect tensile tests can be utilized for evaluation of the material’s behavior. The direct tensile test is inherently complex but the indirect test methods are simple and reliable. Different indirect tensile test methods such as splitting tensile strength, three-point and four-point bending of beam, and semi-circular bend test have commonly been utilized in the rock mechanics field (Aliha, 2013; Perras and Diederichs, 2014).

Splitting tensile strength test is also known as Brazilian indirect tensile strength test or diametrical compression test. Reportedly, two researchers were credited with their independent work and development of this method (Akazawa, 1943; Carneiro, 1943). The diametrical compressive load causes the concentration of tensile stresses at the center of the circular disc or cylindrical specimen, causing the specimen to fail in tension. This method is extensively used for determining the tensile strength of rock and concrete specimens.

In three-point bending and four-point bending of beam test, the beam is supported at two points at the bottom and concentrated load is applied on top of beam at one point and two point
respectively. The upper part of the beam experiences compressive stresses and lower part experiences tensile stresses. This method provides flexural tensile strength of the material. The other method to calculate the flexural tensile strength of the specimen is semi-circular bend test. In semi-circular bend test, the semi-circular specimen is supported at two locations at the bottom and load is applied at the top. The specimen can have a notch at the bottom with variable length to ligament ratio.

While characterizing the mechanical behavior of the material researchers have also utilized digital image correlation (DIC) technique (e.g., Sutton et al., 2007; Lin and Labuz, 2013; Hedayat et al., 2014; Sgambitterra). The DIC techniques helps in obtaining full field strain measurement in a more convenient and efficient manner as compared to the physical strain gauges. In DIC technique, the displacement field is generated by comparing the reference or undeformed image to the target or deformed image. The stochastic speckle pattern on the surface of the specimen facilitates the correlating algorithms to match the area of interest in reference and target image.

In this study, the Brazilian indirect tensile strength test and semi-circular beam test along with Digital image correlation (DIC) technique were used to characterize the mechanical behavior of the alkali activated mine tailing concrete. The strain profile on the disk surfaces will be captured using the DIC correlation software and the critical tensile strain levels associated with the tensile fracture initiation will be identified. In addition, the fracture propagation velocities for different molar concentrations will also be evaluated. The strength development in the geopolymers depend on the percentage of alumina and silicates in the raw mine tailings.
Other factors, which can control the strength of geopolymerized concrete, are type and molar concentration of alkaline solution, the water to binder ratio, the curing temperature and age.

1.2 Research Objectives

The main objectives of this study are appended below:

- Evaluate the tensile strength of geopolymers. Quasi-brittle materials like geopolymers are weak in tensile strength as compare to their compressive strength therefore characterization of tensile strength is important. Brazilian indirect tensile strength method and semi-circular bend test method will be used to ascertain the tensile strength of the material.

- Identify critical strain levels for crack initiations. Crack initiates well below the strength of the material and may not be visible to the naked eye. DIC technique will be used to get the full field strain at the surface of the specimen to identify the critical strain levels

- Determine the mode I fracture toughness of the geopolymers. Fracture toughness is the resistance of the material to failure by fracturing. Occurrence of mode I failure is more frequent as compare to other modes. Semi-circular bend test using notched specimen will be carried out to estimate the mode I fracture toughness of the material.

- Ascertain critical strain energy release rate. Critical strain energy release rate is based on the energy-balanced approach. Critical strain energy release rate can be estimated indirectly using fracture toughness of the material.
To achieve above-mentioned objectives, the following tasks have been performed:

- Casting and curing of specimens in a repeated and systematic way in the laboratory
- Developing the experimental setup for fracture experiments on geopolymers
- Conducting the Brazilian indirect tensile strength tests
- Conducting the semi-circular bend (SCB) tests
- Analysis of experimental results.

1.3 Thesis Outline

This thesis is organized in to six chapters. Chapter 1 introduces the research topic and presents the motivation for this study. Research objectives and research tasks are also enumerated in this chapter.

Chapter 2 covers the relevant literature related to this study. It briefly describes the past research in geopolymer concrete. It also covers the testing methods used in literature like Brazilian indirect tensile test and semi-circular bend test along with digital image correlation technique. The basic concepts of fracture mechanics relevant to this study were also summarized in this chapter.

Chapter 3 deals with geotechnical characterization of the raw tailings, preparation of the specimens and experimental set up for Brazilian indirect tensile test and semi-circular bend test.
Chapter 4 summarizes the results obtained from Brazilian indirect tensile test along with DIC technique. It presents the splitting tensile strength of the material, critical strain levels for crack initiation and estimation of elastic properties of the material from DIC analysis.

Chapter 5 presents the results obtained from semi-circular bend test along with the DIC technique. Fracture toughness, critical strain energy release rate, crack mouth opening displacement and critical strain levels for crack initiation were discussed in this chapter.

Chapter 6 concludes this study by summarizing the main conclusions and provides the recommendations for future work.
CHAPTER 2
REVIEW OF THE RELEVANT LITERATURE

2.1 Geopolymerization/Alkali Activation of the Materials

Geopolymerization is the process of alkali activation of alumino-silicates in raw materials to make construction materials. Industrial wastes like fly ash and blast furnace slag and mining industry wastes such as mine tailings in form of crushed rocks are commonly used raw materials for production of geopolymers. The use of mine tailings to produce geopolymer concrete will not only provide an eco-friendly construction material but also help the mining industry to repurpose the waste material in a cost-effective manner.

Glukhovsky was among the first researchers who reported the alkali activated cements or “Soil Cements” back in 1957 (Krivenko, 2017), and suggested a model to explain the geopolymerization process. This model consists of three stages namely “destruction-coagulation stage”, “coagulation-condensation stage” and “condensation-crystallization stage”. These different stages can occur either simultaneously or in a linear fashion.

Initially, the solid alumino-silicates dissolves in alkali solution and produces aluminates and silicates. An increase in the alkali concentration will increase the pH of the system. This higher pH value creates a conducive environment for the dissolution of alumino-silicates into tetrahedral units of $SiO_4$ and $AlO_4^-$. Aluminum cation has three electrons available for chemical bond but due to bonding with four oxygen atoms, it has an overall negative charge ($AlO_4^-$). This negative charge on $AlO_4^-$ is not stable and needs to be balanced by a cation like Na$^+$ in the system. During coagulation-condensation stage, the aluminates and silicates come closer to each other
and form a gel. Water molecules are extracted during this phase, although water molecules are not present in final form but necessary in the reaction of the system. Rearrangement of the molecules leads to the formation of a ring structure, which is three-dimensional in nature, known as the polymerization process. Oxygen atoms facilitate the connection of Silicon and Aluminum ions and the sharing of oxygen atoms among Si and Al leads to the formation of “sialate (Si – O – Al)”.

Depending upon the ratio of Si to Al, these sialates are classified as “Polysialate”, “polysialate siloxo”, and “polysialate disiloxo”. The term “Geopolymer” was first used by Davidovits during “Fourth Annual Pacific Technical Conference and Technical Display” conference organized by “Society of Plastic Engineers” in 1979 (Davidovits, 1979; Davidovits, 1991). Alumino-silicates play a vital role in the formation of geopolymers upon reaction with the alkali solution. This process can take place at room temperature or higher temperature if higher strength is required at an early stage. These geopolymers are also sometimes referred as inorganic polymers (Singh et al., 2015). Zhang et al., (2021) reported the following equation for the chemical reaction of the geopolymerized material in the presence of NaOH:

\[
(Si_2O_5, Al_2O_2)_n + n H_2 O \rightarrow n(OH)_3 - Si - O - Al - (OH)_3 \quad (2-1)
\]

\[
n(OH)_3 - Si - O - Al - (OH)_3 \rightarrow (-Si - O - Al - O)_n + 3n H_2 O \quad (2-2)
\]

The performance of geopolymers is comparable to ordinary Portland cement with an added advantage to limited Greenhouse gas emissions. Silicon to Aluminum (Si/Al) ratio is one of the fundamental parameters responsible for the mechanical strength gain for the geopolymers.
(Duxson et al., 2007). Other factors include temperature, forming pressure, water content, type and concentration of alkali activating agent.

2.1.1 Use of Industrial Wastes for Geopolymerization

Industrial wastes like those that fly ash and ground granulated blast-furnace slag (GGBS or slag in short) have been investigated to produce alkali activated cementitious materials (Palomo et al., 1999). Ryu et al., (2013) studied the mechanical properties of fly ash based geopolymerized concrete by activating the fly ash using sodium hydroxide (NaOH) at 6, 9 and 12M concentrations along with a percentage of sodium silicates. Uniaxial compressive strength of 50 mm cubic specimens were obtained at 1, 3, 7, 28, 56 and 91 days of curing age. To accelerate the geopolymerization process, all the specimens were heated at 60°C for 24 hours. Five specimens were tested for each curing age and composition. Compressive strength of 12M specimens reached 46 MPa after 56 days of curing. Splitting tensile strength test of fly-ash based geopolymers was also conducted using cylindrical specimens with diameter of 100 mm and thickness of 200 mm. These specimens were cured at room temperature for the initial 24 hours and then placed in oven at 60°C for 48 hours and thereafter at room temperature for 3, 14 and 28 days. Three specimens were tested for each curing age of the specimen. Based on experimental results, a relationship between the compressive and splitting tensile strength was proposed (i.e. \( f_{sp} = 0.17 \times (f'_{c})^{3/2} \), where \( f_{sp} \) is splitting tensile strength and \( f'_{c} \) is the compressive strength). It was concluded that splitting tensile strength for geopolymerized fly ash lies within 8% of the compressive strength.
Alkali-activation of fly ash and slag-based concrete at room temperature were investigated for materials rich in calcium oxide (CaO) (e.g., Lee and Lee, 2013). Calcium oxide (CaO) contributes to the development of strength during alkaline activation. Lee and Lee, (2013) selected the slag in which 56% CaO was present and replaced the percentage of fly ash with slag. 4, 6 and 8 M concentration of NaOH was selected as an alkali activation agent along with Na₂SiO₃. Three cylindrical specimens with the size of 100 x 200 mm were prepared for compressive strength test and splitting tensile test. Compressive strength specimens were tested after 3, 7, 14, 28 and 56 days of curing. The splitting tensile strength test was also conducted on specimens prepared by geopolymerization of fly-ash with slag based specimens. The specimens were cured at 20°C and 60% humidity level. The specimens were tested after 28 days of curing age. Phosphoric acid (H₃PO₄) was used as a retarding agent to gain time for moulding the specimens however; H₃PO₄ reduced the compressive strength of the specimen. They were able to achieve the compressive strength of 45 MPa for 25% slag replacement in fly ash. They proposed a relationship between the splitting tensile strength and compressive strength as $f_{ct} = 0.45 \times \sqrt{f'_c}$ MPa, where $f_{ct}$ is the splitting tensile strength and $f'_c$ is the compressive strength of alkali activated fly ash/slag concrete. The maximum splitting tensile strength of 3 MPa was achieved against the compressive strength of 40 MPa.

Pan et al., (2011) studied the fracture behavior of fly ash based geopolymers; and drew a comparison between geopolymerized concrete and ordinary Portland cement concrete and found a more brittle response from geopolymerized concrete. Almeida et al., (2018) studied the impact of fibre reinforcement on the behaviour of alkali-activated fly ash. They used sisal fibre with
length of 13 mm and 50 mm. The percentage of fibres were varied from 0-1% of the weight of fly ash. The highest unconfined compressive strength of almost 9 MPa was obtained for the 0.2% of the fibres having a length of 13 mm. By increasing the percentage of fibre content, the tensile strength of the specimen also improved. Hamidi et al., (2020) studied the geometry effect on compressive and tensile strength of heavy weight geopolymerized concrete (HWGC) based on fly ash. To measure splitting tensile strength, they used 100 × 200 and 150 × 300 mm cylindrical specimen. Compressive strength test was also conducted on the same size of cylindrical specimens. To study the flexural behaviour of the material they used 100 x 100 x 400 mm specimens. The percentage of magnetite heavy weight aggregate was kept at 0, 50 and 100 % in different batches of the specimens. Solution of sodium hydroxide (NaOH) and sodium silicates (Na$_2$SiO$_3$) were used as alkali activating agent. Water to binder ratio was 12% and alkali activation solution to binder ratio was kept at 40%. 14M concentration of NaOH was mixed with 2.5 times of Na$_2$SiO$_3$. Specimens were tested after 28 days curing with a loading rate of 1kN/sec. They reported decrease in the tensile strength with the increase in specimen size; however, with the increase in percentage of heavy weight aggregate the tensile strength of specimen increased.

2.1.2 Effect of the Curing Environment and Temperature

The curing temperature plays an important role in the geopolymerization in the form of acceleration of the gel formation, which is critical for the strength gain at an early stage (Singh et al., 2015). Palomo et al., (1999) reported specimens with strength as high as 60 MPa compressive strength by alkali activation of fly ash using a solution of NaOH and Sodium Silicate (Na$_2$SiO$_3$) after 5 hours of curing at 85°C. To study the impact of temperature on geopolymerization, they used two curing temperatures of 65 °C and 85 °C and specimens were
cured for 2, 5 and 24 hours before testing the mechanical strength as well as mineralogy and microstructure through X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscope/energy dispersive X-ray spectroscopy (SEM/EDS). Strength of specimens cured at 85°C was higher as compare to the specimens cured at 65°C.

Ahmari et al., (2011) produced bricks for construction industry using geopolymerized copper mine tailings. 10 M to 15 M concentration of NaOH was added to the mine tailings with water content of 8 to 18% of the mine tailings. Forming pressure of 0 to 35 MPa was applied during preparation of bricks, which squeezed out the excess water content from the matrix. The range of curing temperature was kept at 60°C to 120°C for 7 days. Loading rate of the specimen was kept constant at 0.1 mm/min. Unconfined compressive strength of the specimens were found to have increased with the increase in temperature from 60 °C to 90 °C and then there was decrease in the strength for further increase in curing temperature till 120 °C. They argued that this trend is attributed to the gel formation during geopolymerization process. If formation of gel is too quick, it can inhibit the further dissolution of alumina and silica. Therefore, the optimum temperature was suggested to be 90 °C.

2.1.3 Effect of Molar Concentration of the Alkali Activation Agent

The molar concentration of alkali agent plays an important role on strength development in the material. By increasing the molar concentration, the strength of the material increases and there is optimum molar concentration where the achieved strength is maximum. Ahmari et al., (2012a) studied the effect of curing temperature and alkali activator type as well as concentration of alkali agent on the compressive strength of geopolymerized copper mine tailings. They
investigated the effect of alkali concentration by choosing 5, 10 and 15 M concentrations of NaOH. To study the impact of temperature they selected four temperatures of 60, 75, 90 and 120 °C. They found out the optimum curing temperature for 5M and 10M concentration of NaOH specimens to be 75°C and for 15M concentration the optimum temperature was 90°C. Three cylindrical specimens were uniaxially loaded for each condition at the loading rate of 0.1mm/min. They achieved the maximum compressive strength of 20 MP for a 15M specimen cured at 90 °C. The optimum SiO₂/Na₂O ratio for their specimens was 1.2. Ahmari et al., (2015) investigated the geopolymerization of blended mine tailings along with low calcium furnace slag from mining industry. Geopolymerized mine tailings without blast furnace slag was able to achieve the compressive strength of 25 MPa and by adding 50% blast furnace slag the strength of specimen reached to 65 MPa. Addition of blast furnace slag also reduced the requirement of water content consequently 25% reduction in use of NaOH. In this study, the specimens were cured at 90 °C for 7 days and 15 M concentration of NaOH was utilized. Manjarrez and Zhang (2018) studied the effect of molar concentration of NaOH on the uniaxial compressive strength of the geopolymerized mine tailings. They varied the molar concentration of NaOH from 0 to 11%. For specimen with 19% of the water content they obtained the maximum strength of specimen at 11M concentration of the NaOH. By increasing the molar concentration, the strength increased and reached to the peak value at 11M concentration and then the strength decreased on further increase in molar concentration.

2.1.4 Use of Mine Tailings for Geopolymerization

In addition to fly ash and slag, mining wastes were reported to be used for successful geopolymerization (e.g., Ahmari et al., 2011, 2015; Manjarrez and Zhang 2018; Zhang et al.,
Ahmari et al., (2011) produced bricks for construction industry using geopolymerized copper mine tailings. 10 M to 15 M concentration of NaOH was added to the mine tailings with water content of 8 to 18% of the mine tailings. Forming pressure of 0 to 35 MPa was applied during preparation of bricks, which squeezed out the excess water content from the matrix. Effect of water content on the strength of specimen was also investigated and it was found that the strength of specimen increased with the increase in water content to certain level and thereafter the strength decreased. The increase in percentage of water content means more NaOH is available in the solution, thereby, the geopolymerization process is facilitated and more strength is achieved. When specimens with high water content are prepared, the process may leads to the formation of large pores and therefore lower strength. Ahmari et al., (2011) achieved the highest strength of 33 MPa for the specimen with moisture content of 18% and 15M concentration of NaOH. They also confirmed the Geopolymerization process using SEM and XRD methods.

Manjarrez and Zhang (2018) explored the possibility of utilizing geopolymerized mine tailings as road base construction material. The molar concentration and water content were reported as two main influential factors on the unconfined compressive strength. In their study, they used 0 to 11M concentration of NaOH, 11 to 19% moisture content and slightly elevated curing temperature of 35 °C. Cylindrical specimens measuring 30 mm in diameter and 70 mm in height were prepared to ascertain compressive strength of the material at the loading rate of 0.1 mm/min. They found an optimum moisture content of 15% and an optimum Na/Al ratio of 1. Compressive strength of specimens with the curing age of 7 days met the requirements for utilization in road construction industry however; they recommended the leaching test for their
material. Longos et al., (2020) conducted research on geopolymerization of mine waste added with fly ash. For alkali activation, they used sodium hydroxide and sodium silicate. By optimization, they were able to achieve the compressive strength as high as 36 MPa by using 50% mine waste and 50% fly ash with sodium hydroxide to sodium silicate ratio of 0.5. They found that the geopolymerized materials fulfilled the strength requirements for concrete structures and light traffic pavers.

Zhang et al., (2020a) investigated the effect of cyclic loading on compressive strength of geopolymerized mine tailings. They explored the mine tailings activated by NaOH with different molar concentrations ranging from 6 to 8 M. 50 mm cubic specimen were tested after a curing age of 28 days. Each specimen was loaded at the displacement control rate of 0.21 mm/min and unloaded in a force control mode until 2.5 kN. Each specimen was subjected to 10 cycles of loading and unloading, and specimen were compressed to achieve a maximum of 5 % of the strain. The compressive strength of the specimen under cyclic loading increased with the increase in molar concentration of NaOH; this trend is similar to the response of specimen under monotonic loading. In their study, the specimen with 12 M concentration exhibited the stiffer response against the cyclic loading as compared to the specimens with lower concentration of NaOH. The elastic modulus of the specimen increased with the increase in molar concentration of NaOH. For 6 and 8 M concentration specimens, the elastic modulus decreased with each cyclic loading however for 10 and 12 M concentrations the elastic modulus remained rather constant.
Zhang et al., (2020b) studied the uniaxial compressive behavior of the geopolymerized mine tailings with the focus on fracture and failure process. They were successful in modelling the compressive behavior of the specimen using discrete element method (DEM) and validated the results with the experimental data. They used the 150 mm cubic specimen cured at 75 C for initial 24 hours and subsequently placed the specimen at room condition. They tested the specimen after 28 days of curing. They varied the molar concentration of NaOH from 0 to 10 M. For 10 M concentration, they reported the compressive strength as high as 30 MPa. They observed a diagonal fracture pattern for the specimen subjected to uniaxial compressive load. Zhang et al., (2021c) investigated the size effect on the compressive strength of the geopolymerized mine tailings similar to the current study. They used cubic specimen with size ranging from 30 mm to 80 mm. Water to tailing ratio was from 13 % to 20 % and used 10 M concentration of NaOH as an alkali activating agent. The loading was applied in a way to get constant strain rate of 0.0045 per minute for all types of specimens. They reported a decrease in compressive strength of specimen with the increase in the size of the specimen.

2.2 Brazilian Indirect Tensile Strength Test

Extensive studies of the geopolymers were performed to investigate the mechanical behaviors under uniaxial compression (e.g., Canakci et al., 2019; Langos Jr. et al., 2020; Zhang et al., 2020a). However, the tensile properties remained largely unexplored and Brazilian indirect tensile strength test is commonly performed for the determination of the tensile strength of brittle materials. The independent research work by two researchers (Akazawa 1943; and Carneiro 1943) helped in development of this indirect method of ascertaining tensile strength of the material. The diagonal loading causes tensile strain at the center of the specimen. As the material
is usually weak in tension, the failure of the specimen would be tensile in nature and corresponding load obtained at failure of the specimen is consider in calculating the tensile strength of the specimen. ASTM standard C496 provides the detailed procedure to conduct splitting tensile test for concrete specimen. ASTM D3967 gives the detailed procedure to conduct splitting tensile test on rocks. As per ASTM D3967, the specimen should have thickness to diameter ratio ranging from 0.2 to 0.75. ISRM (1978) suggests that the thickness of the specimen should be approximately equal to the radius of the specimen. The splitting tensile strength of the material can be calculated using the following relationship:

\[
\sigma_{\text{splitting}} = \frac{2P}{\pi Dt}
\]  

(2-3)

where, \(P\) is the load applied to the specimen, \(D\) is the diameter of specimen and \(t\) is the thickness of the specimen. The indirect tensile method over-estimates the tensile strength of the specimen; however, its value is lower than the flexural tensile strength (Darwin et al., 2016). Therefore, indirect tensile strength predicts the tensile strength of material more accurately as compare to flexural tensile test.

Splitting tensile test coupled with DIC technique was widely used by researchers in the field of rock mechanics and concrete. Stirling et al., (2012) investigated the effect of change in load platen configuration on the splitting tensile failure mechanism and strain distribution along the diameter of the sandstone specimen using DIC techniques. Three types of the loading platen included flat load platen causing a point load on specimen, circular load platen and flat load platen where the load was uniform on the flatten surface of the specimen. The specimen was loaded in a force control mode with the loading rate of 3.225 kN/min. For the DIC analysis, the
camera system was configured to get the frame rate of 20 frames per second. They found out a uniform loading at the center of the specimen where a flat platen was used to load a specimen whose ends near the platen were also flattened.

Li and Wong (2013) has carried out a detailed review of splitting tensile test for circular rock disc specimens. Different loading configurations were proposed to conduct the splitting tensile test of the specimen. The loading configuration has a large impact on the stress concentration along the diameter of the specimen. Among these loading configuration includes flat platen, platen with a steel rod, platen with a cushion (mostly wooden cushion), circular platen which covers a large portion around the periphery of the specimen. The crack can initiate near the platen in the case of flat steel platens (Hudson et al., 1972; Li and Wong, 2013).

Sgambittera et al., (2017) studied the tensile behavior of geopolymer mortar and drew a comparison between ASTM standard, ISRM recommended method and proposed a flattened Brazilian disk method. They also used the DIC technique to get full field strain data. To get the stress contours along the surface of the specimen they proposed the following equations (Sgambittera et al., 2017):

\[
\sigma_x = \frac{2P}{\pi t} \left[ \frac{(D-y)x^2}{((\frac{D}{2}-y)^2+x^2)^2} + \frac{(D+y)x^2}{((\frac{D}{2}+y)^2+x^2)^2} - \frac{1}{D} \right]
\]

\[
\sigma_y = \frac{2P}{\pi t} \left[ \frac{(D-y)^3}{((\frac{D}{2}-y)^2+x^2)^2} + \frac{(D+y)^3}{((\frac{D}{2}+y)^2+x^2)^2} - \frac{1}{D} \right]
\]
\( \tau_{xy} = \frac{2P}{\pi t} \left[ \frac{\left( \frac{D}{2} - y \right)^2 x}{\left( \left( \frac{D}{2} - y \right)^2 + x^2 \right)^2} + \frac{\left( \frac{D}{2} + y \right)^2 x}{\left( \left( \frac{D}{2} + y \right)^2 + x^2 \right)^2} - \frac{1}{D} \right] \)  

(2-6)

where, \( P \) is the tensile load applied to the specimen, \( D \) is the diameter of specimen and \( t \) is the thickness of the specimen. \( x \) and \( y \) represent the Cartesian coordinates of the point at the surface of the specimen based on the origin at the centre of the specimen. It is pertinent to mention that at the centre of the specimen (i.e., \( x = y = 0 \)), the equation (2-4) will be the same as the equation (2-3).

Aliabadian et al., (2019) analyzed the crack development in sandstone using Brazilian indirect tensile method along with DIC to measure the crack initiation and tensile strain concentration. Disc specimens with 30 mm diameter and 15 mm thickness were used in their study. Flat loading platen as well as loading platen with wooden cushion were used to load the specimen. The loading rate of the specimen was 0.03 mm/min for flat platen loading configuration and 0.3 mm/min for platens with wooden cushion. The stress concentration portion at the center of the specimen became narrow for the case with wooden cushion, as compare to the loading configuration with flat platen. The strain values from DIC and strain gauges had a good match, which confirmed the accuracy of DIC method. The horizontal strain values for different points along the crack were plotted with time. The variation of horizontal strain with time provided an insight to the behavior of material at different stages of loadings. At early stage, the behavior was elastic in nature followed by fracture process zone formation, and subsequently the onset of macro cracks leads to the failure. The gradient of the horizontal strain along the \( x \)-axis of the specimen at different stages helped in identification of crack initiation stage. Their
study shows higher sensitivity of subset size as compare to the step size in DIC correlation. The crack initiation point was at the center of specimen in horizontal direction but in vertical direction, the location of crack initiation is not at the center rather at the mid distance between the center of specimen and the platens. They got the peak load of 2.5 kN for their specimens.

Belrhibi et al., (2017) conducted Brazilian indirect tensile test coupled with DIC on refractory materials. They carried out the test in a displacement control mode with the loading rate of 0.05 mm/min. The specimen was 50 mm in diameter and 10 mm in thickness. They calculated the fracture energy of the material based on the dissipated energy per unit surface area of the crack. The dissipated energy is the area under the load displacement curve of the material. Initially the material behaved in elastic manner, so the release of energy was recoverable. However, after the elastic region, there was energy dissipation, which was not recoverable. They argued that this dissipation of the energy controls the behavior of the material.

Similarly, Li et al., (2020) adopted DIC method to analyze the effect of loading platens on tensile failure of sandstone in splitting tensile test. They registered higher strength of the specimen loaded with circular platen as compared to the flat platen. They drew comparison for different types of rocks (e.g., marble, sandstone, granite and Basalt with tensile to compressive strength ratio of 0.05, 0.063, 0.075 and 0.1 respectively). It was observed that the crack initiation point is not always at the center of the specimen and it could be off from the center specifically for the specimen with higher tensile to compressive strength ratio.
2.3 Semi Circular Bend (SCB) Test

Semi-circular bend test is used to ascertain the tensile strength of the material. In literature, the semi-circular bend test with a notched specimen has been used to estimate the fracture toughness of the material. (e.g., Kuruppu and Chong, 2012; Kataoka et al., 2017). Chong and Kuruppu, (1984) first introduced the SCB to ascertain the flexural strength and fracture toughness of brittle materials like rocks, concrete etc. (Kuruppu and Chong, 2012). SCB method has also been widely used in characterizing the fracture behavior of the asphalt concrete (e.g., Elseifi et al., 2012; ASTM D8044). Lim et al., (1993) proposed a relationship to find mode I fracture toughness using SCB test:

\[ K_I = \frac{F\sqrt{\pi a}}{2RT} Y_I \] (2-7)

where \( K_I \) is the stress intensity factor, \( F \) is the load, \( a \) is the notch depth, \( R \) represents the radius of the specimen, \( T \) denotes the thickness of the specimen, and \( Y_I \) is the dimensionless stress intensity factor that can be calculated based on the numerical analysis results (Lim et al., 1993; Kuruppu and Chong, 2012):

\[ Y_I = \frac{S}{D} \left[ 2.91 + 54.39 \left( \frac{a}{R} \right) - 391.4 \left( \frac{a}{R} \right)^2 + 1210.6 \left( \frac{a}{R} \right)^3 - 1650 \left( \frac{a}{R} \right)^4 + 875.9 \left( \frac{a}{R} \right)^5 \right] \] (2-8)

where \( S \) is the span ratio and \( D \) is the diameter of the specimen. SCB test can be used to either ascertain mode I fracture toughness or the mixed mode fracture toughness of the material based on the angle of the notch in the specimen (Lim et al., 1994; Lin et al., 2020).
Zhang et al., (2021a) investigated the fracture toughness of geopolymerized mine tailings using notched semi-circular bend test. They used the raw tailings from a gold mine in San Juan de Chorunga, Arequipa, Peru. The diameter of the specimen was 75 mm, and the thickness of the specimen was 47 mm. They varied the ratio of length of the specimen to the radius of the specimen (a/R) from 0.2 to 0.4. Specimens were loaded at a constant rate of 0.05 mm/min. They reported an average tensile strength of 1.5 MPa for un-notched semi-circular specimen. Fracture toughness of their specimen was around 450 kPa.\( \sqrt{m} \). Using DIC, the estimate length of fracture process zone ahead of the tip of the notch was 8 mm and crack mouth opening displacement was approximately 23 µm.

Zhang et al., (2021b) investigated the compacted mine tailings without adding alkali agent. Specimens with a diameter of 75 mm and thickness of 40 mm were prepared by tamping the mine tailings and optimum moisture content of 18 % was used. The length of the notch to the radius of the specimen (a/R) ratios from 0 to 5 were selected in their study. The loading rate of the specimens was 0.1 mm / min. Fracture toughness of the specimen were calculated based on effective length of the notch considering the plastic deformation in fracture process zone. Without considering the effect of fracture process zone, the calculated fracture toughness would be underestimated. The Poisson’s ratio was calculated based on DIC analysis by converting the Green-Lagrange strains into Engineering strain and taking the ratio of lateral strain to the axial strain, which gave a Poisson’s ratio of 0.286. The Elastic Modulus was calculated based on stress strain diagram of the specimen. These elastic properties were used in William’s series to calculate the stress intensity factors. They compared the fracture toughness values for the specimens prepared by compacting the mine tailings without any addition of alkali activation.
agent or geopolymerization. For comparison they used different methods i.e., (1) using William’s series, (2) method proposed by Kuruppu et al., (2012), and (3) method based on DIC results. Thereby, they found that Kuruppu et al., (2012) method underestimated the fracture toughness of the material as it is based on linear elastic behavior and in reality; there are plastic deformations in form of fracture process zone ahead of the tip of the notch.

2.4 Review of Relevant Concepts from Fracture Mechanics

2.4.1 Energy Balanced Approach

Fracture mechanics has evolved over the past century and many models have been set forth to explain the effect of crack on ductile and brittle materials. Inglis (1913) is considered as the pioneer in this field as he studied stress concentration along the elliptical holes. However, his mathematical modeling had an issue: the stresses near the tip of sharp crack approached infinity, which is physically not possible as it was based on elastic assumptions and in reality, there are local plastic deformations. Griffith (1921) published his well-known theory about fracture mechanics in brittle materials. His theory was based on surface energy of the material, which explained the reason for reduction in strength of the material in presence of discontinuities or flaws as compared to the theoretical strength and often this reduction in strength is an order of the magnitude. He assumed an infinite plate with an edge crack of length $a$. Stress was applied at far end away from the crack and the stress was perpendicular to the plane of the crack. Based on release of total energy which is related to unloading of the portion of the specimen due to crack growth and surface energy, $\gamma$ which is absorbed by the formation of new crack surfaces, the failure stress can be predicted for the critical crack length:
\[ \sigma_f = \sqrt{\frac{2Ey}{\pi a}} \]  

(2-9)

where \( \sigma_f \) is the stress at failure, \( E \) is the elastic modulus of the material, \( \gamma \) is the surface energy of the material, and \( a \) is the length of the crack. As Griffith’s equation was developed based on brittle material (glass), Irwin (1948) and Orowan (1949) extended his work for the ductile material by incorporating the energy required for plastic deformation of the ductile material and proposed a general form of the equation, as follows:

\[ \sigma_f = \sqrt{\frac{2EG}{\pi a}} \]  

(2-10)

where \( G \) is the critical strain energy release rate. Similarly, the remaining variables are same as used in equation (2-9) (i.e., \( \sigma_f \) is the stress at failure, \( E \) is the elastic modulus of the material, \( \gamma \) is the surface energy of the material, and \( a \) is the length of the crack). The critical length of the crack for a specimen is independent of the size of the specimen. The smaller specimen cannot accommodate large cracks in it therefore the length of the crack may not be able to reach to a critical value; this phenomenon explains the fact that small specimens are typically stronger as compare to the large specimen.

The critical strain energy release rate can be calculated using different numerical and experimental methods. Compliance method is one of the methods used to calculate the critical strain energy release rate. Compliance, \( C \) itself is the reciprocal of the stiffness parameter, \( E \). It means the stiffer materials will have less compliance under the given load. The following relationship is used to calculate the strain energy release rate:
\[
G = \frac{\partial U}{\partial a} = \frac{1}{2} P^2 \frac{\partial C}{\partial a}
\] (2-11)

Westergaard (1939) proposed the relationship to estimate the stresses near the tip of the crack. An infinite plate with a crack at the center with the length 2a was considered and then subjected to the isotropic tension of \(\sigma_\infty\). He assumed a function based on complex numbers (i.e., \(z = x + iy\)) for Airy stress function \(\phi\) and solved the bi-harmonic partial differential equation \(\left(\frac{\partial^4 \phi}{\partial x^4} + 2 \frac{\partial^4 \phi}{\partial x^2 \partial y^2} + \frac{\partial^4 \phi}{\partial y^4}\right) = 0\). The solution of the partial differential equation lead to a relationship that provides an estimate for the stresses at the crack tip:

\[
\sigma_{xx} = \sigma_{yy} = \frac{\sigma_\infty}{\sqrt{1-(\frac{x}{a})^2}}
\] (2-12)

where \(a\) is the length of the crack, \(\sigma_\infty\) is the applied biaxial stress and \(x\) is the arbitrary distance away the crack tip. If \(x\) approaches to zero (i.e., at the crack tip), the stresses reaches to infinity. Here, Airy stress function (\(\phi\)) provides a convenient way to solve for the stresses. Only one unknown \(\phi\) needs to be solved using a bi-harmonic equation. If Airy stress function is not used then multiple equations must be solved to ascertain the multiple unknowns to get the stress state of the body.

### 2.4.2 Stress Intensity Factor Approach

Stress intensity factor is an alternate approach to ascertain the impact of crack on the strength of the material. \(K\) denotes the stress intensity factor and it depends on the configuration of crack and loading. If the loading is applied normal to the plane of the crack, it is considered as mode I stress intensity factor, \(K_I\); also known as opening mode. If the loading is applied parallel
to the plane of the crack, which causes in-plane shear and direction of applied load is in the
direction of crack tip then it will be considered as mode II stress intensity factor, $K_{II}$; also called
sliding mode. Similarly if the applied load causes in-plane shear and direction of loading is
perpendicular to the tip of the crack then it is considered as mode III stress intensity factor, $K_{III}$;
also known as tearing mode. The loading of the specimen and crack orientation will lead to one
or combination of these modes of failures. Among all these modes, mode I is considered
important and gained much attention by the researchers as mode I is the most frequently
encountered fracture mode.

Irwin (1957) is credited with the development of stress intensity factor term. He used the
polar coordinate system and assumed a function (i.e., $z = a + re^{i\theta}$) and solved the Airy stress
function equation in a similar way as Westergaard (1939) did. He came up with the approximate
solution for the stresses near the crack tip:

$$
\sigma_{yy} = \frac{\sigma_{\infty} \sqrt{\pi a}}{\sqrt{2\pi r}} \cos \frac{\theta}{2} \left(1 + \sin \frac{\theta}{2} \sin \frac{3\theta}{2}\right)
$$

(2-13)

For, $\theta = 0^\circ$

$$
\sigma_{yy} = \frac{\sigma_{\infty} \sqrt{\pi a}}{\sqrt{2\pi r}}
$$

(2-14)

In equation (2-14), $K = \sigma_{\infty} \sqrt{\pi a}$, is the stress intensity factor which is dependent on the applied
stress on the body and crack length, $a$. In comparison to Westergaard exact solution (equation (2-12)),
equation (2-13) developed by Irwin (1957) provides a good approximation of stresses near
the crack’s tip; however, away from crack’s tip, the approximation is not as accurate as the exact
solution provided by Westergaard (1939). Note that the stress state at the crack tip controls the material response to the crack growth so the Irwin (1957)’s proposed approximate solution serves the intended purpose. In addition, it provides an easy understanding of physical phenomenon in a mathematical setting.

2.4.3 Relationship between Stress Intensity Factor and Energy Release Rate

The stress intensity factor, $K$ can be related to Griffith’s energy release rate, $G = \frac{\sigma^2 \pi a}{E}$:

For plane stress conditions:

$$G = \frac{K^2}{E}$$

(2-15)

For plane strain conditions:

$$G = \frac{K^2}{E'}$$

(2-16)

where, $E' = \frac{E}{1 - \nu^2}$

2.4.4 Effect of Plane Stress and Plane Strain Conditions on Stress Intensity Factor

For plane stress conditions, the fracture process zone is larger as compared to the plane strain conditions. For a thick specimen, the bulk of the portion of the specimen is under plane strain conditions; however, at the outer surface the plane stress conditions prevails at the material surface. It can also be argued that for plane strain condition, there exist stress triality at the center near the crack tip, thereby the shear stress which is the difference between the major and minor principle stress would also be lower. In terms of strains, there is a lateral constraint for the
material which reduces the deformation and that’s the reason of smaller plastic process zone in plane strain conditions. As the fracture process zone is larger in case of plane stress condition, it can accommodate more strain energy by distributing it over the larger area. Therefore, the stress intensity factor for plane stress is higher than the plane strain conditions. For design purposes the plane strain stress intensity factor is used which is the critical toughness of the material independent of the thickness of the material. This critical stress intensity factor is also referred as fracture toughness. Fracture toughness is the measure to describe the ability of material to resist the failure (i.e., unstable crack growth in fracture mechanics). Fracture toughness is usually expressed in the units of stress and crack length as $MPa\sqrt{m}$. Fracture toughness increases with the increase in strain rate (Mahanta et al., 2017).

2.4.5 Calculating Stress Intensity Factor using Williams’ Series

Williams (1957) proposed an over deterministic approach by using an infinite series to ascertain the stress intensity factor at the crack tip. The expansion series is based on displacements, shear modulus, G, and polar coordinates of the selected points in the near vicinity of crack tip. In this series, the values of displacements can be obtained from a finite element analysis or DIC. The series is used to obtain both mode I and mode II stress intensity factors.

\[
\begin{align*}
ux(r, \theta) &= \sum_{n=1}^{N} A_{in} r^n \left\{ \kappa \cos \frac{n}{2} \theta - \frac{n}{2} \cos \left( \frac{n}{2} - 2 \right) \theta + \frac{n^2 + (-1)^n}{2} \cos \frac{n}{2} \theta \right\} - \\
&\quad \sum_{n=1}^{N} A_{in} r^n \left\{ \kappa \sin \frac{n}{2} \theta - \frac{n}{2} \sin \left( \frac{n}{2} - 2 \right) \theta + \frac{n^2 - (-1)^n}{2} \sin \frac{n}{2} \theta \right\} \\
uy(r, \theta) &= \sum_{n=1}^{N} A_{in} r^n \left\{ \kappa \sin \frac{n}{2} \theta - \frac{n}{2} \sin \left( \frac{n}{2} - 2 \right) \theta - \frac{n^2 + (-1)^n}{2} \sin \frac{n}{2} \theta \right\} - \\
&\quad \sum_{n=1}^{N} A_{in} r^n \left\{ \kappa \cos \frac{n}{2} \theta - \frac{n}{2} \cos \left( \frac{n}{2} - 2 \right) \theta + \frac{n^2 - (-1)^n}{2} \cos \frac{n}{2} \theta \right\}
\end{align*}
\]
where \( u_x, u_y \) are the \( x \) and \( y \)-displacements, \( G \) is the shear modulus, \( N \) is the number of the terms, \( \kappa \) is Kolosov’s constant that equals to \((3 - \nu)/(1 + \nu)\) for plane stress and \(3 - 4\nu\) for plane strain, \( \nu \) is the Poisson’s ratio. The \( r \) and \( \theta \) represents the polar coordinates of the selected nodes/points where displacement has been calculated. \( A_{I_n} \) and \( A_{II_n} \) are the coefficients of the Williams’ series and the series is solved to ascertain the values for these unknowns. The first two term of the series i.e., \( A_{I1} \) and \( A_{II1} \) are important as they lead to the calculation of mode I stress intensity factor, \( K_I \) and mode II stress intensity factor, \( K_{II} \) as shown below:

\[
A_{I1} = \frac{K_I}{\sqrt{2\pi}}, \quad A_{II1} = \frac{K_{II}}{\sqrt{2\pi}}
\]

\( A_{I1} \) and \( A_{II1} \) are the first terms in the Williams’ series given in equations (2-17) and (2-18). To predict the values for these two terms in other higher order terms are also included in the analysis. Normally in literature, the number of terms vary from four to ten. The second term \( A_{I2} \) is used to calculate another important parameter T-stress (i.e., tangential stress acting parallel to the crack) which is also responsible for the crack growth behavior in many cases. T-stress could be positive (tensile) or negative (compressive) in nature. The negative T-stress causes an increase in fracture toughness of the material. However, for a small crack length to ligament ratio, the effect of T-stress is limited especially on mode I opening (Zhao and Xu, 2014). This T-stress term along with stress intensity factor is an essential part of the bi-parametric fracture mechanics.

### 2.4.6 Cohesive Zone Model

Dugdale (1960) came up with the idea of cohesive zone in front of the crack tip for the ductile materials. Cohesive traction is present in the cohesive zone, which opposes the crack
opening. This traction initially increases and consequently traction free surfaces emerge in the
direction of crack growth. There are different models to capture this cohesive traction force
namely Dugdale model, linear softening model, trapezoidal model, and exponential model. As
per all these models, the crack grows when the separation distance between two surface reaches
to a critical value called critical separation distance and failure occurs. Barenblatt (1962) set
similar concept of cohesive zone forth for the brittle materials. This model considered the
cohesive zone at the crack tip and extended it towards the crack opening (not into the material in
front of the crack tip). As the distance between two surfaces near the crack tip is less, it was
assumed that the atomic bonding forces between two surfaces are holding the two atomic layers
together (Sun and Jin, 2012).

2.4.7 Crack Band Model

Bažant and Oh (1983) proposed crack band model to capture the non-linear behaviour in
quasi brittle material (eg., concrete, rocks). The fracture process zone ahead of crack tip is larger
in case of quasi brittle materials as compared to the brittle as well as ductile materials. This
increase in length of fracture process zone is attributed to the presence of heterogeneity in the
concrete and rocks. Crack is modelled as a layer of smeared crack with constant height. Within
the crack band the stress softening occurs which leads to gradual reducion of stresses. In case of
rocks under compressive loading the understanding of the fracture mechanics helps in explaining
different process. The failure process for rock has been characterized by different stages i.e.,
crack closure, elastic deformation, crack initiation at micro level, growth of micro-cracks, micro
crack coalescence to form macro cracks, macro-crack coalescence which leads to ultimate failure.
However, under tensile loading the crack closure process will be absent. The failure theories for
brittle material in fracture mechanics can also be extended to rocks (Bieniawski, 1967; Martin and Chandler, 1994).

2.5  Digital Image Correlation (DIC) Technique

2.5.1 Historical Development of DIC Technique

DIC is a well-accepted non-contact technique to obtain the displacement and strain behaviors of the brittle material. This method is also known as digital speckle correlation method (Pan et al. 2009). DIC provides full field non-contact measurement of strains on the surface of the specimen as compare to the conventional strain gauge, which provides only data for a certain point or region. The advancement in hardware and software technology of high-speed photography renders it most appropriate non-contact method to measure surface strain (Sutton et al., 2007; Lin and Labuz, 2013; Aliabadian et al., 2019;). Over the last century, the high-speed photography had witnessed a dramatic evolution. The most important step in DIC is acquisition of high-quality images of the specimen with a stochastic speckle pattern having a uniformly distributed gray-scale intensity histogram (Pan et al., 2009; Sutton et al., 2009; Hedayat et al., 2014; Shirole et al., 2020).

Historically, the application of photography for scientific research can be traced back to 1872 when the photographer, Eadweard Muybridge conducted motion analysis. He used 12 cameras placed at an interval to capture the images of a galloping horse. By 1930s the camera by Kodak was able to reach as high frame rate as 5000 fps. With the development of charged-coupled devices (CCD) and complementary metal oxide semiconductor (CMOS), the digital cameras can capture images at the rate as high as 10 million frames per second (Xing, 2017).
As far as the origin of image correlation technique is concerned, Peters and Ranson (1982) are credited with the development computer based digital image technique for stress analysis in solid mechanics. Sutton et al. (1983) generated a numerical algorithm to perform digital image correlation. In 1986, Sutton et al. (1986) further improved the efficiency of the method by improving the algorithm using gradient search method which enhanced the accuracy of the method up to the sub pixel level (Sutton et al., 2009). Before the development of DIC techniques, other photogrammetric techniques like Holography interferometry, speckle interferometry and moiré interferometry were famous among the researchers (Pan 2009).

2.5.2 Fundamental Principles of DIC Technique

For DIC, the random speckled images are captured during the testing of the specimen. The frame rate of the camera system and resolution should be compatible for the intended purpose. For a high-speed phenomenon, a higher frame rate of imaging is required to sufficiently capture the whole process. Area of interest, AOI (also called Region of Interest) is selected. The AOI is further divided into the subset of suitable size. The subset size effect the result of the correlation. The subset should be neither too coarse nor too fine. By making the subset coarser, the results will be incorrect and making too finer will reduce the efficiency the algorithm to differentiate between different subsets. A small subset size may be insufficient in capturing a random pattern and may introduce noise. The speckle size on the physical specimen surface should cover at least three pixels in the image. The step should be smaller than the subset size and typically less than half the size of the subset is appropriate (Hedayat et al., 2014).
The subset in the target frame is correlated to the reference frame based on the correlation coefficients. Different algorithms are available to carry out this correlation. The two main categories are cross correlation coefficient ($C_{CC}$) and sum of squared differences correlation coefficient ($C_{SSD}$). Other algorithms, which are commonly used by researchers, are the normalized forms of these correlation coefficients. Examples include normalized sum of squared difference (NSSD), zero normalized sum of squared difference (ZNSSD), normalized cross correlation (NCC) and zero normalized cross correlation (ZNCC) (Hedayat et al., 2014). Cross correlation coefficients are calculated by multiplying the pixel intensity of the point in reference frame to the pixel intensity of same point in spatial or temporal coordinates in the deformed frame and then add these values for each point in the subset. The maximum value of cross correlation coefficient ($C_{CC}$) will represent a better match. In case of sum of squared difference correlation coefficient, the pixel intensity of the point in the deformed frame are subtracted from the pixel intensity of the same point in reference frame and square the result to remove the negative sign. Then all the values for the points in a subset are added together. For the sum of squared difference correlation coefficient ($C_{SSD}$), the best match will have the lowest value.

\[
C_{CC} = \sum_{i=-M}^{M} \sum_{j=-M}^{M} [f(x_i, y_j)g(x'_i, y'_j)]
\]  
\[
C_{SSD} = \sum_{i=-M}^{M} \sum_{j=-M}^{M} [f(x_i, y_j) - g(x'_i, y'_j)]^2
\]  

where $M$ represents the half length of subset, the pixel intensity at the point with coordinates of $(x_i, y_j)$ in the reference image is given by $f(x_i, y_j)$. Similarly, the pixel intensity at the point with coordinates of $(x'_i, y'_j)$ in the deformed image is given by $g(x'_i, y'_j)$. The issue with the
standard form of correlation coefficients is that they are prone to error for the data with skewed values or unexpected high intensity. The normalized form of the correlation coefficients performs well under the change in illumination or scale effect; however, the normalized forms are not simple and they are computationally expensive as compared to the standard form. The normalized form of the correlation coefficients is presented as follows:

\[
C_{NC} = \sum_{i=-M}^{M} \sum_{j=-M}^{M} \left[ \frac{f(x_i, y_j)g(x'_i, y'_j)}{\bar{f} \bar{g}} \right]
\quad (2-22)
\]

\[
C_{NSSD} = \sum_{i=-M}^{M} \sum_{j=-M}^{M} \left[ \frac{f(x_i, y_j)}{\bar{f}} - \frac{g(x'_i, y'_j)}{\bar{g}} \right]^2 \quad (2-23)
\]

where,

\[
\bar{f} = \sqrt{\sum_{i=-M}^{M} \sum_{j=-M}^{M} \left[ f(x_i, y_j) \right]^2} \quad (2-24)
\]

\[
\bar{g} = \sqrt{\sum_{i=-M}^{M} \sum_{j=-M}^{M} \left[ g(x'_i, y'_j) \right]^2} \quad (2-25)
\]

The zero normalized form of correlation coefficient is given by:

\[
C_{ZNCC} = \sum_{i=-M}^{M} \sum_{j=-M}^{M} \left\{ \frac{f(x_i, y_j) - f_m}{\Delta f} \times \frac{g(x'_i, y'_j) - g_m}{\Delta g} \right\} \quad (2-26)
\]

\[
C_{ZNSSD} = \sum_{i=-M}^{M} \sum_{j=-M}^{M} \left[ \frac{f(x_i, y_j) - f_m}{\Delta f} - \frac{g(x'_i, y'_j) - g_m}{\Delta g} \right]^2 \quad (2-27)
\]

where,
ZNSSD (Zero Normalized sum of squared Difference) and ZNCC (Zero Normalized Cross Correlation) are highly recommended methods for digital image correlation. Both methods are insensitive to the effect of change in illumination intensity (Pan et al., 2009; Sutton et al., 2009).

Displacement fields are mapped from the deformed image with respect to the reference image. The displacements are calculated based on the relationship between the reference coordinates \((x_0)\) to the deformed coordinates \((x')\):

\[
x' = x_0 + \Delta x + u + \frac{\partial u}{\partial x} \Delta x + \frac{\partial u}{\partial y} \Delta y
\]

\[
y' = y_0 + \Delta y + v + \frac{\partial v}{\partial x} \Delta x + \frac{\partial v}{\partial y} \Delta y
\]

where \(u\) and \(v\) are the displacement vectors of the center of the subset in x and y direction. \(\Delta x\) and \(\Delta y\) represents the distance of the point from the center of the subset. Optimum values of \(u\), \(v\), \(\frac{\partial u}{\partial x}\), \(\frac{\partial u}{\partial y}\), \(\frac{\partial v}{\partial x}\) and \(\frac{\partial v}{\partial y}\) are estimated based on Newton-Raphson method to get the best value for the correlation coefficients. Newton–Raphson (NR) method is suitable method, which also cater...
for the deformation of the subset while estimating the solution for DIC correlation. For Newton-Raphson method, the initial guess is critical otherwise there will be convergence issue (Pan 2009). The correction term for the initial guess in the Newton-Raphson method is given by:

$$\Delta P = -H^{-1}(P_i) \times \nabla (P_i)$$

(2-34)

where \(\nabla (P_i)\) is the Jacobian matrix and \(H(P_i)\) is the Hessian matrix. \(P_i\) is the function of unknown elements for which equation (2-34) will be solved. \(P_i\) is given by:

$$P_i = \begin{bmatrix} u \\ v \\ \frac{\partial u}{\partial x} \\ \frac{\partial u}{\partial y} \\ \frac{\partial v}{\partial x} \\ \frac{\partial v}{\partial y} \end{bmatrix}$$

(2-35)

Nonlinear shape function is used to interpolate the values of displacements at sub pixel level. Strains are calculated based on the displacement vectors using Green Lagrange strain tensor. The calculated strains are made smooth by using the average value of the neighboring pixels. The larger filter size provides smoother results but at the expense of resolution. If a small filter size is applied then the resultant strain field will be noisy. To smoothen the strain field, different type of filters can be used (e.g., Gaussian filter or the uniform filter). In uniform filter, all the values of strain for neighboring pixels get equal weightage, while in Gaussian filter the central pixel gets a higher weight.
\[ \varepsilon_{xx} = \frac{1}{2} \left[ 2 \frac{\partial u}{\partial x} + \left( \frac{\partial u}{\partial x} \right)^2 + \left( \frac{\partial v}{\partial x} \right)^2 \right] \]  
(2-36)

\[ \varepsilon_{xy} = \frac{1}{2} \left[ \frac{\partial u}{\partial y} + \frac{\partial v}{\partial x} + \frac{\partial u}{\partial x} \frac{\partial u}{\partial y} + \frac{\partial v}{\partial x} \frac{\partial v}{\partial y} \right] \]  
(2-37)

\[ \varepsilon_{yy} = \frac{1}{2} \left[ 2 \frac{\partial v}{\partial y} + \left( \frac{\partial u}{\partial y} \right)^2 + \left( \frac{\partial v}{\partial y} \right)^2 \right] \]  
(2-38)

This Green Lagrange strain (\(\varepsilon_{GL}\)) can be converted to the Engineering strain, (\(\varepsilon_{engr}\)) using following relationship.

\[ \varepsilon_{engr} = \sqrt{2\varepsilon_{GL} + 1} - 1 \]  
(2-39)

2.5.3 Experimental Considerations for Application of DIC Technique

The speckle pattern should be randomly distributed and the intensity histogram of the image should be uniform and lies between 50 to 220 gray scale values (Lin and Labuz, 2013; Shirole et al., 2020a). The speckle pattern should be randomly oriented without any bias in a particular direction. The speckle pattern should cover at least three pixels in the image (Sutton et al., 2009; Garg et al., 2019; Butt et al., 2020; Shirole et al., 2020b). There are different ways of applying speckle pattern like using stencil scale, computerized printed speckle pattern, and spray paints. For the spray paints, either use black over white background or white over black background. However, in literature most of the experiments were conducted with the specimens prepared by spraying the black paint over the white background. To minimize excessive glare, matt spray paints are recommended.
The illumination should be sufficient to illuminate the whole specimen. The frame rate of the camera should be based on the intended purpose of the research. If the frame rate is too low then it may not be able to capture the phenomenon properly to deduce some meaningful result. The resolution of the camera should be able to generate a high-quality image. The shutter speed should be adjusted to get un-blurred images. The interval of shutter opening should be less than the time interval between capturing of two successive images. There are many TTL (transistor-to-transistor logic) devices available to synchronize the image capturing process and commencement of loading of the specimen.

The field of the view of the camera should be adjusted to cover the whole specimen and maximum number of pixels will be available to register the image. This will increase the resolution of the image. To adjust the field of view of the camera, extension tubes are available for the lens, which will change the magnification of the lens. The image should be properly focused. For 2d DIC, both the specimen surface and the plane of camera must be parallel to each other. The calibration is done using the identified length between two known points in the physical domain and compares the same distance on the image based on the number of pixels. For 3D DIC, the elaborate calibration process is followed by using triangulation.
3.1 Raw Material

In this study, the raw mine tailings were procured from a gold mine in Mollehuaca, Arequipa, Peru. Geotechnical, mineralogical, and chemical characterizations were carried out to ensure that the mine tailings contain the desired amounts of alumino-silicates for their potential application for geopolymerization. For geotechnical characterizations, applicable ASTM standards (D6913, D7928, and D4318) were followed. The particle size distribution of raw mine tailings is presented in Figure 3-1.

![Figure 3-1 Grain size distribution of the raw mine tailings used in this study](image)

As per Unified Soil Classification System (USCS), the mine tailings were “gravelly lean clay with sand (CL)” having a low plasticity with a plasticity index (PI) of 9.8%. There were 49.9% fine particles present by weight in the mine tailings. The particle size for $D_{10}$, $D_{30}$ and $D_{60}$
were 0.003, 0.029, and 0.097 mm, respectively. The coefficient of uniformity \((C_u)\) was 32.33 and the coefficient of curvature \((C_c)\) was 2.89. The raw mine tailing had low capacity of holding water as evident by lower value of the activity constant (i.e., \(A=0.195\)). The important geotechnical properties are summarized in Table 3-1.

Based on the results of the X-ray diffraction (XRD) test, the mineral composition of the tailings consists of 77.7% quartz, 14.30% muscovite, 4.20% calcite, and 7.6% other crystalline minerals. XRD pattern and the percentage composition of the tailings are presented in Figure 3-2. By performing the Scanning Electron Microscopy/Energy Dispersive X-Ray Spectroscopy (SEM/EDS), the chemical elemental composition of the raw tailings was determined and consisted of three dominant elements of silicon \((Si)\), iron \((Fe)\), and aluminum \((Al)\). These mine tailings had a silicon to aluminum ratio \((Si:Al)\) of approximately 2.55 which has a good potential for geopolymerization (e.g., Ahmari and Zhang, 2013; Zhang et al., 2020a).

Table 3-1 Geotechnical properties of the raw mine tailings

<table>
<thead>
<tr>
<th>Properties</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific gravity, (G_s)</td>
<td>2.70</td>
</tr>
<tr>
<td>Liquid Limit, LL (%)</td>
<td>22.8</td>
</tr>
<tr>
<td>Plastic Limit, LL (%)</td>
<td>13.0</td>
</tr>
<tr>
<td>Plasticity Index, PI (%)</td>
<td>9.76</td>
</tr>
<tr>
<td>(D_{10}) (mm)</td>
<td>0.003</td>
</tr>
<tr>
<td>(D_{30}) (mm)</td>
<td>0.029</td>
</tr>
<tr>
<td>(D_{60}) (mm)</td>
<td>0.097</td>
</tr>
<tr>
<td>Pass #200 sieves, F (%)</td>
<td>49.9</td>
</tr>
<tr>
<td>Coefficient of uniformity, (C_u)</td>
<td>32.3</td>
</tr>
</tbody>
</table>
Table 3-1 Continued

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coefficient of curvature, $C_c$</td>
<td>2.89</td>
</tr>
<tr>
<td>Optimum moisture content (%)</td>
<td>15.5</td>
</tr>
</tbody>
</table>

Figure 3-2 XRD pattern along with percentage of each mineral in raw tailings (Q: quartz; M: muscovite; C: calcite)

3.2 Specimen Preparation

Specimens were prepared for Brazilian indirect tensile strength test and semi-circular bend test. General procedure to prepare the specimens were similar except the dimensions of the specimens. For SCB specimens, saw cutter was used to cut the circular specimens in two halves. For the Brazilian indirect tensile test, the molar concentration of NaOH was a variable whereas
for the SCB specimens the ratio of notch length to the radius (a/R) of the specimen was varied. The detailed procedure to prepare the specimens for Brazilian indirect tensile test and semi-circular bend test is discussed in the following sections.

3.2.1 Specimen Preparation for Brazilian Indirect Tensile Test

Mine tailings based geopolymer concrete specimens were prepared by adding sodium hydroxide (NaOH) as an alkali activation agent at different molar concentrations of 8M, 10M and 12 M. Generally, NaOH is highly soluble in water. 50% by weight solution of NaOH is a highly saturated solution, its molarity is approximately 19M, and it contains 762.2 gm of NaOH in 1000 ml solution. In literature, different ranges of NaOH concentrations have been explored in other similar studies. Manjarrez and Zhang, 2018 used up to 13M concentration in their study. For a similar raw material, Zhang et al., 2020a used up to 12 M concentration of the NaOH to study the compressive behavior of the mine tailings. NaOH pellets were dissolved in water to make the solutions. The water to tailing ratio was kept constant at 20% by weight. A solution with 1M concentration of NaOH requires 40 gm of NaOH pellets. Exothermic reaction occurs by adding NaOH in water and the temperature raises. The solution was kept at room temperature for 30 minutes to cool down before adding to the raw tailings. The paste was then thoroughly mixed and was left at the room temperature for 30 minutes for heat diffusion. Then specimens were molded using steel molds having a diameter of 50 mm and depth of 33 mm by referring to ASTM standard (D-3967), where the circular disc specimens with thickness to diameter ratio from 0.2 to 0.75 are recommended. In this study, the thickness to diameter ratio of 0.66 was used.
Cubic specimens were also prepared to confirm the repeatability of the specimens. Each side of the cubic specimen was 2 inch in length. Two different curing conditions were used to compare the results. In first case the specimen were cured at room temperature and without any airtight container. In second case, the specimens were cured in an airtight container. For both the cases, the specimens were placed in an oven for initial 24 hours to accelerate the geopolymerization process. The specimens cured in airtight container provided higher and repeatable strength of the specimens. Therefore, curing of the specimen in an airtight container was adopted.

A total of 45 disk specimens were prepared for Brazilian indirect tensile test which means 15 disk specimens for each molar concentration. The specimens were cast in the mold in three layers and each layer was tamped 25 times by using Harvard miniature compaction tamper to ensure proper compaction. Then, the specimens were placed in an oven for 24 hours at an elevated temperature of 75°C. In literature, the curing temperature ranges between 60°C to 120°C. Ahmari et al., (2011) reported an increase in strength from 60°C to 90°C and then decrease in strength if temperature is further increased. In this study, the middle range of the temperature was selected between 60°C to 90°C. This ensured reasonable strength gain with less energy consumption. Specimens were wrapped in polyethylene sheet to avoid moisture loss. After 24 hours, the specimens were removed from the oven and then were demolded. These specimens were wrapped in a polyethylene sheet again and were placed in an airtight container for the remaining period of curing. Five specimens for each molar concentration were tested after 7 days and 28 days of curing. Prior to the testing, specimens were placed in an oven at a temperature of 75°C for 24 hours to achieve the dry condition.
To utilize the DIC technique, a stochastic gray-scale speckle pattern was applied to the specimen surface by spray painting. For this purpose, the white color was sprayed as a background and then black spray paint was used to apply the speckle pattern. The speckle pattern should be randomly oriented without any bias in a particular direction. The paint was sprayed at a distance of two feet to create small speckles. When the paint is sprayed too close, instead of creating speckles, the surface tends to be covered in black color. To ensure a reduction in excessive glare from the surface of the specimen, matt color paints were sprayed on the specimens. The speckle size and the number of pixels assigned to the object ensured that each speckle was imaged by at least three pixels in the image, as suggested by Sutton (2009). A schematic presentation of the specimen preparation is shown in Figure 3-3. Pictorial view of the different stages of the specimen is shown in Figure 3-4.
Figure 3-3  Procedure for preparation and testing for Brazilian indirect tensile testing

Table 3-2 Preparation of different types of specimen for Brazilian tensile test

<table>
<thead>
<tr>
<th>Molar concentration of NaOH (M)</th>
<th>Water Content (%)</th>
<th>Curing age (days)</th>
<th>Number of specimens tested</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>20</td>
<td>7 &amp; 28</td>
<td>15</td>
</tr>
<tr>
<td>10</td>
<td>20</td>
<td>7 &amp; 28</td>
<td>15</td>
</tr>
<tr>
<td>12</td>
<td>20</td>
<td>7 &amp; 28</td>
<td>15</td>
</tr>
</tbody>
</table>
3.2.2 Specimen Preparation for Semi-circular Bend Test

For semi-circular bend (SCB) test, 10 M concentration of NaOH was chosen as the representative condition to further explore the ensile and fracturing characteristics of the geopolymerized tailings. Three different notch length to radius ratios (a/R) of 0.2, 0.25, and 0.3 were selected. The specimen preparation method was similar to the Brazilian indirect tensile method, except that the dimensions of the molds were different. The water to tailing ratio was kept constant at 20 % by weight. A solution of 10 M concentration of NaOH was added to the mine tailings. The solution was kept at room temperature for 30 minutes to cool down before adding to the raw tailings. The paste was thoroughly mixed and then left for 30 minutes to diffuse the heat. The paste was then molded into circular specimens using steel molds with
diameter of 75 mm and height of 40 mm. The schematic of semi-circular bend test is presented in Figure 3-5.

The specimens were prepared by proper compaction in three layers. Each layer was compacted 25 time using Harvard miniature compaction tamper. Five specimens were prepared for each notch to radius ratio. The specimens were wrapped in polyethylene sheet and placed in oven at 75°C for 24 hours. The specimens were then removed from oven and demolded. After demolding, the specimens were cut into two pieces using a bench top scroll saw, as shown in Figure 3-6. A notch was then created at the center of each semi-circular specimen. The specimens were again wrapped in polyethylene sheets and placed in an airtight container for remaining period of curing. Specimens were tested after seven days of curing age.

Table 3-3 Preparation of different types of specimen for semi-circular bend test

<table>
<thead>
<tr>
<th>Notch length to radius ratio (a/R)</th>
<th>Molar Conc of NaOH</th>
<th>Water Content (%)</th>
<th>Curing age (days)</th>
<th># of specimens</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.20</td>
<td>10</td>
<td>20</td>
<td>7</td>
<td>5</td>
</tr>
<tr>
<td>0.25</td>
<td>10</td>
<td>20</td>
<td>7</td>
<td>5</td>
</tr>
<tr>
<td>0.30</td>
<td>10</td>
<td>20</td>
<td>7</td>
<td>5</td>
</tr>
</tbody>
</table>
Figure 3-5 Schematic of the specimen for semi-circular bend test

Figure 3-6 Preparation of semi-circular specimen for SCB test
3.3  Experimental Setup

3.3.1  Experimental Setup for Brazilian Indirect Tensile Strength Test

The specimens were diagonally loaded using MTS loading frame. The loading frame was controlled using a Multipurpose TestWare system. The capacity of the loading machine was 100 kN. Flat platens were used to load the specimen. The test was conducted using force control mode with a loading rate of 2 Newtons per second (N/s). Loading machine was configured to get the data point after every 42 milliseconds. The application of diagonal load caused the stress concentration at the center of the specimen. This stress is tensile in nature. The peak load at the failure of the specimen was used to ascertain the splitting tensile strength of the specimen.

Through the experiment, the images of the specimen were recorded for DIC application. The loading machine and the camera system were synchronized using transistor-to-transistor logic (TTL) device to allow camera triggering at the same time as the start of the test. The experimental setup is shown in Figure 3-7.

FLIR grasshopper camera (GS3-U3-23S6M) was used to capture the images throughout the duration of the test. The camera was configured to capture images after every 42 milliseconds thereby achieving a frame rate of approximately 23.8 frames per seconds. The image resolution of the camera was 1920 x 1200 pixels. The distance between specimens and the camera lens was kept at 20 inches. The extension tube of 35 mm was used to narrow the field of view as per the dimensions of the specimen. This practice ensured that maximum numbers of the available pixels were allotted on the surface of the specimen. The camera system was controlled through the computer system using Vic Snap software. Vic Snap is a commercial image capturing software.
developed by Correlated Solutions. For illumination purposes, two lighting system with halogen bulbs were used.

![Experimental Setup for Brazilian indirect tensile test]

Figure 3-7 Experimental Setup for Brazilian indirect tensile test

**DIC Correlation**

DIC correlation analysis was done using zero normalized squared difference correlation (ZNSSD) method, which has potential to offset the noise in images due to fluctuation in lightening (Sutton et al., 2009). Other algorithms which are commonly used by researchers are sum of squared difference (SSD), normalized sum of squared difference (NSSD), cross correlation (CC), normalized cross correlation (NCC) and zero normalized cross correlation.
(ZNCC) (Hedayat et al., 2014). The algorithm was implemented through commercial software Vic 2D (Correlated solutions 2009).

In DIC, the selection of subset size and step size is critical for achieving the highest accuracy. The subset size refers to the number of pixels that are chosen for the correlation and therefore is adjusted to provide a random speckle pattern for the correlation. A small subset size may be insufficient in capturing a random pattern and may introduce noise. In this study, through comparative evaluations, the subset was selected as 15 pixels and step size was chosen to be 5 pixels. The step should be smaller than the subset size and typically less than half the size of the subset is appropriate (e.g., Hedayat et al., 2014). In DIC, the deformed image is correlated to the reference images.

The displacements and strains were calculated by correlating the differences between reference and deformed images. Lagrange strain tensor was used for calculation of the strain. For calculation of strain the filter size was kept at 5 pixels. The lower size of filter provides high resolution of the full-field strain but the values are noisy. Too high size of the filter makes the strain field smooth but at the expense of the resolution. Gaussian filter was used to calculate the strain, which assigned higher weightage to the point of interest as comparing to its neighboring points. In this study, one pixel of the image corresponded to 0.045 mm (45 microns) in physical dimensions. The sub-pixel level accuracy makes it possible to ascertain even small values of the strain. In this experimental setup, system was able to capture strains as low as 0.004% strain. Cho and Chasiotis, (2007) suggested the accuracy level upto 1/8th of the pixel level which is approximately 0.01% of strain for this setup.
Virtual strain gauges were applied to measure the tensile strain in transverse direction to the applied load. A total of nine virtual strain gauges were applied as shown in Figure 3-8. The distance between the strain gauges was kept as 5 mm.

![Figure 3-8 Location of virtual strain gauges on the specimen for Brazilian indirect tensile strength test](image)

3.3.2 Experimental Setup for Semi-circular Bend Test

The semi-circular specimens with a notch were loaded using three point bending fixture. MTS loading machine was used to load the specimen. The maximum capacity of this loading machine was 100 kN. Specimens were loaded under displacement control mode. The loading rate was set at 0.05 mm/ min. The Multipurpose TestWare was used to control the loading machine. Data acquisition rate from the loading machine was configured to get data points after every 42 milliseconds. The peak load at the failure of the specimen was used to ascertain the flexural strength of the specimen using following relationship:

\[ \text{Flexural Strength} = \frac{P}{b.d} \]

Extensometer was attached at the bottom of the specimen to capture the crack mouth opening displacement. To attach the extensometer with the specimen two metallic strips were fabricated and attached on both sides of the notch using epoxy. The distance between the
metallic strips was kept between 12 to 13 mm so that the extensometer can easily fit in the gap.

Experimental setup for semi-circular bend test is presented in Figure 3-9.

During the loading of the specimen, images were captured for DIC analysis. FLIR grasshopper camera (GS3-U3-23S6M) was used to capture the images throughout the duration of the test. The camera was configured to capture images after every 42 milliseconds thereby achieving a frame rate of approximately 23.8 frames per seconds. DIC analysis was done in a similar approach as discussed in section 3.3.1.
CHAPTER 4

BRAZILIAN INDIRECT TENSILE STRENGTH TEST

4.1 Splitting tensile strength of the specimen

For splitting tensile strength 8M, 10M and 12M specimen were tested after 7 days and 28 days of curing age. Five specimens were tested for each molar concentration and curing age. Based on the experimental results from Brazilian tensile strength tests, the tensile properties of geopolymerized mine tailings can be characterized. The representative load displacement curves are presented in Figure 4-1; these curves are based on the specimen with average load from each category of the specimens. It is evident that the strength of specimen increased with the increase in molar concentration and curing age of the specimens.

Figure 4-1 Representative load-displacement curves for geopolymerized samples based on molar concentration and curing age (M: molar concentration of NaOH and D: curing age in days)
The splitting tensile strength of the specimen was calculated using the equation (2-3) mentioned in section 2.2. The average splitting tensile strength of the raw tailings are shown in Figure 4-2. The result of the splitting tensile test is summarized in Table 4-1.

![Figure 4-2 Tensile strength of the specimen for different molar concentrations and curing age (M: molar concentration of NaOH and D: curing age in days)](image-url)
Table 4-1 Summary of Splitting tensile test

<table>
<thead>
<tr>
<th>Group</th>
<th>Specimen Number</th>
<th>Splitting Tensile Strength kPa</th>
<th>Coefficient of Variation %</th>
</tr>
</thead>
<tbody>
<tr>
<td>8 M-7days</td>
<td>1</td>
<td>176.02</td>
<td>7.2</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>158.41</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>192.25</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>172.04</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>158.88</td>
<td></td>
</tr>
<tr>
<td>10 M-7days</td>
<td>1</td>
<td>376.50</td>
<td>4.9</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>384.63</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>422.70</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>425.60</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>406.88</td>
<td></td>
</tr>
<tr>
<td>12 M-7days</td>
<td>1</td>
<td>548.45</td>
<td>4.2</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>494.40</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>526.78</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>510.99</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>489.59</td>
<td></td>
</tr>
<tr>
<td>8M-28days</td>
<td>1</td>
<td>1523.86</td>
<td>4.4</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>1482.62</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>1409.16</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>1360.01</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>1376.39</td>
<td></td>
</tr>
<tr>
<td>10M-28days</td>
<td>1</td>
<td>1556.63</td>
<td>3.4</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>1573.02</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>1638.56</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>1687.72</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>1540.25</td>
<td></td>
</tr>
<tr>
<td>12M-28days</td>
<td>1</td>
<td>1725.41</td>
<td>4.4</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>1769.65</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>1951.53</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>1754.90</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>1802.42</td>
<td></td>
</tr>
</tbody>
</table>
4.2 DIC results

For DIC analysis, the grayscale intensity of the image, as demonstrated through its distribution, was within acceptable limits and with proper distribution. When the image is too dark or too bright, the distribution can be skewed towards the lower or higher greyscale intensity values. To analyze the grayscale intensity of the image, a histogram was drawn between grayscale intensity of the image versus the frequency of the speckles and result is presented in Figure 4-3. It is clearly visible that the image lies at the center of the grayscale intensity. The lowest limit of grayscale represents the black color with a value of 0 and maximum value of grayscale represents the white color with the value of 255. In our case, the bulk of the speckles on the surface of the specimen lied between 100 and 200 grayscale level and peak lied at grayscale intensity of 160.

Figure 4-3 Histogram representing the grayscale intensity of the speckle pattern on the surface of the specimen for Brazilian indirect tensile test
The full-field strain values were calculated using the DIC techniques. Virtual strain gauges were applied to measure the tensile strain in transverse direction to the applied load. Nine virtual strain gauges were applied as shown in Figure 4-4. The distance between the strain gauges was kept as 5 mm. Figure 4-5, and Figure 4-7 to Figure 4-9 show the graph of strain throughout the duration of test along with the corresponding load for each case of the specimen: 8M and 10M specimens for 7 days and 28 days curing age. The strain graph was magnified closed to the failure and four points were identified to demonstrate the significant changes in the strain values. Strain gauges were labeled from top to the bottom starting from S-1 to S-9. Strain gauge S-5 was located at the center of the specimen, half way between the opposite loading platens. The strain gauges S-1 to S-4 were above the center of the specimen and strain gauges S-6 to S-9 were located below the center of the specimen.

![Figure 4-4 Location of the virtual strain gauges (labeled as S-1 to S-9)](image)

Figure 4-5 shows the strain graph for 8M specimen with a curing age of 28 days. The specimen achieved the ultimate load of 3.49 kN. For this specimen, all strain gauges exhibited a similar trend up to \( t = 1721.7 \) seconds. At this time, the strain at the center of specimen was 0.03
%. A drop in the strain value for all strain gauges was observed between t=1721.7 and 1722 seconds. The strain gauges below the center of the specimen (i.e., S-6 to S-9) showed a larger tensile strain as compare to the strain level observed by the strain gauges above the center (i.e., S-1 to S-4). This stage corresponds to the crack initiation stage of the specimen. The strain gauges at the upper portion of the specimen showed higher amount of drop and reached a negative strain amount, indicating compression. This phenomenon can be attributed to the fact that near the failure, the lower half of the specimen was in tension but the upper half was subjected to compression due to the application of an internal moment to the fracture plane. This phenomenon occurred for a short period and all strain gauges continued to register tensile strains (positive values). After t=1722 seconds, the strain at the center of the specimen raised to 0.06%. Then, there was a drastic change in the curve with strain levels rising as high as 2%.

Figure 4-5 Variation of strain and load over the time for 8 M-28 days specimen
Figure 4-6 shows the full field horizontal strain profile of the specimen at different time frame as marked alphabetically (a, b, c, and d) on the curves in Figure 4-5 and Figure 4-7 to Figure 4-9. The points (a-d) are selected based on the strain concentration. At point "a" no strain concentration has occurred, point "b" is the point where the curve changed from positive to negative strain values, point "c" is in the valley of the curve (as strain is negative/compressive in nature); and point "d" is selected just prior to failure. The corresponding strain profile for each stage of a-d marked on Figure 4-5, are shown in Figure 4-6(1). Initially there was no strain concentration. With the increase in the applied load, the strain continued to increase and started concentrating at the center. The central part of the specimen was under extreme tensile stress, which led to failure in tensile mode. In Figure 4-6(1c), the strain localization at the center of the specimen is clearly visible. This strain concentration started moving towards the load platens in both direction. Figure 4-6(1d) shows the strain profile just prior to the failure of the specimen.
Figure 4-6 (2) shows the strain profile for the 8M specimen cured for 7 days. Initially, there was no strain concentration except some near the loading platens. With the increase in the applied load, the strain continued to increase and started concentrating at the center. The central part of the specimen was under extreme tensile stress, which led to failure in tensile mode. In Figure 4-6(2c), the strain localization near the platens increased and then strain concentration occurred starting from the bottom of the specimen near the platen and gradually moved towards the upper platen. This trend is evidenced by the higher strain values for bottom strain gauges as
compared to the strain gauges at the top portion of the specimen as shown Figure 4-7. The Figure 4-6(3) represents the 10M specimen loaded after 28 days of curing. The strain concentration occurred first at the upper portion and then migrated towards the bottom platen. Figure 4-6(4) depicts the full-field strain values for the 10M specimen, cured for 7 days. The strain concentration occurred at the lower portion of the specimen, that with further application of the load, migrated towards the bottom platen first and then the top platen. In all cases presented in Figure 4-6, the tensile strain concentration occurred along the diameter of specimen parallel to loading path, resulting in splitting of the specimen in two halves, which is a typical response of splitting tensile strength test.

The representative strain-time curve for the 8M specimen cured for 7 days is shown in Figure 4-7. Results show that the load at failure is approximately 0.38 kN. All strain gauges showed a similar trend up until t = 175 seconds after which the lower most strain gauge (S-9) deviated from other strain gauges. At this stage, the strain at the center of the specimen was 0.0239%. The strain gauges below the center of the specimen (i.e., S-6 to S-9) showed a larger tensile strain as compare to the strain level observed by the strain gauges above the center (i.e., S-1 to S-4). This stage corresponds to the crack initiation stage of the specimen. At t=184.7 seconds, the strain at the center of the specimen raised to 0.03% and all the strain gauges exhibited a non-linear response and within a time frame of less than a second, there was a drastic change in the curve with strain levels rising as high as 2%.

The representative strain evolution graph for 10 M specimen with a curing age of 28 days is presented in Figure 4-8. In this case, the specimen failed at 3.7 kN. Strain values observed by
different strain gauges deviated from similar trends at t=1830.7 seconds. Figure 4-9 shows the 10M specimen with curing age of 7 days. The specimen sustained a maximum load of 0.95 kN before failure. All the strain gauges followed a similar trend until t= 460 seconds of loading at which stage the strain at the center of the specimen reached the value of 0.02%. At this point, the strain gauges located at the upper portion of the specimen registered a compressive (negative) strain. This is again likely due to the application of internal moment to the fracture plane. Upon further application of the load, the specimen failed and sudden rises in the strain levels for all gauges were observed. By comparing the strain levels near to the failure, it can be concluded that the 10M specimens with 28 days of curing accumulated lower strain values compared to other cases. The specimen with 10M concentration exhibited higher strength as expected because the increase in the alkali activator facilitated higher geopolymerization and consequently higher gel formation, which is responsible for the increase in strength of the specimen. The increase of compressive strength with respect to the increase in concentration of alkali agents were reported by several researchers (e.g., Ahmari and Zhang, 2013; Zhang et al., 2020a, b, 2021). After 28 days of curing, strain values at the center of specimen at failure were 0.03% and 0.02% for the 8M specimen and 10M specimens, respectively.
Figure 4-7 Variation of strain and load over the time for 8M-7 days specimen

Figure 4-8 Variation of strain and load over the time for 10M-28 days specimen
4.3 Determining the elastic properties using DIC results

The elastic properties (e.g., elastic modulus, $E$ and Poisson’s ratio, $\nu$) can be determined from the full field displacement profile from the DIC result through linear regression. The relationship is based on plane stress conditions (Sgambitterra et al., 2018).

\begin{align*}
    u &= \frac{1}{E}K_{1x} + \frac{\nu}{E}K_{2x} \quad (4-1) \\
    v &= \frac{1}{E}K_{1y} + \frac{\nu}{E}K_{2y} \quad (4-2)
\end{align*}

where $u$ and $v$ represents the horizontal and vertical displacement at a point, respectively. $E$ and $\nu$ are the elastic properties i.e., elastic modulus and Poisson’s ratio respectively. $K_{1x}$, $K_{2x}$, $K_{1y}$ and $K_{1y}$ are the parameters based on loading, geometry and location of the point of interest at the surface. These parameters can be calculated:

\begin{align*}
    K_{1x} &= -\frac{P}{\pi t} \left[ (\theta_1 + \theta_2) - \frac{1}{2} \sin(2\theta_1) - \frac{1}{2} \sin(2\theta_2) - \frac{2x}{D} \right] \quad (4-3)
\end{align*}
\[ K_{2x} = -\frac{P}{\pi t} \left[ 2\ln \left( \frac{r_2}{r_1} \right) + \frac{1}{2} \cos(2\theta_1) - \frac{1}{2} \cos(2\theta_2) - \frac{2y}{D} \right] \]  
\tag{4-4}

\[ K_{1y} = \frac{P}{\pi t} \left[ (\theta_1 + \theta_2) + \frac{1}{2} \sin(2\theta_1) + \frac{1}{2} \sin(2\theta_2) - \frac{2x}{D} \right] \]  
\tag{4-5}

\[ K_{1y} = -\frac{P}{\pi t} \left[ \frac{1}{2} \cos(2\theta_1) - \frac{1}{2} \cos(2\theta_2) + \frac{2y}{D} \right] \]  
\tag{4-6}

where,

\[ \theta_1 = \tan^{-1} \left( \frac{x}{\frac{D}{2} - y} \right) \]  
\tag{4-7}

\[ \theta_2 = \tan^{-1} \left( \frac{x}{\frac{D}{2} + y} \right) \]  
\tag{4-8}

\[ r_1 = \sqrt{(x)^2 + \left( \frac{D}{2} - y \right)^2} \]  
\tag{4-9}

\[ r_1 = \sqrt{(x)^2 + \left( \frac{D}{2} + y \right)^2} \]  
\tag{4-10}

Equations (4-7) to (4-10) were used to calculate the values for \( \theta_1, \theta_2, r_1 \) and \( r_2 \) which are shown in Figure 4-10. Using equations (4-1) and (4-2), the analytical values for the horizontal and vertical displacements were calculated.
Figure 4-10 Schematic for calculation of elastic properties from DIC analysis

To calibrate this analytical model to the experimental results, the sum of least squared difference method was used.

\[
\text{Least square difference error} = \sqrt{(u_{exp} - u_{calc})^2 + (v_{exp} - v_{calc})^2}
\]  

(4-11)

where \(u_{exp}\) and \(v_{exp}\) are the experimental horizontal and vertical displacements and \(u_{calc}\) and \(v_{calc}\) are the calculated horizontal and vertical displacements based on equation (4-1) and (4-2).

By minimizing the error from equation (4-11), the elastic properties can be predicted.

Here the experimental results from 10M-28 day experiment was used to calculate the elastic properties. Near to the loading points, the values of displacements are affected by localized stresses; therefore, a square with a size of 15 mm x 15 mm was selected at the center of the specimen away from the loading points as shown in Figure 4-10. Through an iterative
process, the least square error was minimized to 0.00075. The corresponding value for the elastic modulus was 1.5 GPa and for the Poisson’s ratio was 0.25. Then, the horizontal stresses, $\sigma_x$ at the center of the specimen were calculated using the Lagrange strain in horizontal ($\varepsilon_x$) and vertical direction ($\varepsilon_y$) with the help of following relationship based on plane stress conditions:

$$
\sigma_x = \frac{E}{1-\nu^2} \left[ \varepsilon_x + \nu \varepsilon_y \right]
$$

(4-12)

where the value for E was 1.5 GPa and value for $\nu$ was 0.25 as calculated above. The strain values were obtained from the DIC analysis. The stress along the line mid-way from the loading point is shown in Figure 4-11. Based on DIC analysis, the stress at the center of the specimen reached as high as 30 MPa. The reason of this high stress is stress concentration at the center of the specimen and excessive strain near to the failure. Full field horizontal stress near to the failure of the specimen is shown in Figure 4-12.

![Figure 4-11 Horizontal stress at the center of the specimen based on DIC analysis](image)
Figure 4-12 Full field horizontal stress at the surface of the specimen near to the failure of the specimen.
5.1 Results from Semi-circular Bend Test without Notch

Semi-circular bend test (SCB) was used to measure the flexural tensile strength of the specimen. The specimen with notch was used to ascertain the fractural properties of the material. Following the experimental setup described in section 3.3.2, three specimens without notch were tested to establish the baseline information. Figure 5-1 shows the load-displacement result for the specimens without any notches.

Figure 5-1 Load displacement curve for SCB specimen without notch
To ascertain the flexural strength of the specimen, the following relationship was used
(Aliha, 2014; Zhang et al., 2021(a)):

\[
\sigma_{\text{tensile}} = \frac{2P}{\pi t D} \left[ 0.146 \left( \frac{t}{D} \right) + 0.8896 \right] \left[ 4.02 \left( \frac{a}{D} \right) + 1.052 \right]
\] (5-1)

where, \( t \) is the thickness of the specimen, \( D \) is the diameter of the specimen and \( s \) is the span
distance (distance between the supports). Based on the dimensions of the specimen used (i.e.,
thickness \( t = 40 \text{ mm} \), diameter, \( D = 76 \text{ mm} \), span distance, \( s = 49 \text{ mm} \)) and the average load
values at the failure, the flexural strength of the specimen was determined to be approximately
equal to 1.71 MPa.

5.2 Calculation of Fracture Toughness using SCB Test with a Notched Specimen

The SCB specimen with a notch was used to calculate the fracture toughness under mode
I loading for the material. Three different notch length to radius (a/R) ratios were used in this
study with the values of 0.2, 0.25 and 0.3. The following relationship was used to calculate the
fracture toughness of the specimen (Kuruppu and Chong, 2012; Zhang et al., 2021(a)):

\[
K_I = \frac{F \sqrt{\pi a}}{2RT} Y_I
\] (5-2)

where \( K_I \) is the stress intensity factor, \( F \) is the load, \( a \) is the notch depth, \( R \) represents the radius
of the specimen, \( T \) denotes the thickness of the specimen, and \( Y_I \) is the dimensionless stress
intensity factor that can be calculated based on the numerical analysis results (Lim et al., 1993;
Kuruppu and Chong, 2012):

\[
Y_I = \frac{S}{D} \left[ 2.91 + 54.39 \left( \frac{a}{R} \right) - 391.4 \left( \frac{a}{R} \right)^2 + 1210.6 \left( \frac{a}{R} \right)^3 - 1650 \left( \frac{a}{R} \right)^4 + 875.9 \left( \frac{a}{R} \right)^5 \right]
\] (5-3)
where $s$ is the span ratio and $D$ is the diameter of the specimen. SCB test can be used to either ascertain mode I fracture toughness, or the mixed mode fracture toughness of the material based on the angle of the notch in the specimen (Lim et al., 1994; Lin et al., 2020). The result of fracture toughness obtained by using equation (5-2) is presented in Figure 5-2.

![Figure 5-2 Mode I Fracture toughness from SCB notched tests](image)

The fracture toughness value for the specimen with $a/R = 0.2$ was 207 kPa$\sqrt{m}$. The average fracture toughness for $a/R = 0.25$ and 0.3 was 217 kPa$\sqrt{m}$ and 232 kPa$\sqrt{m}$, respectively. The average fracture toughness of the material was 220 kPa$\sqrt{m}$. The calculated
fracture toughness of the material lies at the lower end of the spectrum of values for concrete 
(0.2 MPa√m to 1.4 MPa√m) (Callister and Rethwisch, 2008).

5.3 Correlation between Fracture Toughness and Splitting Tensile Strength

The results of mode I fracture toughness obtained from SCB test for 10 M specimens were compared with the tensile strength results obtained from Brazilian Indirect tensile strength test for 10 M specimens after the curing age of 28 days. A correlation can be developed between the fracture toughness and splitting tensile strength of the specimen. Zhang (2002) have
developed a similar kind of correlation between splitting tensile strength, \( \sigma_t \) and mode I fracture toughness, \( K_{IC} \) for different types of rock. The following correlation was obtained based on the experimental results in our study:

\[
\sigma_t = 7.27 \times K_{IC} \tag{5-4}
\]

5.4 Estimation of the Critical Energy Release Rate

The critical energy release rate \( G \) can be calculated from fracture toughness. The units of critical energy release rate are expressed in N/m. The average fracture toughness of the material was estimated as 220 kPa√m. Based on plane stress conditions, the following relationship can be used to estimate the critical energy release rate:

\[
G = \frac{K^2}{E} \tag{5-5}
\]
where $K$ is the fracture toughness and $E$ is the elastic modulus. The elastic modulus of the material was estimated as 1.5 GPa in section 4.3; therefore, the critical energy release rate can be estimated to be equal to 32.2 N/m.

### 5.5 Crack Mouth Opening Displacement

For the notched specimen, extensometer was used to record the crack mouth opening displacement under loading. The results from DIC and extensometer were compared in Figure 5-3. The general trends from DIC analysis and extensometer are similar, confirming the accuracy of the implemented DIC setup. However, the DIC has captured less opening as compared to the extensometer. The crack mouth opening just prior to failure was 0.03 mm, while after reaching the peak load, this value increased to 0.3 mm.

![Figure 5-3 Crack mouth opening displacement based on extensometer and DIC analysis](image)
5.6 Full Field Tensile Strain in Horizontal Direction from DIC Analysis

The full-field strain values for the tensile strain (in horizontal direction) were calculated using DIC for the SCB specimens with a notch. In total, nine virtual strain gauges were virtually included to ascertain the values for the strain throughout the testing of the specimen under loading machine. Figure 5-4 shows the location of the strain gauges at the surface of the SCB specimen. The distance between the strain gauges was 4 mm. Figure 5-5 to Figure 5-7 represent the evaluation of strain throughout the experiment along with the loading of the specimen for a/R values of 0.2, 0.25, and 0.3, respectively. The strain evolution trends near the failure were magnified and embedded within the graph for each case. One point has been identified on the main image and three points were identified inside the magnified image and marked as ‘a’, ‘b’, ‘c’, and ‘d’. The full field strain profile for each marked point for the three curves are provided in Figure 5-8. Strain gauges were labeled from bottom to the top and labelled starting from S-1 to S-9. The S-1 strain gauge was placed at the mid height of the notch and recorded the highest strain values.

![Figure 5-4 Location of virtual strain gauges for SCB Specimen](image)
Figure 5-5 shows the strain graph for SCB specimen with $a/R = 0.2$. The specimen achieved an ultimate load of 2.2 kN. For this specimen, all strain gauges exhibited a similar trend up to $t= 600$ seconds. At this time, the strain at the center of specimen was 0.03%. The lowest strain gauge then deviated from the remaining strain gauges. Near the failure at $t = 871$ seconds, all the strain gauges registered a sudden jump in strain values with the jump ranging from 0.1-1%. The lower strain gauges recorded higher values of the strain as compared to the upper strain gauges. This phenomenon can be attributed to the fact that near the failure, the lower half of the specimen was subjected to tension but the upper half was subjected to compression due to the application of load in three-point bending configuration.

![Strain graph for SCB specimen with $a/R = 0.2$.](image.png)

Figure 5-5 Evaluation of horizontal strain and force throughout the experiment for SCB specimen with $a/R = 0.2$
The strain-time curve for the SCB specimen with $a/R = 0.25$ is shown in Figure 5-6.

Results show that the load at failure is approximately 2.1 kN. All strain gauges showed a similar trend up until $t = 540$ seconds after which the lower most strain gauge (S-9) deviated from other strain gauges. At $t=584$ seconds, the other strain gauges also showed non-linear response. Near the failure at $t = 618$ seconds there was a drastic change in the values of strain from 0.1% to as high as 1%. The strain graph for SCB specimen with $a/R = 0.3$ is presented in Figure 5-7. In this case, the specimen failed at 2.02 kN. Strain values observed by different strain gauges deviated from similar trends at $t=293$ seconds.
Figure 5-7 Evaluation of horizontal strain and force throughout the experiment for SCB specimen with a/R = 0.3

Figure 5-8 shows the full field horizontal strain profile of the specimen at different time frame as marked alphabetically (a, b, c, and d) on the curves in Figure 5-5, Figure 5-6 and Figure 5-7. The points (a-d) are selected based on the strain concentration. At point ‘a’ no strain concentration has occurred, point ‘b’ is the point where the curve changed from linear to non-linear. Point ‘c’ is just prior to the failure and point ‘d’ is after the peak or the failure. The corresponding strain profile for each stage of a-d marked on Figure 5-5, are shown in Figure 5-8(1). Initially, there was no strain concentration but with the increase in the applied load, the strain continued to increase and started concentrating near the tip of the notch. In Figure 5-8(1c), the strain localization at the center of the specimen is clearly visible. This strain concentration started moving towards the upper load platens. Figure 5-8(1d) shows the strain profile just prior to the failure of the specimen.
Figure 5-8(2) shows the strain profile for the SCB specimen with \( a/R = 0.25 \). Initially there was no strain concentration except some near the loading platens. With the increase in the applied load, the strain continued to increase and started concentrating at the tip of the notch. Failure occurs in the flexural mode as expected. The Figure 5-8(3) represents the SCB specimen with \( a/R = 0.3 \). The strain concentration occurred first at the tip of the notch and then moved towards the upper loading platen. In all cases presented in Figure 5-8, the tensile strain concentration occurred near the tip of the notch, which moved upward direction towards the upper loading platen resulting in failure under flexural mode, which is a typical response of SCB test.
Figure 5-8 Full field strain profile for selected points (a, b, c and d) along the curve for SCB specimens (1) a/R = 0.2, (2) a/R = 0.25 and (3) a/R = 0.3
5.7 **Width of the Fracture Process Zone**

The width of the fracture process zone was calculated based on the horizontal displacement field on the either side of the crack. For this analysis, six horizontal lines along the specimen were identified and marked in Figure 5-9 for the SCB specimen with $a/R = 0.2$. The coordinate system was transformed from the center of the specimen to the tip of the notch of the specimen. The displacement profile was plotted up to 20 mm on either side of the notch. The right side of the notch was considered positive, and the left side was assumed as negative distance from the notch tip. More number of the horizontal lines ensured that we are not only analyzing the area near to the tip but also beyond the tip because the higher displacement gradient near the tip can affect the results.

![Figure 5-9 Six Horizontal Lines marked on the specimen to identify the width of fracture process zone (origin of the coordinates system is transformed at the notch tip)](image)

Figure 5-10 shows the horizontal displacement at different heights from the tip of the notch for 10 M specimen with $a/R = 0.2$. Near to the path of the crack, there is a sudden jump in the values of the horizontal displacement. The direction of displacement vector changes across
the crack. By measuring the central portion along the crack, it is evident that the width of this zone is approximately 2.7 mm.

Figure 5-10 Horizontal displacement of specimen at different heights from the notch tip

Figure 5-10 Horizontal displacement of specimen at different heights from the notch tip
CHAPTER 6
CONCLUSION AND FUTURE WORK

6.1 Conclusion and Future Work

The research objectives of this study were to evaluate the splitting tensile strength and flexural strength of the geopolymer concrete, identify critical strain levels for crack initiation, determine the mode I fracture toughness of the material, and ascertain the critical strain energy release rate. The objectives were achieved by using well established methods of Brazilian indirect tensile strength test and Semi-circular bend (SCB) test. Different tasks were performed to accomplish the main objectives of this study. The research tasks performed and the findings of this study are summarized in sub sections.

6.1.1 Research Tasks

The main tasks performed to accomplish the research objectives of this study are summarized as follows:

- Geotechnical characterization of the raw tailings was carried out.
- Circular specimen were prepared for Brazilian indirect tensile strength test and semi-circular specimen with notch and without notch were prepared for semi-circular bend (SCB) test.
  - For Brazilian indirect tensile test, the circular specimens with a diameter of 50 mm and thickness of 30 mm were used. The thickness to diameter ratio was 0.6, which was within the recommended range as proposed by ASTM D3967.
For SCB test, the semi-circular specimen had a diameter 76 mm and thickness of 40 mm. The SCB specimen with notch was tested to ascertain the tensile strength of the specimen. The SCB specimens with a notch were used to study the fractural response of the material. The notch to radius ($a/R$) ratios were varied from 0.2 to 0.3.

- Experimental set up for Brazilian indirect tensile strength test and semi-circular bend test was developed.
- The experimental set up was augmented by Digital image correlation (DIC) set up.
- For DIC analysis, stochastic speckle pattern was drawn on the surface of the specimen using white and black spray paints. To ensure, reduction in excessive glare from the surface of the specimen, matt color paints were sprayed on the specimens. The speckles covered at least three pixels in the image.
- Five specimens for each molar concentration were tested after 7 days and 28 days of curing. Prior to the testing, specimens were placed in an oven at a temperature of 75°C for 24 hours to achieve the dry condition
- For Brazilian indirect tensile test, the specimens were diagonally loaded using MTS loading frame. The loading frame was controlled using Multipurpose TestWare system. The capacity of the loading machine was 100 kN. Flat platens were used to load the specimen. The test was conducted using force control mode with a loading rate of 2 Newtons per second (N/s). Loading machine was configured to get the data point after every 42 milliseconds.
• FLIR grasshopper camera (GS3-U3-23S6M) was used to capture the images throughout the duration of the test. The camera was configured to capture images after every 42 milliseconds thereby achieving a frame rate of approximately 23.8 frames per seconds

• For semi-circular bend (SCB) test, the specimen were loaded in displacement control mode and loading rate was set at 0.05 mm/min.

• Extensometer was also attached at the bottom of the specimen to capture the crack mouth opening displacement. For this purpose, the customized metallic strips were fabricated and attached at the bottom of the specimen on both sides of the notch.

6.1.2 Research Findings

The research findings of this study are summarized as follows:

• Materials rich in silica and alumina can be converted into geopolymerized cementitious materials. The raw tailings used in this study had silica to alumina (Si/Al) ratio of 2.5, which makes them suitable candidate for prospective geopolymerization process.

• At an average 1.5 MPa splitting tensile strength was recorded for 10M specimen at curing age of 28 days and 1.8MPa splitting tensile strength was achieved for 12M specimen at curing age of 28 days.
• The strength of the material increased with the increase in curing age for all molar concentrations of NaOH. However the specimens exhibited brittle response after 28 days of curing age as compared to 7 days of curing age.

• Full field displacement obtained from DIC analysis can be used to estimate the elastic properties of the material i.e., elastic modulus and Poisson’s ratio, $\nu$ through least square error method.

• The average tensile strength achieved from SCB test based on 10M concentration of the specimen was approximately 1.71 MPa.

• The mode I fracture toughness was estimated by conducting SCB test using specimens with a notch. The notch length to the radius ($a/R$) ratio varied from 0.2 to 0.3. The average fracture toughness of the material was $220 \, kPa\sqrt{m}$ or $0.22 \, MPa\sqrt{m}$.

• The average critical energy release rate, $G$ of the material was estimated to be 32.2 N/m.

• Based on the strength requirement for the construction material for different construction projects, this material can be utilized for following purposes:

<table>
<thead>
<tr>
<th>Table 6-1 Strength requirements of construction material for different construction purposes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Construction Purpose</td>
</tr>
<tr>
<td>Concrete building brick</td>
</tr>
<tr>
<td>Concrete for foundation</td>
</tr>
</tbody>
</table>
Table 6-1 Continued

<table>
<thead>
<tr>
<th>Material</th>
<th>Value (Unit)</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type S masonry cement</td>
<td>14.5 (2100)</td>
<td>ASTM C91</td>
</tr>
<tr>
<td>Cement-stabilized base for pavement construction</td>
<td>2 - 5.5 (300 - 800)</td>
<td>Hein et al., 2017 (FHWA)</td>
</tr>
<tr>
<td>Base material for pavement</td>
<td>3.5 (500)</td>
<td>Hurtado 2019 (FDOT)</td>
</tr>
<tr>
<td>Stabilized subgrade material for pavement</td>
<td>1.1 - 3.5 (160 - 500)</td>
<td>CDOT</td>
</tr>
</tbody>
</table>

### 6.2 Recommendations for Future Work

To further analyze the application of this material in real time projects, the behavior of the material may be studied under cyclic loading. The dynamic tensile strength test using split Hopkinson bar system may be conducted.

The effect of the addition of fiber reinforcement may be studied. The chemical reaction of the material with steel reinforcement may also be studied. For environmental concerns, the leaching behavior is important which needs to be studied. Effect of addition of other material like fly ash, blast furnace slag and ordinary Portland cement may be carried out to see the impact on the achieved strength of the specimen. The bond of this cementitious material with the coarse aggregate may also be studied.

The mode II and mixed mode fracture properties may be studied by using semi-circular bend test with notch at different angles. Similarly, the Brazilian indirect tensile test with a center notch may be studied. Crack coalescence behavior may also be studied with two or more notches at the center of the Brazilian disc specimen. The effect of the specimen thickness on the fractural properties of the specimen may also be explored.
The construction material should be durable to withstand the severe environmental conditions over its design life. The material should be further analyzed to assess its performance under different conditions (e.g. immersed in a saline water, immersed in an acidic solution) to document the strength losses (if any) with the targeted strength. The material should also conform the standards/guidelines developed by the agencies like the Environmental Protection Agency (EPA) regarding the leaching of heavy metals, which should be below the desired limits for the widespread use in the Construction industry.
REFERENCES


