Mechanical and fracture behaviour of micro steel fibre-reinforced fly ash-based geopolymer paste containing nano CaCO₃

Thamer Alomayri

Department of Physics, Faculty of Applied Science, Umm Al-Qura University, Makkah, Saudi Arabia

ABSTRACT

The present study has investigated the mechanical properties of micro steel fibre-reinforced fly ash-based geopolymer pastes by adding different contents of nano CaCO₃. Three contents (0%, 1%, 2%, and 3% by weight of fly ash) were evaluated for nano CaCO₃ while the content of micro steel fibres was kept constant at 0.5% by weight of paste. A control mix containing 0.5% micro steel fibres without any content of nano CaCO₃ was fabricated for comparison purposes. The evaluated mechanical characteristics of micro steel fibre-reinforced geopolymer pastes were the compressive strength, flexural strength, elastic modulus, Charpy impact strength, hardness, fracture toughness, toughness modulus, bending stress–strain response, bending load–deflection behaviour, compressive stress–strain response and toughness indices. The outcomes of the present study showed that the utilization of 2% nano CaCO₃ in micro steel fibre-reinforced GPC introduced the optimum mechanical performance for compressive strength, flexural strength, fracture toughness, elastic modulus, toughness modulus, and hardness.

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CONTACT

Thamer Alomayri  tsomayri@uqu.edu.sa

Department of Physics, Faculty of Applied Science, Umm Al-Qura University, Makkah 21955, Saudi Arabia

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1. Introduction

High carbon footprints of cement manufacturing are resulting in environmental pollution. According to an estimate, 5% of global production of carbon dioxide is being produced as a by-product of cement production [1]. Furthermore, the demand for improved cementitious pastes is increasing in the construction industry for high-performing concretes [2–4]. The outstanding features of geopolymer composite (GPC) solidly substantiate their applicability and suitability to replace Portland cement in the construction area [5–8]. By using an alkali in these GPC mixes, the cementitious pastes can be employed as a binder material in concrete construction works. The aluminosilicate compounds present in these pastes can be activated by using an alkali such as sodium hydroxide or sodium silicates to enforce them to perform as a binder material [9]. The performance of GPC is similar to or higher than that of normal concrete as evidenced by a large number of experiments in the previous studies [10–12]. However, there is a need to furthermore explore and improve the mechanical performance of GPC by using innovative ways and techniques so that these advanced cementitious composites can be encouraged and implemented in the construction industry. Moreover, these fly ash-based GPC depicts lower values of strengths in the early ages and shows brittle performance that limit their practical applications in construction industry where relatively higher strengths are required. Therefore, it is of utmost importance for GPC to improve its performance using innovative and advanced techniques.

Various investigations on the mechanical performance of GPC and normal strength plain concrete concluded that the incorporation of different types of fibres enhanced the compressive and tensile properties of these pastes [13–23]. The availability of fibres can be utilized to delay the production of cracks inside the compressive strength by exchanging stresses between the components within the GPC [6,13,24,25]. Later investigation displayed that the mechanical properties of GPC consolidating micro-fibres are higher than that of the control geopolymer without micro-fibres [13–16,20,26]. The dry shrinkage was diminished whereas the compressive quality and setting times were expanded by supplanting the fly ash with micro-fibres in GPC [24]. So also, the investigation about the impact of micro-fibres in fly ash-based GPC firmly substantiated that the enhancement of micro-fibres progressed the compressive quality of GPC by 37% when the fibres were included by 10% of weight [15]. Whereas the enhancement of micro-fibres by 15-30% by weight of paste did not appear any noteworthy enhancement within the compressive quality of GPC. Hence, the micro-fibres can be considered as one of the viable materials as a reinforcement for the production of GPC.

Additionally, numerous later works about the applications of nanomaterials within the development industry have been carried out to make strides in the execution of development composites [27–33]. These works uncovered that the utilization of nanomaterials at lower doses rather than micro materials is compelling to improve the mechanical and strength behaviour of cementitious and Compressive strength. The enhanced properties of developed composites due to the enhancement of nanomaterials may be credited to their ultra-fine sizes coming about within the increasing speed of the geopolymerization response by acting as nanofillers and nucleation locales. Even though a few exploratory examinations have been carried out to investigate the impact of nanomaterials on the mechanical characteristics of plain concrete as a binding material [34,35], exceptionally limited work is accessible within the writing on the applications of nanomaterials in geopolymers. Moreover, the past examinations on the utilization of nanomaterials in developed composites utilized either nano-silica [36,37] or nano-titanium oxide [38,39] having small knowledge on the influence of nano calcium carbonate (i.e. nano CaCO3). However, a recent study by Assaedi et al. [30] has inspected the utilization of nano CaCO3 up to an amount of 3% in GPC. This examination concluded that the mechanical properties of GPC can be made strides by the enhancement of nano CaCO3 up to a measurement of 2% whereas it decreased the mechanical behaviour of GPC by expanding the amount of nano CaCO3 to 3%. Hitherto, no analysis has been done on the combined utilization of micro steel fibres and n-calcium carbonate in GPC. It is necessary to examine the impact of the combined utilization of micro steel fibres and n-calcium carbonate on the mechanical performance of GPC to execute their usage within the development industry. Hence, the current test programme has endeavoured to investigate the impact of distinctive quantities of n-calcium carbonate on the mechanical efficacy of micro steel fibre-reinforced GPC.

The main objective of the present study is to examine and enhance the mechanical efficacy of GPC by using multiple quantities of n-calcium carbonate (0%, 1%, 2%, and 3% by weight) and 0.5% micro steel fibres. Several mechanical characteristics, fracture characteristics, and microstructural characteristics of GPC were aimed to investigate for all four fabricated GPC mixes. The efficiency of accumulating micro steel fibres and n-calcium carbonate on the failure efficacy and bonding efficacy of several ingredients of GPC was examined. This type of cementitious composite can be employed for replacing the cement in concrete construction in order to develop a sustainable and friendly environment. The good results of this study will encourage the practical usage of micro steel fibre-reinforced GPC with n-calcium carbonate in development projects due to the improved mechanical efficacy of these GPC mixes.
2. Experimental procedure

2.1. Constituents materials

Class F fly ash has been employed in the present study as an aluminosilicate precursor. The chemical composition of fly ash is reported in Table 1. The SEM micrographs of fly ash have been shown in Figure 1(a).

The binary alkali activator used for the fabrication of GPC consisted of sodium silicate (Na$_2$SiO$_3$) and sodium hydroxide (NaOH). The purity of NaOH was 98% and achieved in form of flakes while the Na$_2$SiO$_3$ was achieved in form of a solution. The Na$_2$SiO$_3$ contained three different constituents comprising 14.7% Na$_2$O, 29.4% SiO$_2$, and 55.9% H$_2$O. The SEM analysis of CaCO$_3$ is shown in Figure 1(b). The particle size distribution of fly ash is shown in Figure 1(c). The micro steel fibres were added for the improvement of the tensile behaviour of the GPC. The length, tensile strength, and the elastic modulus of micro steel fibres were 13 mm, 2500 MPa, and 210 GPa, respectively. Table 2 reports various properties of micro steel fibres employed in the present experimental programme.

Four different GPC pastes (illustrated in Table 3) with changing quantities of n-calcium carbonate by mass of the fly ash were created and observed for flexural strength, toughness indices, compressive strength, impact strength, toughness modulus, compressive

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**Table 1.** Chemical composition of class F fly ash (LOI is the loss on ignition).

| Compound | Composition (%) | Compound | Composition (%) |
|----------|----------------|----------|----------------|
| SiO$_2$  | 60.1           | Na$_2$O  | 0.76           |
| Al$_2$O$_3$ | 25.8         | MgO      | 0.67           |
| CaO      | 2.53           | SO$_3$   | 0.15           |
| Fe$_2$O$_3$ | 2.59          | LOI      | 1.46           |
| K$_2$O   | 2.03           | –        | –              |

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**Figure 1.** SEM analysis of (a) fly ash (b) nano-CaCO$_3$ (c) particle size distribution of fly ash.

**Table 2.** Various physical and chemical features of micro steel fibres.

| Shape     | Density (kg/m$^3$) | Specific gravity (g/cm$^3$) | Tensile strength (MPa) | Elastic modulus (GPa) | Diameter (mm) | Length (mm) | Aspect ratio |
|-----------|--------------------|----------------------------|------------------------|-----------------------|----------------|-------------|--------------|
| Straight  | 7850               | 7.8                        | 2500                   | 210                   | 0.12           | 13          | 108          |
stress–strain behaviour, bending load-deflection curves, elastic modulus, fracture toughness, bending stress–strain behaviour, and hardness. The proportion of binary alkali solution to fly ash was kept at 0.45 while the proportion of Na₂SiO₃ answer for NaOH solution was kept at 2.5 for all GPC pastes [40]. The NaOH mixture having a concentration of 10 M was obtained by dissolving the pellets in water and blending in with the Na₂SiO₃ solution around 24 h before the blending cycle of geopolymer pastes. All GPC pastes were built up with 0.5% micro steel fibres and n-calcium carbonate was utilized at the doses of 1%, 2%, and 3%, respectively due to the better efficacy of steel fibres at this quantity [17,41]. Table 3 presents the GPC mixes developed in the present study (Figure 2).

### 2.2. Sample preparation and curing

A mechanical blender having a volumetric limit of 0.15 m³ and an upset speed of 20 rev/min. was utilized for the blending of GPC pastes. While completing the blending cycle of geopolymer blends, the dry blending of fly ash and micro steel fibres was done for five minutes at a low speed. After dry blending of these two materials, the blending of soluble base arrangement containing various amounts of nano-CaCO₃ was completed at a fast speed of 20 rev/min. This blending process proceeded until a homogenous geopolymer blend was acquired. The new pastes have been filled into the molds and put in an oven curing process at a temperature of 80°C for the following 24 h [40]. After taking out the samples from the oven, they were demoulded and the curing process was begun at room temperature for the following 28 days to acquire a definitive strength. Geometry of cubic and rectangular specimens is represented in Figure 3. The development process of geopolymer nanocomposites is represented in Figure 4.

### 2.3. Testing procedures

#### 2.3.1. Compressive strength

A sum of 24 cube samples having measurements of 20 mm × 20 mm × 20 mm for four diverse GPC mixes were created and observed to fail following the methodology of ASTM D 695 [40]. A sum of six samples was observed for each geopolymer paste having 0%, 1%, 2%, and 3% nano CaCO₃, respectively. The current examination experimented with every one of the 24 samples for the compressive strength and the average outcomes for each geopolymer blend were accounted for and discussed in this study. For measuring the compressive strength of samples, a maximum load taken from the compressive stress–strain behaviour was divided by the total area of the samples.

#### 2.3.2. Flexural strength

Like the samples for measuring the compressive strength of four GPC mixes, 24 samples with measurements of 60 mm × 20 mm × 20 mm were created for estimating the flexural strength of the GPC composites. The three-point bending testing of the created samples having 0%, 1%, 2%, and 3% nano CaCO₃ were performed

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**Table 3. Mix design of all four mixes.**

| Geopolymer mix | Fly ash (g) | Sodium hydroxide (g) | Sodium silicate (g) | Micro steel fibre (wt.%) | Nano-CaCO₃ (g) | Nano-CaCO₃ (wt.%) |
|----------------|-------------|----------------------|--------------------|-------------------------|----------------|------------------|
| SGPC-0%CaCO₃   | 1000        | 214                  | 536                | 0.5                     | 0              | 0                |
| SGPC-1%CaCO₃   | 1000        | 214                  | 536                | 0.5                     | 10             | 1                |
| SGPC-2%CaCO₃   | 1000        | 214                  | 536                | 0.5                     | 20             | 2                |
| SGPC-3%CaCO₃   | 1000        | 214                  | 536                | 0.5                     | 30             | 3                |

**Table 4. Flexural toughness indices of GPC mixes.**

| GPC mix label | Maximum load (kN) | Maximum stress (MPa) | Mid-span deflection (mm) | Flexural toughness indices | Increase in load (%) | Increase in deflection (%) | Increase in indices (%) |
|---------------|-------------------|----------------------|--------------------------|---------------------------|----------------------|--------------------------|-------------------------|
|               | l₅                | l₁₀                  | lᵣ                      | I₅                        | I₁₀                  | Iᵣ                      | I₅                      |
| SGPC-0%CaCO₃  | 0.27              | 3.19                 | 1.31                     | 34                        | 100                  | 145                      | –                       |
| SGPC-1%CaCO₃  | 0.38              | 3.33                 | 1.39                     | 41                        | 136                  | 148                      | 40.74                   |
| SGPC-2%CaCO₃  | 0.59              | 5.47                 | 1.46                     | 35                        | 110                  | 154                      | 118.52                  |
| SGPC-3%CaCO₃  | 0.34              | 3.27                 | 1.41                     | 39                        | 126                  | 163                      | 25.93                   |

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*Figure 2. Geometry of cubic and rectangular specimens.*
following the methods of ASTM C-293 [41]. Six samples for each GPC blend were observed and their average outcomes were utilized in the current examination for the conversations. The loading rate for the three-point bending testing was fixed at 1 mm/min. A definitive bending loading was acquired from the bending stress–strain behaviour that was utilized for measuring the flexural strength dependent on the measurements and the span of samples. The flexural strength ($\sigma_F$) was estimated by employing the following equation.

$$\sigma_F = \frac{3 P_m S}{2 BW^2}$$

where $P_m$ describes the maximum bending load at crack extension, $S$ describes the span of the samples, $B$ describes the width of samples, and $W$ describes the thickness or depth of samples.

2.3.3. Hardness

The hardness of a material is the measure of the resistance to localized plastic deflections made by either mechanical abrasion or indentation. In the present study, the hardness of GPC was measured on the Rockwell H scale by utilizing an Avery Rockwell hardness tester according to the methodology proposed by ASTM D785 [42]. A total of 24 samples were fabricated i.e. six samples were constructed for each GPC mix. The average measurements were used for determining the hardness of GPC pastes in this study. Before testing the specimens, their surface was cleaned, polished, and smoothened to secure more accurate measurements. All the specimens were observed for hardness at room temperatures.

2.3.4. Charpy impact strength

Six samples having a length of 40 mm were fabricated from each mix to determine the impact strength of all GPC mixes. A Zwick Charpy impact testing instrument with a 1 J pendulum hammer was employed for measuring the Charpy impact strength of manufactured samples. The Charpy impact strength ($\sigma_I$) was measured using Eq. (2) [30], where $E$ shows the impact energy required to break a sample with a ligament of area $A$.

$$\sigma_I = \frac{E}{A}$$

2.3.5. Fracture toughness

The fracture toughness of a material is a measurable way of presenting the resistance of that material to crack propagation. The fracture toughness of the GPC was evaluated by testing the samples having the measurements of 60 mm $\times$ 20 mm $\times$ 20 mm. Like the samples for measuring the compressive and flexural values of four GPC, 24 samples were created for estimating the crack strength of the GPC composites. The average outcomes were discussed in the current examination. Initially, a break having a proportion of 0.4 for its length to thickness was created in the samples. At that point, the samples were set in the flexural testing equipment for three-point flexural testing for measuring the fracture toughness at a loading frequency of 0.5 mm/min. The measurements of the fracture toughness were performed using Equation (3).

$$K_{IC} = \frac{P_m S}{BW^2} f\left(\frac{a}{W}\right)$$

where $K_{IC}$ describes the fracture toughness, $P_m$ describes the maximum bending load at the crack, $S$ describes the span of samples, $B$ describes the width of samples and $f\left(\frac{a}{W}\right)$ is a coefficient that is determined by employing Equation (4) [43]. Additionally, $a$ describes the initial crack length and $W$ describes the initial crack thickness.

$$f\left(\frac{a}{W}\right) = \frac{3(a/W)^{1/2}[1.99 - (a/W)(1 - a/W)]}{2(1 + 2a/W)(1 - a/W)^{3/2}} \times [2.15 - 3.93a/W + 2.7a^2/W^2]$$
2.3.6. SEM analysis
Scanning electron microscopy (SEM) has been utilized for analyzing the microstructural properties containing the failure system and surface morphology of cracked GPC samples. The observed samples were dried out in a vacuum desiccator for 48 h and afterward stuck to an aluminium stub. To control the charge delivered by the electrons, the observing samples were covered with gold. The broken surface process and failure development performance of geopolymer pastes with micro steel fibres and nano CaCO₃ were noticed utilizing a magnifying instrument and the technical explanations behind their failure were assessed.

3. Results and discussion
3.1. Compressive strength
The impact of adding different amounts of nano CaCO₃ (0%, 1%, 2%, and 3%) to geopolymer paste with 0.5% micro steel fibres on the compressive strength of composite pastes is depicted in Figure 4. It very well may be inferred that the enhancement of nano CaCO₃ prompts an increase in the compressive strength up to an amount of 2% while the enhancement of substance of n-calcium carbonate by 3% reduces the compressive strength of GPC blend altogether due to the agglomeration and dilution effect making fewer hydration by-products [40]. GPC having the amount of 1% (SGPC-1%CaCO₃) and 2% (SGPC-2%CaCO₃) nano CaCO₃ showed the increases of 14.18% and 30.12% in compressive strength, individually contrasted with that of the paste without nano CaCO₃. This can be attributed to the explanation that the low amounts of nano CaCO₃ can adequately go about as a filler to seal the nano and miniature voids between the geopolymer paste and micro steel fibres. While the enhancement of 3% nano CaCO₃ brought about an improvement of 3.57% in the compressive strength of GPC blends having 0.5% micro steel fibres. The improvement of the compressive strength with the enhancement of nano CaCO₃ can be credited to the ability of enough nanomaterials to impede the nano and miniature voids joined with the capacity of the nano CaCO₃ to revive the geopolymerization cycle. The geopolymerization cycle can be speed up by the enhancement of nano CaCO₃ because of their ability to go about as nucleation sites for creating the beneficial products inside the GPC [29,44,45]. Subsequently, to get the optimal outcomes for the compressive strength of micro steel fibre built up geopolymer paste, 2% of nano CaCO₃ as can be viably utilized. Furthermore, the reduction in compressive strength at 3% nanoparticles may be associated to the agglomeration of nano CaCO₃ resulting to a weaker matrix.

Generally, the compressive strength estimations got for the different GPC having nano CaCO₃ (0%, 1%, 2%, and 3%, independently) without micro steel fibres are less than that of the current investigation estimations with micro steel fibres [30]. This determinedly proves the proficiency of adding 0.5% micro steel fibres to geopolymer pastes with the different substances of nano CaCO₃. The improvement in the compressive strength of geopolymer pastes with the enhancement of micro steel fibres might be credited to a more densified microstructure and a comparable higher axial compressive loads of pastes as demonstrated in Figure 4. Hence, it is presumed that the compressive strength of GPC can be improved by at the same time adding a bigger measure of nano CaCO₃ (up to 3%) and micro steel fibres. The relative compressive qualities of GPC paste with the different substances of nano CaCO₃ contrasted and the control blend without CaCO₃ (SGPC-0%CaCO₃) are presented in Figure 5. The compressive strength of GPC blend with 1%, 2%, and 3% substance of nano CaCO₃ and 0.5% micro steel fibres were 114.18%, 130.12%, and 103.58% of the compressive strength of control blend having 0.5% micro steel fibres and no substance of nano CaCO₃, separately.

![Figure 4](image_url). Influence of different quantities of nano CaCO₃ on the compressive strength and compressive loads of GPC.
3.2. Compressive stress–strain curves

The compressive stress–strain curves of GPC paste samples with 0%, 1%, 2%, and 3% nano CaCO$_3$ containing 0.5% micro steel fibres are presented in Figure 6. The control samples (SGPC-0%CaCO$_3$) depicted less compressive stiffness than the samples containing different measures of nano CaCO$_3$ uncovering that the increasing amount of nano CaCO$_3$ improves the compressive stress making the GPC paste generally solid to bear higher compressive loads at lower values of compressive strains as depicted in Figure 6. The compressive stress–strain behaviour likewise portrays that the increment in the substance of nano CaCO$_3$ improves the values of compressive stress at lower values of compressive strain. This might be credited to denser geopolymer paste and speed up cycle of geopolymerization by the increasing amount of nano CaCO$_3$ because of their ability to go about as nucleation sites for creating the strengthening items inside the GPC portraying higher stiffness [29,44,45]. Besides, the samples containing the different substances of nano CaCO$_3$ introduced higher values of compressive strength at lower compressive strains. This may likewise be credited to the higher stiffness of samples before the post-top stage that brought about the breaking of geopolymer pastes in the post-stacking locale of stress–strain curves because of the greater brittle behaviour of the pastes having nano CaCO$_3$.

The samples containing the different substances of nano CaCO$_3$ announced higher slopes of stress–strain curves because of the higher stiffness of the geopolymer pastes. Subsequently, the elastic modulus of these pastes was bigger than that of the control blend (SGPC-0%CaCO$_3$) as demonstrated in Figure 7. The elastic modulus of sample SGPC-1%CaCO$_3$ was 25.7% higher than the control blend (SGPC-0%CaCO$_3$) having 0.5% micro steel fibres and no substance of nano CaCO$_3$. Also, the elastic modulus of samples SGPC-2%CaCO$_3$ was 51.4% larger than the control blend (SGPC-0%CaCO$_3$) having not placated with nano CaCO$_3$. The elastic modulus of sample SGPC-3%CaCO$_3$ was 29.3% higher than
the control blend (SGPC-0%CaCO₃) having 0.5% micro steel fibres and no measure of nano CaCO₃. Accordingly, the sample with 2% nano CaCO₃ revealed the most elevated stiffness with the most elevated elastic modulus of 424 MPa. This might be credited to the viable mix of 2% nano CaCO₃ and 0.5% micro steel fibres in the GPC paste.

### 3.3. Flexural strength

The impact of adding 0.5% micro steel fibres and different substances of nano CaCO₃ (0%, 1%, 2%, and 3%) to geopolymer paste on the flexural strength of composite pastes is shown in Figure 8. The flexural strength of a composite material estimates the ability of that composite material to counter the bending when presented to bending loads. The average exploratory estimations of six examples from each GPC blend depicted that the flexural strength of the pastes intensified up to 2% nano CaCO₃ content before a decrease in the flexural strength began. In any case except 2% amount, the flexural strength of all pastes with nano CaCO₃ was higher than that of the control blend (SGPC-0%CaCO₃) while 2% reported the highest values of flexural strength. This enhancement in the flexural strength of manufactured blends might be credited to the improvement of the microstructure with the nano CaCO₃ particles and the accelerating of the geopolymerization interaction prompting the densification of the composite blend. Moreover, the bending loads of samples expanded by the increasing of nano CaCO₃ particles because of the increment in the bending stiffness of pastes as demonstrated in Figure 8.

The agglomeration of nano CaCO₃ may be credited to the reduction in flexural strength at 3% nanoparticles. In any case, the compressive strength results (revealed in Figure 4) showed similar conduct for 1%, 2%, and 3% nanoparticles. Accordingly, the authors prescribed a broad exploratory programme to additionally investigate the conduct of such pastes by adding nanoparticles to comprehend this behaviour. Comparative estimations were seen in a past report [30] with unreinforced GPC pastes having up to 3% nano CaCO₃ particles. The nano CaCO₃ amounts of 1% and 2% also displayed a increased flexural strength without micro steel fibres in the former study [30]. In any case, the GPC with 0.5% micro steel fibres depicted an expanded flexural strength at all measurements of 1%, 2%, and 3% nano CaCO₃ compared with the control blend (SGPC-0%CaCO₃). The use of 2% nano CaCO₃ introduced the ultimate increase in the flexural strength of GPC pastes. The testing carried out by Alomayri [46] illustrated that the enhancement of nano alumina into GPC at the measures of 1% and 2% improved the flexural strength by 5.6% and 44.5%, separately. However, the use of nano CaCO₃ at the substance of 1% and 2% in the current examination prompted an improvement in flexural strength by 4.39% and 71.47%, individually. Accordingly, the use of nano CaCO₃ along with the support of micro steel fibres is more employable in improving the flexural strength related to using just nano alumina. Figure 9 reports the relative flexural strength of geopolymer pastes with the different substances of nano CaCO₃ compared with the control blend without CaCO₃. The flexural strength of GPC blend in with 1%, 2%, and 3% substance of nano CaCO₃ and 0.5% micro steel fibres were 104.39%, 171.47%, and 102.51% of the flexural strength of control blend having 0.5% steel strands and no substance of nano CaCO₃, separately.

### 3.4. Bending stress–strain behaviour

The bending stress–strain curves of GPC pastes having different substances of nano CaCO₃ with 0.5% micro steel fibres are presented in Figure 10. The control sample (SGPC-0%CaCO₃) showed the lower benefits of bending stresses at expanded strains announcing less stiffness than the samples containing different measures of nano CaCO₃ (as demonstrated in Figure 11) indicating that the addition of nano CaCO₃ improves the bending stresses creating a stiffer GPC paste to endure higher bending loads at lower bending strains. The geopolymer paste with 2% nano CaCO₃ portrayed the higher bending stiffness with the most noteworthy toughness modulus. The bending stress–strain behaviours likewise depict that the improvement in the measure of nano CaCO₃ expands the benefits of bending loads at lower bending strains. The expanded measure of bending stresses at lower benefits of bending strain might be related with denser GPC paste and speed up the improvement of geopolymerization measure by the joining of nano CaCO₃ because of their capacity to go about as nucleation sites for creating the advantageous items inside the pastes depicting higher bending stiffness [29,44,45]. Besides, the samples including different measures of nano CaCO₃ revealed a higher decrease of bending strength at lower benefits of bending strains. This may likewise be related to the higher stiffness of tests before the post-collapse performance that prompted the breaking of composite pastes in the post-collapse phase of stress–strain behaviour because of higher brittle conduct of the GPC containing nano CaCO₃.

Figure 11 shows the measurements of toughness modulus of different GPC observed in the current investigation. The samples containing 2% substance of nano CaCO₃ detailed higher slopes of stress–strain curves because of the higher stiffness of the geopolymer pastes. Accordingly, the toughness moduli of these pastes were bigger than that of the control blend (SGPC-0%CaCO₃). The toughness modulus of samples SGPC-1%CaCO₃ was 15.5% higher than the control
Figure 7. Elastic moduli and compressive stiffness of micro steel fibre-reinforced GPC paste with various contents of nano CaCO₃.

Figure 8. Influence of different quantities of nano CaCO₃ on the flexural strength and bending loads of mixes.

Figure 9. Relative flexural strength of micro steel fibre-reinforced GPC paste with various contents of nano CaCO₃.
portraying higher bending stiffness of GPC pastes [29,45].

3.5. Load-deflection curves and flexural toughness indices

Figure 12 shows the bending load-deflection curves for the micro steel fibre-built-up GPC paste samples comprising of the various substance of nano CaCO$_3$. It was seen that a maximum load of geopolymer paste without nano-CaCO$_3$ essentially increment after the incorporation of nanoparticles. The area under the bending curve depicts that the geopolymer containing different nano CaCO$_3$ substances accomplished higher deflection compared with the geopolymer without nano-CaCO$_3$. This improvement nearby under the area can be related to the influence of nanoparticles bringing about crossing over the matrix cracking and diminishes crack development rate. Right when the created cracks approach the steel strands, they are refracted at the interface between the geopolymer matrix and fibre. In the bending test, geopolymer with a lower substance of nano-CaCO$_3$ (2%) shows the clear failure performance. By expanding the bending loads, the GPC nanocomposites show elastic failure and observable deflections in the underlying stage. In the wake of intersection as far as possible, the loading makes plastic distortions until the maximum load is reached. After this stage, the applied loading is continuously decreased with the upgrade in the deflection, making a long tail, prompting higher fracture toughness values. Among various nano-CaCO$_3$ substances, the deflection of the geopolymer grid containing 2% nano-CaCO$_3$ was most elevated than others. The improvement in uprooting might be credited to a decent fibre-matrix bond, which upgrades the capacity of these pastes to retain energy by beginning tortuous pathways for the crack spread that builds the bending strength [47]. These load-deflection bends decidedly prove that the addition of nano-CaCO$_3$ upgrades the mechanical presentation of the geopolymer mix; they additionally suggest that an increase in nano-CaCO$_3$ content up to 2% is truly significant for improving the crack resistance of a micro steel fibre-built up geopolymer matrix.

Nonetheless, deflections diminished when the nano-CaCO$_3$ content upgraded beyond 2%. It has been reasoned that the addition of nano-CaCO$_3$ results in attractive redirection is an outcome of the improvement in fibre-matrix bond, yet its enhancement likewise delivered a fragile composite appearance lower deflections for all samples. The addition of nano-CaCO$_3$ improved the connection among micro steel fibres and geopolymer matrix, however, decreased the energy absorption measure given by the pull-out and deboning process of fibres. This depicts that the consolidation of 3% nano-CaCO$_3$ produces a stronger interfacial bond in GPC paste as compared with that made utilizing 2% nano-CaCO$_3$. Because of the presence of a more fragile interfacial grip, the crack commencement, and propagation bringing about the more prominent pull-out lengths of micro steel fibres. Then again, in the fibre matrix, the break of fibres is more dominant than their pull out. Interestingly, when the addition of nano-CaCO$_3$ expanded from 2% to 3%, the bending deflection decreased.

The flexural toughness indices of micro steel fibre-reinforced GPC samples were measured following the procedure of ASTM C1018 [48]. Three toughness indices consisting of $I_5$, $I_{10}$ and $I_{\text{failure}}$ were observed to explore the flexural toughness. Index $I_5$ is demarcated as the ratio of three times the area of bending load-deflection response up to the deflection at the initiation of the first

![Figure 10. Bending stress-strain curves of micro steel fibre-reinforced GPC paste with various contents of nano CaCO$_3$.](image)
Figure 11. Toughness moduli and bending stiffness of micro steel fibre-reinforced GPC paste with various contents of nano CaCO$_3$.

Figure 12. Load-deflection curves of micro steel fibre-reinforced GPC mixes.

Figure 13. Toughness indices ($I_5$, $I_{10}$, and $I_{\text{failure}}$) for all micro steel fibre-reinforced GPC specimens.

crack to the area of the curve at the first crack. Correspondingly, Index $I_{10}$ is demarcated as the ratio of 5.5 times the area of bending load-deflection response up to the deflection at the initiation of the first crack to the area of the curve at the first crack. Furthermore, $I_{\text{failure}}$ is the ratio of the area of whole load-deflection response curves to the area of curves up to the deflection at the initiation of first cracks. The flexural toughness indices of micro steel fibre-reinforced GPC samples are presented in Table 4.
It has been observed from the literature studies that GPC without any fibres incorporation is weaker in tension and displays brittle nature under compressive and tensile loading. Thus, they exhibit lower toughness and require to be combined with fibres [30]. Figure 13 displays the toughness indices ($I_5$, $I_{10}$, and $I_{\text{failure}}$) for all micro steel fibre-reinforced GPC samples. The addition of 1% nano-CaCO$_3$ improved the toughness indices of micro steel fibre-reinforced GPC samples by 20.58%, 36%, and 2% for $I_5$, $I_{10}$, and $I_{\text{failure}}$, respectively associated with the samples without nano-CaCO$_3$. Correspondingly, the addition of 2% nano-CaCO$_3$ improved the toughness indices of micro steel fibre-reinforced GPC samples by 2.94%, 10%, and 6.2% for $I_5$, $I_{10}$, and $I_{\text{failure}}$, respectively related with the control samples. This shows that the 1% nano-CaCO$_3$ offered improved toughness indices than 3% nano-CaCO$_3$. Besides, the addition of 3% nano-CaCO$_3$ improved the toughness indices of micro steel fibre-reinforced GPC samples by 14.7%, 26%, and 12.41% for $I_5$, $I_{10}$, and $I_{\text{failure}}$, respectively related with the control samples. This may be associated with a stronger bond between the fibres and GPC paste due to the high amounts of GPC gel having a low content of nano-CaCO$_3$ possessing the fibre-matrix adhesion [47]. Therefore, the ultimate toughness indices ($I_5$ and $I_{10}$) were secured using 1% nano-CaCO$_3$.

3.6. Fracture toughness

The fracture toughness of composite materials is a decent indication of their ability to counter the spreading of break width inside the composites material. Figure 14 depicts the values of fracture toughness of each of the four micro steel fibre-built-up GPC containing 0%, 1%, 2%, and 3% nano CaCO$_3$. It has been portrayed by Figure 14 that the geopolymer pastes with nano CaCO$_3$ depicted higher values of fracture toughness than the control samples (SGPC-0%CaCO$_3$). In addition, the fracture toughness values of all samples paying little attention to nano CaCO$_3$ content were larger than 0.4 MPa.m$^{1/2}$. The expanded values of fracture toughness might be related to the presence of steel strands prompting the improvement in it by giving a stiffer lattice. The bridging property of micro steel fibres in the GPC paste expanded the fracture toughness of pastes which moves the interior stresses with the matrix prompting the improvement in the opposition against the break initiation and propagation.

The fracture toughness of GPC mixes is constantly enhanced by growing the amount of nano CaCO$_3$ from 1% to 2% reporting the maximum fracture toughness i.e. 0.54 MPa.m$^{1/2}$ at 2% nano CaCO$_3$. The fracture toughness of all GPC mixes comprising of nano CaCO$_3$ is more prominent than that of the pastes having no substance of nano CaCO$_3$ demonstrating that the nano CaCO$_3$ improved the strength of the geopolymer pastes. The fracture toughness of the geopolymer sample SGPC-1%CaCO$_3$ was 4.4% higher than the control sample SGPC-0%CaCO$_3$. Also, the fracture toughness of the geopolymer sample SGPC-2%CaCO$_3$ was 20% higher than the control sample SGPC-0%CaCO$_3$. The utilization of 3% nano CaCO$_3$ decreased the fracture toughness compared with different substances for a sample having 1% and 2% contents. The sample SGPC-3%CaCO$_3$ depicted an increment of 13.3% in fracture toughness compared with that of the control sample SGPC-0%CaCO$_3$. The improvement in the fracture toughness of geopolymer pastes because of the enhancement of nano CaCO$_3$ might be credited to the capacity of nanoparticles of CaCO$_3$ to improve the interfacial zone between the geopolymer and micro steel fibres prompting the upgrade in the interfacial zone of steel strands inside the GPC paste. The nanoparticles inside the paste improve the bond strength between geopolymer paste and fibres strands bringing about a denser and harder paste to oppose the breaking interaction [49]. In this way, the addition of
nanoparticles speeds up the geopolymerization phenomenon and refines the microstructure of geopolymer paste by improving the connection between elements of composite matrix and micro steel fibres.

3.7. Hardness

The hardness of GPC paste is the capacity of this paste to bear the localized plastic distortions. Figure 15 depicts the impact of the enhancement of 0%, 1%, 2%, and 3% measurements of nano CaCO₃ on the hardness of micro steel fibre-built up GPC pastes. The outcomes illustrated that the addition of 1%, 2% and 3% nano CaCO₃ improved the hardness of geopolymer pastes. As compared with the control sample SGPC-0%CaCO₃, the sample SGPC-1%CaCO₃ introduced an increment of 1.2% in hardness while the example SGPC-2%CaCO₃ detailed an improvement of 3.5% in hardness of pastes. The sample SGPC-3%CaCO₃ revealed an increment of 8.1% in hardness contrasted and the control test. These expanded values of hardness might be credited to the high strength of nano CaCO₃ particles communicated consistently to the GPC framework bringing about a high interfacial region between geopolymer lattice and nanoparticles during the transmission of stresses [50]. Moreover, the enhancement of 3% nano CaCO₃ brings the nanoparticles a lot nearer to one another creating a stiffer paste to show higher values of hardness. The nanoparticles scatter all through the matrix and consume the vacant spaces between chains of GPC that improve the obstruction of the matrix to deformations and the development of cracks. Alomayri [46] depicted that the geopolymer paste without consolidation of strands and nanoparticles portrayed the hardness of 80 HRH. Accordingly, in the current examination, the higher values of hardness greater than 80 HRH may likewise be credited to the enhancement of micro steel fibres in GPC paste depicting the positive impact of micro steel fibre in the composite paste.

3.8. Impact strength

The Charpy impact strength of different micro steel fibre-built up GPC with the various substance of nano CaCO₃ is shown in Figure 15. The outcomes demonstrated that the impact strength of GPC paste is improved by the enhancement of nano CaCO₃ particles. The effect energy of nanocomposites is expanded persistently up to the enhancement of 2% nano CaCO₃ after which the effect energy is diminished however bigger than the control sample without nano CaCO₃. The greatest impact strength of micro steel fibre-built up GPC paste is achieved utilizing 2% nano CaCO₃. The impact strength of sample SGPC-1%CaCO₃ was higher than that of control sample SGPC-0%CaCO₃ (with 35.9% enhancement) without nano CaCO₃. The impact strength of sample SGPC-2%CaCO₃ was 63.6% bigger than that of the control sample SGPC-0%CaCO₃. Additionally, the effect strength of sample SGPC-3%CaCO₃ was 10.9% more noteworthy than that of the control sample SGPC-0%CaCO₃ without nano CaCO₃. The failure of the composite paste occurred because of the agglomeration of nanoparticles inside the paste proceeding as a beginning place of break. This might be credited to the explanation that the nanoparticles inside the GPC improve the bond strength among matrix and supporting strands prompting a denser and strong grid to counter the breaking process of geopolymer paste [49]. A composite matrix can work viably if the load transferring properties between the nanoparticles are acting appropriately. Realizing that the capacity of CaCO₃ nanoparticles relies on the load-moving component from the composite matrix to CaCO₃, the interfacial toughness between the lattice and nano CaCO₃ has extraordinary significance. Besides, the enhancement of micro steel fibres improves the load moving component and interface by upgrading the bond strength and giving a connecting process between elements of paste. Hence, a more

![Figure 15. Hardness and impact strength of micro steel fibre-reinforced GPC paste with various contents of nano CaCO₃.](image-url)
grounded interfacial surface will give a superior load transferring and improve the impact strength. In addition, the connecting property of nano CaCO3 and steel strands brought about an upgrade in the Charpy impact energy of micro steel fibre-built up GPC paste with different amounts of nano CaCO3.

3.9. Microstructural properties

After completing the fracture toughness test, SEM micrographs were utilized to analyze the microstructure of GPC paste samples consolidating different substances of nano CaCO3. Figure 16 shows the SEM examination of samples containing 0%, 1%, 2%, and 3% nano CaCO3. The SEM micrographs showed that a large geopolymerization response was done because the majority of the unreacted fly ash was utilized for the formation of C–S–H gel leaving a significant measure of it in the microstructure of GPC [40]. The consolidation of nano CaCO3 speed up the geopolymerization cycle shaping C–S–H gel. These C–S–H intensifies spread between the fly ash particles and work as seeds even in the pore space to give a more compacted and denser microstructure showing great attachment among nanoparticles and fastener stage [30,51,52]. Besides, the addition of nano CaCO3 prompted a denser matrix by making up for the shortcomings between fly ash particles. Some of the fly ash particles remained unreacted within the GPC matrix. By expanding the substance of nano CaCO3, the breaks decreased giving the denser paste. The nanoparticles of CaCO3 functioned as an extension with the load transferring instrument and conceded the propagation of cracking. At this stage, nano CaCO3 assisted with forestalling the commencement of nano-cracks by making the connection between the products got from the geopolymer hydration measure. The SEM micrographs likewise show that the micro steel fibres and geopolymer matrix have a decent holding that might be related to the ability of nano CaCO3 to refine and give a denser microstructure of geopolymer pastes. An improvement in fibre-rich areas shows that the GPC matrix is adequately restrained with micro steel fibres and the stress is more uniformly distributed resulting to enhancement in composite stiffness. SEM micrographs prevented the pull-out of micro steel fibres due to improvement in fibre-matrix interfacial bonding by the increased contents of nanoparticles. The occurrence of nano-filler was found to improve the adhesion between the GPC matrix and the micro steel fibres.

4. Conclusions

The following are the main findings of the present work:

(1) The maximum COMPS of micro steel fibre-reinforced GPC pastes was attained utilizing 2% n-calcium carbonate that was 3.55% of that of the control paste having no n-calcium carbonate.

**Figure 16.** SEM micrographs of: (a) control specimen SGPC-0%CaCO3 (b) SGPC-1%CaCO3 (c) SGPC-2%CaCO3 (d) SGPC-3%CaCO3.
This enlargement can be attributed to the ability of enough nanoparticles to hinder the nano and microvoids alongside the capacity of the n-calcium carbonate to speed up the geopolymerization process.

(2) The maximum values of FLEXS were achieved utilizing 2% n-calcium carbonate in the GPC mix that was about 71.47% of that of the control sample. This increment in the FLEXS of GPC mixes may be accredited to the improvement of the microstructure with the n-calcium carbonate particles and the accelerating of the geopolymerization interaction prompting the densification of the composite lattice. The TOUGI announced the most elevated values for GPC utilizing 3% n-calcium carbonate.

(3) The FRACT of the GPC mix was expanded by the addition of n-calcium carbonate depicting the most elevated values at 2% n-calcium carbonate that was 44.4% larger than the control mix. The IMPAS of GPC paste was enhanced by the accumulation of n-calcium carbonate particles. This is due to the explanation that the nanoparticles inside the GPC upgrade the bond strength among matrix and fibres prompting a denser and strong lattice to oppose the breaking process of GPC.

(4) The SEM micrographs portrayed that the enhancement of n-calcium carbonate prompted a denser cover matrix by filling the nanovoids among fly ash particles and preventing the fibre pull-out due to enhancement in fibre-matrix interfacial bonding by the increased quantities of nanoparticles. The SEM micrographs likewise showed that the micro steel fibres and GPC matrix have a decent holding that may be accredited to the capacity of n-calcium carbonate to refine and give a denser microstructure of GPC. Subsequently, the enhancement of n-calcium carbonate upgrades the mechanical presentation of micro steel fibre-reinforced GPC paste that can be utilized in concrete construction for a sustainable environment.

Disclosure statement
No potential conflict of interest was reported by the author(s).

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