



# Influence of Silicon NanoSheet (SiNS) on the toughness of Biphasic Calcium Phosphate (BCP) composites

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Fracture and Structural Integrity - Frattura ed Integrità Strutturale

## Visual Abstract

Influence of Silicon NanoSheet (SiNS) on the toughness of Biphasic Calcium Phosphate (BCP) composites

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**KEYWORDS.** Silicene, Bioceramic composite, Silicon nanosheet, Fracture toughness, Flexural strength, BCP.

## INTRODUCTION

Calcium and phosphate comprise over 70% of human teeth and bone composition. Because of their chemical compatibility and bioactivity bioceramics, which are composed primarily of calcium and phosphate, are referred to as the "golden substance" because they have a wide range of applications in the regeneration and replacement of human bone tissue.



Because hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , or HA) is biologically active, absorbable, and has a high compatibility with natural bone, it is employed as a basic material in the manufacturing of implants. In non-loaded locations, HA has been employed in loose implants [1].

Because it offers superior biological characteristics to metals, it is frequently employed as coatings on metals in bearing applications, either for bone or as a coating put on the metal used in implants. Implant failure may result from coating separation during the implant process because contact pressures during the implant process are greater than the adhesion forces of the metal coating layer [2], [3].

Generally, nanomaterials as fillers can be classified based on their shapes into three groups. These categories are: zero, one, and two dimensions. The mechanical characteristics of materials have generally been improved through using various nanomaterials fillers, such as graphene, CuO, and carbon nanotubes as 0D, 1D, and 2D scales [4], or using organic resources like eggshells and peanut hulls [5-8].

Another bioceramic that has been demonstrated to be both bioactive and biodegradable is tricalcium phosphate (TCP). Compared to HA,  $\beta$ -TCP degrades 3–12 times more quickly [9]. Bone adherence to the ceramic is promoted by the partial breakdown of calcium phosphates [10]. It has created a novel chemical material called biphasic calcium phosphate (BCP) in the last few years [11]. First recorded in a publication by Nery et al., the phrase refers to a combination consisting of hydroxyapatite and tri-calcium phosphate (HA +  $\beta$ -TCP) that was discovered using X-ray diffraction research while preparing 'tri-calcium phosphate' [12]. On the other hand, Akao et al. researched the fracture toughness of HAp, or sintered hydroxyapatite tricalcium phosphate (TCP) and  $\beta$ -TCP. The investigation also found that, when temperature was compared between two temperature ranges (1000°C and 1300°C), the rate of fracture strength rose.

The comparison's findings showed that fracture toughness rates rise with temperature and that 'TCP samples' fracture toughness was superior to that of HAp samples. It also showed that the fracture toughness rate of HAp samples decreased as the research temperature increased [13]. Maria A. Lopes and colleagues investigated the glass-reinforced hydroxyapatite samples' fracture toughness. The research findings indicated that fracture toughness was directly impacted by the percentage of secondary  $\beta$   $\alpha$  phases of tri-calcium phosphate in the compounds' structures, as well as by each of the fine and porosity structural features. Additionally, the composite's performance on the ensuing crack deflection was improved. Regarding the denser  $\beta$ -TCP phase [14].

Others investigated the impact of adding magnesium oxide to hydroxyapatite samples containing 1–10% of the mineral on their fracture toughness. By increasing the fracture toughness to 1% and improving the explanation, the study found that magnesium oxide can assist prevent granule formation and produce a microstructure with greater durability. Because magnesium oxide breaks down the apatite structure, increasing magnesium oxide by more than 1% resulted in both an increase in microporosity and a drop in fracture toughness. Fracture toughness was directly impacted by each of the fine and porosity structural characteristics as well as the proportion of secondary  $\beta$ -phase  $\alpha$ -tricalcium phosphate in the compound structures.

In addition, the more solid  $\beta$ -TCP phase has improved the compounds' performance in terms of fracture deflection. When blocks of macroporous BCP (60% HA, 40%  $\beta$ -TCP) were incubated in simulated bodily fluid, needle-shaped apatite crystals precipitated onto their surfaces [15]. Additionally, the area around the HA crystals, where epitaxial growth was placed, has a higher concentration of apatite crystals. Apatite may nucleate and grow orientatedly on HA crystals with less energy than it can on  $\beta$ -TCP crystals, which have a different lattice structure. This is because the lattice structures of precipitated apatite and HA are identical. Accordingly, adding BCP as a filler to the composite system might increase bioactivity without compromising degradability. To create composites that are strong, biocompatible, and bioactive, the idea of bioceramic particles in conjunction with self-reinforcement has been used [16]. In order to create a matrix that is further reinforced with PLA fibres, this work creates triphasic composites of biodegradable PLA reinforced with BCP bioceramic particle filler. Compression moulding preimpregnated sheets is one way to create the triphasic composite [16]. PLA fibres are drawn through a PLA matrix and BCP filler solution during the prepregging process, which creates a layer on top of the fibres.

Considering all that has been discussed thus far, further research is necessary to enhance the mechanical properties of bioceramics materials through the use of innovative nanostructured fillers. Prior research has concentrated on enhancing bioceramics with 0D and 1D fillers, such as carbon nanotubes and TiO<sub>2</sub>. Afterwards, 2D fillers are used in the studies in place of graphene to improve the material's characteristics. Finding novel nanostructured fillers becomes essential as a result. Thus, the current study uses silicon nanofillers (SiNS) to measure the strength and fracture toughness of HA/ $\beta$ -TCP (BCP) composite at different ratios. Applications of this compound in bioengineering, include bone grafting and synthesising and replenishing human tissue.



## MATERIALS AND METHODS

### *BCP Composite Synthesis*

Tab. 1 displays the five different ratios of BCP composite that were created depending on the weight percentage of  $\beta$ -TCP and HA ceramics. To guarantee uniform mixing and prevent agglomeration, the powders were combined using a mixer-type NQM-0.4 model planetary ball mill running at 850 rpm for one hour. To make the combination more homogeneous, ethanol was added to the powder in the form of a liquid suspension. The mixture was then removed and allowed to dry for 24 hours at 80°C in the oven. This mixing procedure is consistent with other research, such as ref. [17].

Subsequently, the blend was forced into a steel mould including a rectangular section cavity of 3 x 4 x 40 mm. Paraffin was used to lubricate the inner wall of the mould in order to lower friction between the material and the wall and make it easier to remove the sample from the mould following the pressing process. After being sintered for two hours at a rate of 5°C/min to reach a temperature of 1200 °C, the generated samples were cooled at the same rate to room temperature. Tab. 1 shows the various weight ratios of BCP composites.

Sample	Composition of HA (wt.%)	Composition of $\beta$ -TCP (wt.%)
1	100	0
2	75	25
3	50	50
4	25	75
5	0	100

Table 1: Various ratios of BCP composite.

### *Silicon Nanosheet (SiNS) Synthesis*

The hexagonal honeycomb structure of silicene, a two-dimensional allotrope of silicon, is comparable to that of graphene nanostructures. It has a unique low-buckled structure and is composed of a single, thick atomic layer. Its better qualities, which it shares with graphene, make it conceptually interesting [18]. It can be produced using chemical reactions between several materials as follows [18]:

The use of sodium chloride, magnesium powder, and montmorillonite clay (MMT) (Sigma-Aldrich, St Louis, MO, USA) in the following ratios (1:3:0.7), respectively, is essential to this process. To prevent oxidation at high temperatures, the mixture was put in a stainless-steel reactor, which was then placed in a tube furnace in an inert argon atmosphere. Subsequently, the furnace was run at 650 °C for five hours. To eliminate the NaCl, the liquid was cooled and then stirred for three hours using a magnetic stirrer. After adding 1 millilitre of hydrochloric acid to eliminate the magnesium, silicene was finally extracted using 0.2% HF and dried for 12 hours at 80°C.

Owing to the effects of light and moisture on silicene, the product was kept in an opaque, dry, and inert container [19].

### *Setting Up BCP/SiNS*

Following the completion of the BCP composite and Silicene preparation, each BCP composite previously listed in Tab. 1 was mixed with Silicene to create the BCP/SiNS composite mixture. The silicene addition was made in a weight percentage of (1, 3, 5). First, a ball milling mixer operating at 850 rpm for one hour was used to combine the dry powders. Subsequently, 10% of methylcellulose (MC) was added as a binder to each combination. Lastly, the mixture was sintered and pressed using the method that was described previously.

## EXPERIMENTAL TESTS

### *Fracture toughness test*

The field of study known as fracture mechanics explains how materials behave when they have flaws, microcracks, or weak spots in the material such as holes and gaps. They have the potential to cause a fracture that breaks the system. The magnitude of this load can be determined by the fracture's strength. To conduct this test, find a defect that is known to exist and its dimensions, then apply pressure on the affected area. It is used to obtain the stress intensity factor

( $K_{Ic}$ ) a value that promotes the propagation of cracks and ultimately results in system failure. According to the formula (1) [20]:

$$K_{Ic} = f\sigma\sqrt{\pi a} \quad (1)$$

The crack's geometric factor ( $f$ ), sample pressure ( $\sigma$ ), and crack size ( $a$ ) are represented by these values. A gadget (Universal Computer Control Electronic Testing Machine-Laryee Technology-UE34300) was used to perform the fracture test. As indicated in Fig. 1, samples were prepared in accordance with the specification (ASTM C1421-10), with sample dimensions of 3 x 4 x 40 mm and a crosshead speed of 0.5 mm/min. To determine the specimen's fracture toughness under load, a central incision is made. The sample is supported by two structures at both ends, and the centre part of the sample bears the weight across the area of the incision. Test results are obtained from a computer that is linked to a device that displays the test's stress-strain curve. This experiment was conducted three times at ambient temperature and 15% relative humidity.

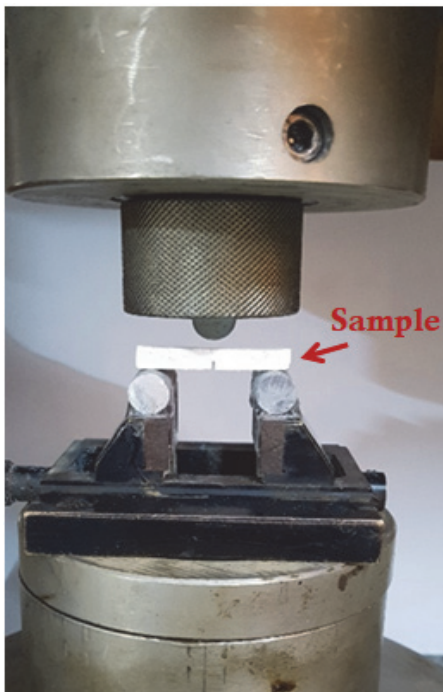


Figure 1: The fracture toughness test device.

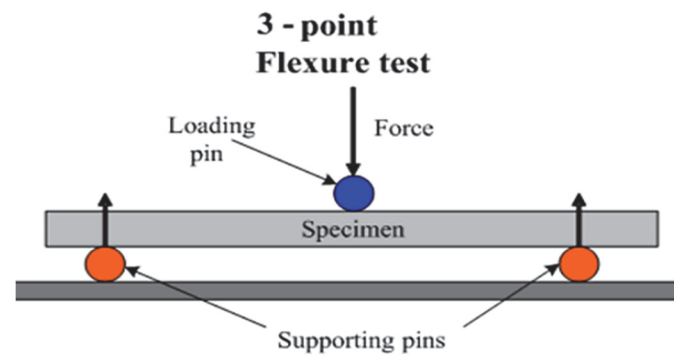


Figure 2: Three-point bending test.

### *Flexural strength test*

A material testing approach known as the three-point bending technique measures the flexural stress that results from setting up a material sample as a simply supported beam with two supports and applying a load at the position indicated in Fig. 2. This test is appropriate for bones to make sure the response is primarily flexural. Unlike the results from the compression tests, these results are more amenable to modelling studies since the mechanical response of the structure is essentially represented by a single mode of deformation. Thus, the results of three-point bending tests have found widespread use in basic research on bone mechanics.

Bending tests were performed in accordance with ASTM C1161-13 standard. For flexural tests, beam specimens measuring length ( $l$ ) = 40 mm, width ( $b$ ) = 4 mm, and thickness ( $t$ ) = 3 mm were ready; all measurements were taken with an accuracy of  $\pm 0.03$  mm. The following formula can be used to determine the flexural strength:

$$\sigma_f = \frac{3Pl}{2bt^2} \quad (2)$$

where  $\sigma_f$  and P mean the flexural strength, and brake force respectively. Three-point bending tests were performed utilising a Universal Computer Control Electronic Testing Machine (Laryee Technology-UE34300) with displacement control (cross-head feed rate equal to 0.5 mm/min). Three runs of this experiment were carried out with 15% relative humidity and room temperature.

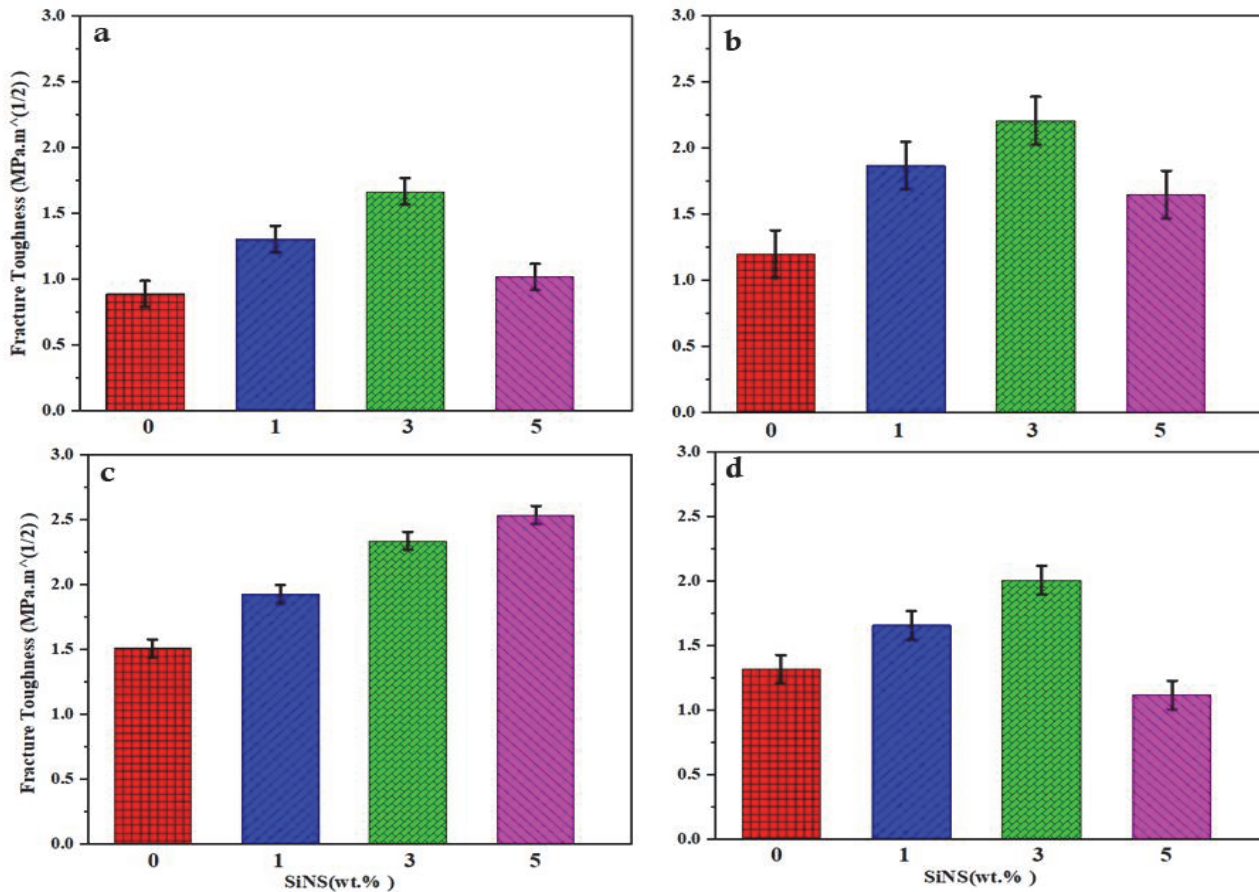
## EXPERIMENTAL RESULTS

Fracture toughness, or a material's ability to withstand the formation of cracks, is another crucial property of materials. As a result, it provides a clue about the critical assessment of the material's behaviour during work. Fig. 3 displays the findings from the examination of the fracture toughness test.

Tab. 2 displays the fracture strength values in general. The fracture strength values of the compounds when compared to the base material demonstrate a significant variation. Regardless of the weight ratio between HA and TCP, the graph (Fig. 3) indicates that when the reinforcing particles (SiNS) are introduced, the BCP composite's fracture toughness gradually increases.

The percentage of rising toughness values for the composites (100% HA + 0% TCP), (100% TCP + 0% HA), (25% HA + 75% TCP), (50% HA + 50% TCP), and (75% HA+25% TCP) are 47.19, 55.83, 27.81, 25.75, and 40.17%, respectively, when the content of SiNS was (1%).

By raising the SiNS concentration from 1% to 3%, the percentage of toughness increase persisted. where the percentage that was increasing varied from 33 to 87.64 percent. With an increase of 87.64%, the composite (100% HA + 0% TCP) had the largest value of increase, while the composite (75% HA + 25% TCP) saw the lowest. Ultimately, as some percentages climbed and others fell in toughness, the results of the fracture toughness of the samples containing 5% SiNS started to diverge, as shown in Fig. 4.



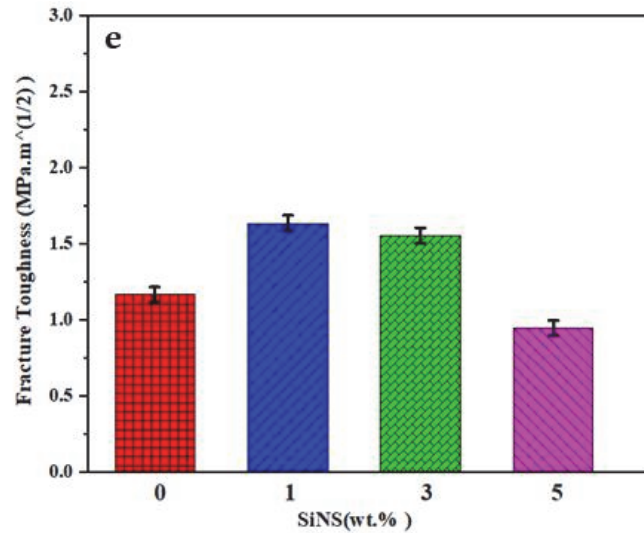


Figure 3: The relationship between the average fracture toughness and the weight percentage of the filler SiNS for (a) HC, (b) TCP, (c) 25%HA+75%TCP, (d) 50%HA+50%TCP, and (e) 75%HA+25%TCP samples.

SiNS(wt.%)	Fracture Toughness ( $MPa.m^{1/2}$ )				
	HA $\pm 0.1$	TCP $\pm 0.18$	25%HA+75%TCP $\pm 0.07$	50%HA+50%TCP $\pm 0.11$	75%HA+25%TCP $\pm 0.05$
0	0.89	1.2	1.51	1.32	1.17
1	1.31	1.87	1.93	1.66	1.64
3	1.67	2.21	2.34	2.01	1.56
5	1.02	1.65	2.54	1.12	0.95

Table 2: Fracture Toughness test results.

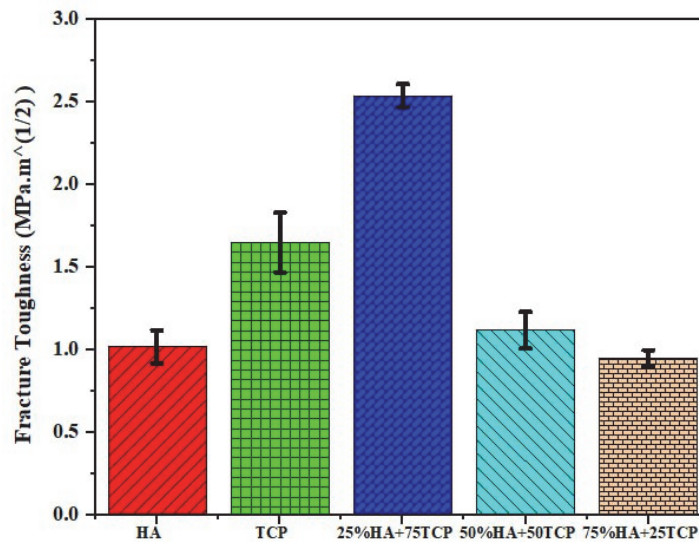


Figure 4: The fracture toughness at 5% SiNS.

On the other side, Fig. 5, and Tab. 3 show the relationship between the flexural strength and the weight content of (SiNS) at various BCP composites which are shown in Tab. 1.

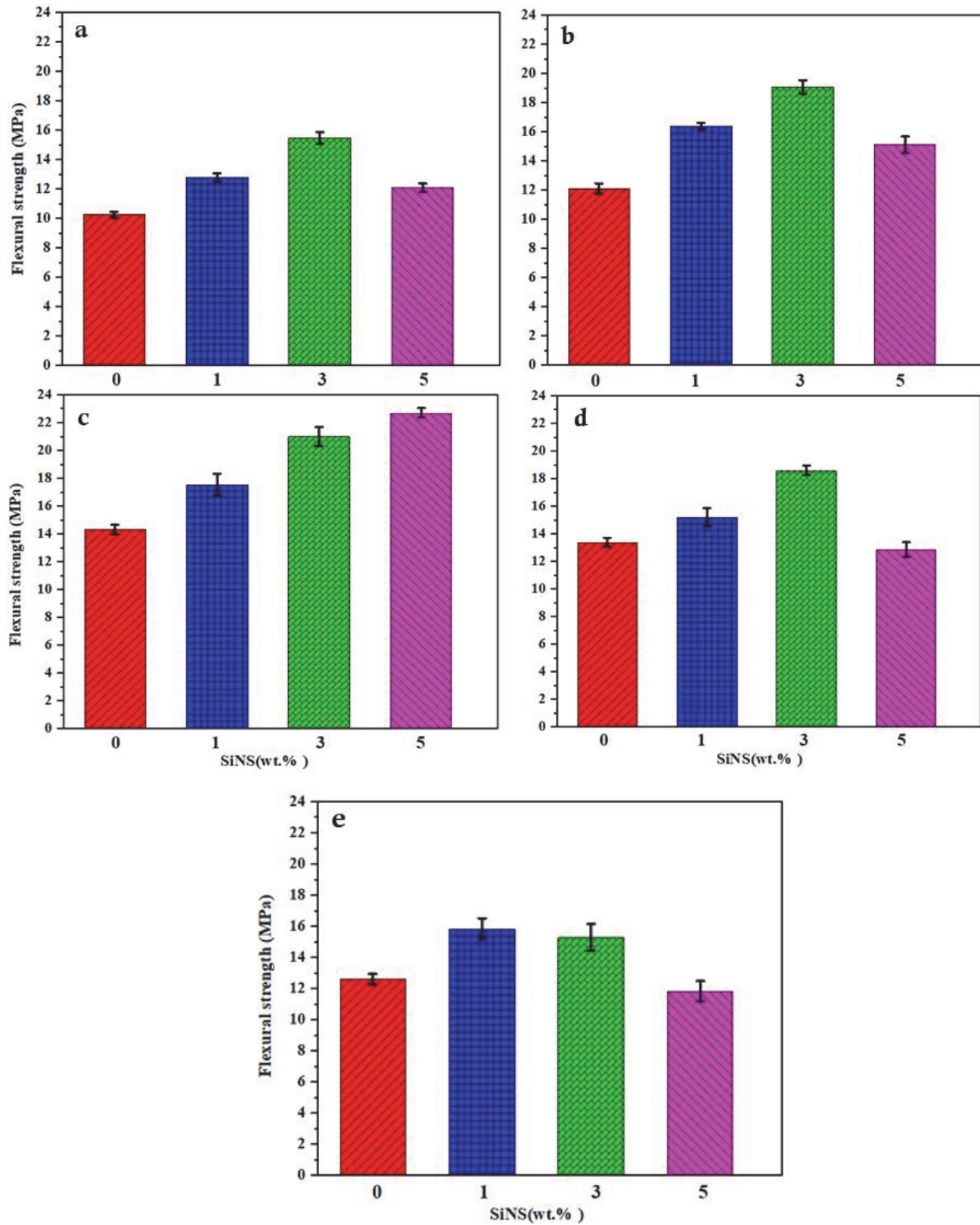


Figure 5: The relationship between the flexural strength and the weight percentage of the filler SiNS for (a) HC, (b) TCP, (c) 25%HA+75%TCP, (d) 50%HA+50%TCP, and (e) 75%HA+25%TCP samples.

SiNS (wt.%)	Flexural Strength (MPa)				
	HA ±1.1	TCP ±0.8	25%HA+75%TCP ±1.7	50%HA+50%TCP ±0.6	75%HA+25%TCP ±0.35
0	10.28	12.12	14.33	13.4	12.61
1	12.8	16.4	17.58	15.24	15.87
3	15.5	19.1	21.03	18.64	15.31
5	12.1	15.13	22.73	12.88	11.85

Table 3: Flexural Strength test results.

The percentage increase in the flexural strength was maintained when the SiNS concentration was increased from 1% to 3%, where the growing percentage ranged from 14 to 60 percent. The composite consisting of 100% HA and 0% TCP experienced the highest rise, measuring 60 %, whereas the composite consisting of 75% HA and 25% TCP saw the lowest increase (15%). In the end, there was a divergence in the flexural strength results of the samples containing 5% SiNS as some percentages increased and others decreased, as shown in Fig. 6.

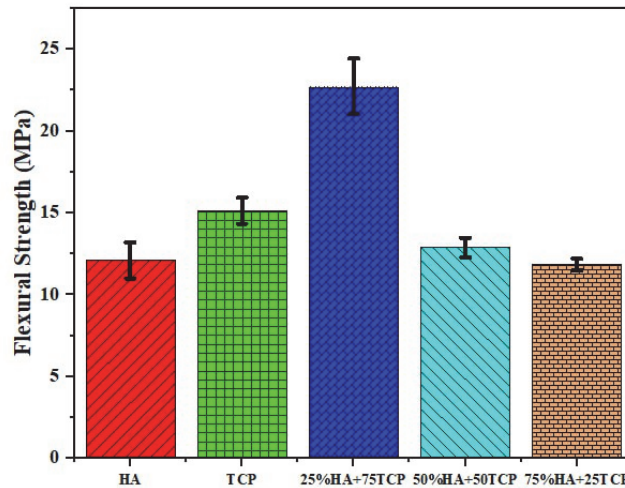


Figure 6: The flexural strength at 5% SiNS.

## DISCUSSION

The most obvious finding to emerge from the analysis, as shown in Figs. (3 and 5), is that fracture toughness and bending strength have very similar trends, with differences in the percentages of increase or decrease. Several cases were identified through the analytical drawing and the prior data on the values of the bending resistance. These cases included a rise in strength values when fillers were added to both basic and BCP composites because SiNS played a part in these processes.

The BCP composite consisting of 25% HA and 75% TCP exhibited the largest average increase in fracture strength value (185%) when compared to the pure HA material.

The basic trade-off connection between hardness and fracture toughness, which is recognised in mechanical behaviour and applies to ceramic materials as well, is well known [21]. Nevertheless, it was shown that the hardness increase rose in tandem with the hardness to the level of 3% filler, without having any effect on fracture resistance. This shows how fillers can effectively alter the behaviour of bioceramics to become one that is distinct and unique while also getting two key features. This is because the filler (SiNS) has the capacity to prevent cracks from growing while also preserving crystalline tissue, which makes it a crucial defence against fracture propagation. In terms of fracture resistance, this behaviour of SiNS is comparable to that of graphene, which is comparable to the crystalline form of silicene [21], [22].

The levels observed in this investigation are close to those observed by [23] when they used 1.5 wt% graphene nanoplates to improve the bending strength and fracture toughness of BCP composite to reach about 55% and 75%, respectively. In the current study, the bending strength and fracture toughness of the BCP composite increased about 68.2% and 58.62%, when SiNS was added by 3%.

Due to silicene's ability to limit grain growth in the substrate, this increases the pace at which grain boundaries form and produces a finer grain size structure. Grain fineness increases the number of grain borders and, consequently, the amount of variations in the fracture development route across the grain boundaries [20].

This, in order to facilitate growth, requires the fracture to use more energy; this idea is one that is incorporated into the strengthening methods used to strengthen ceramics [24], [25]. Additionally, as Fig. 7 illustrates, an increase in pore rate is the cause of the decline in the fracture toughness value when the filler (SiNS) rose by more than 3%. This concentration of silicene is present at the substrate's grain boundaries and causes additional aberrations in the intergranular fractures' route.

As a result, these pores function as flaws, causing weaknesses and fractures that lower fracture toughness. Further to the variations in the thermal expansion coefficients of the SiNS material and the base material. As a result, internal stresses increased and were concentrated in the weakest areas of the material that may have a cracking area defect. This helped to form tensile and compressive stresses as a result of the difference in contraction and expansion, which differed from this case in terms of the percentage of the compound with the ratio (25%HA + 75%TCP).

This did not much increase porosity because it increased by 3% over the prior ratio—a relatively small proportion that contributes to durability—despite the silicene content increasing by more than 5%.

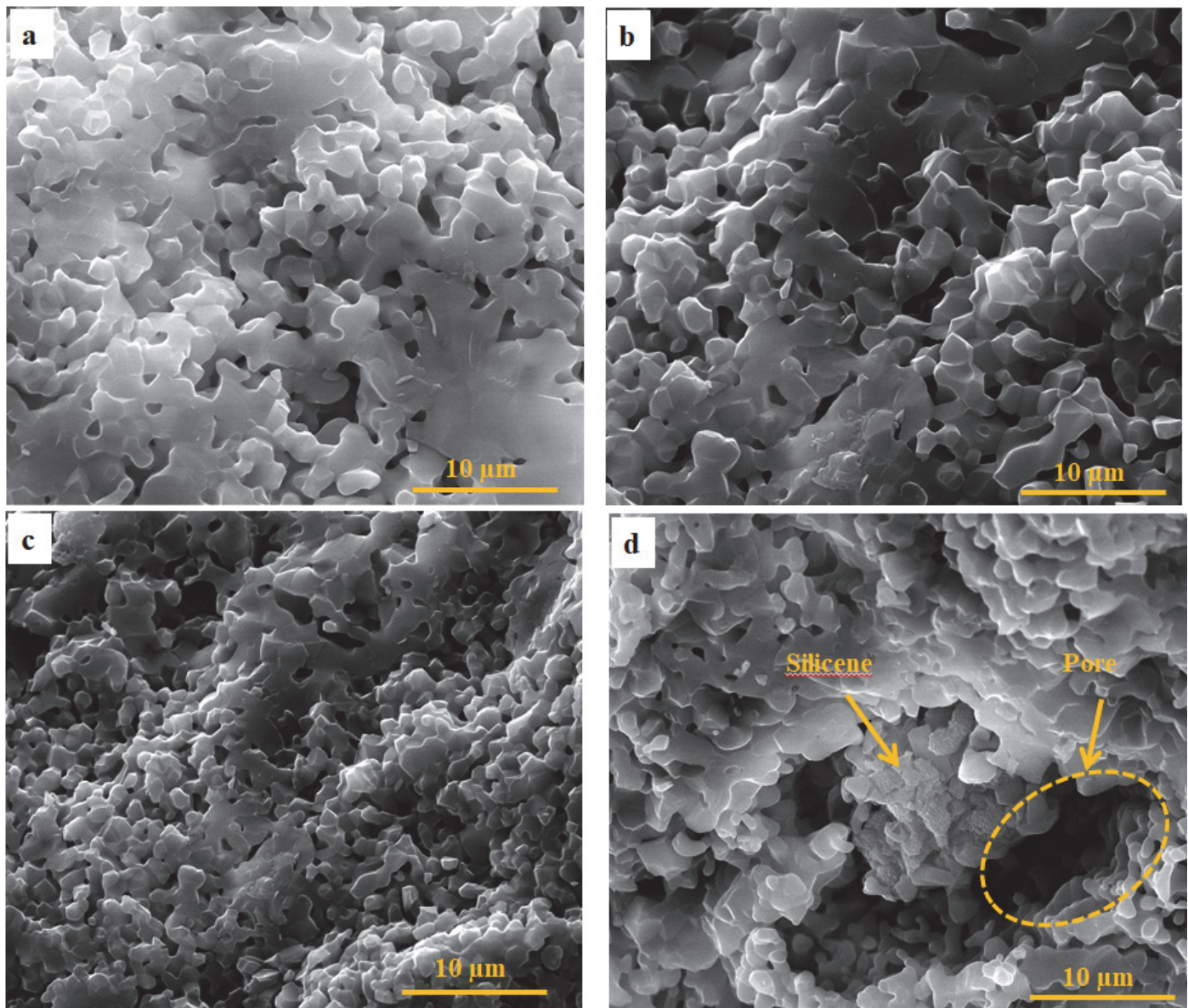


Figure 7: Increase of porous with increased filler for (a) (50%HA+50TCP), (b) (50%HA+50TCP) +10% Silicene, (c) (50%HA+50TCP) +30% Silicene, and (d) (50%HA+50TCP) 50% Silicene samples.



## CONCLUSION

The present study was designed to determine the effect of adding the silicon nanosheet (SiNS) on the toughness behaviour of BCP composites. Through the current research, SiNS was synthesized using the chemical reactions and mixed at various weight ratios (1, 3, and 5%) with BCP composites. While, BCP composites were produced at various ratios of mixing HA and TCB bioceramics.

The most obvious finding to emerge from this study is that the addition of SiNS enhances the strength and fracture toughness of HA, TCP, and BCP composites. The results of this research indicate that the fracture toughness of BCP at various ratios of HA and TCP increased between 33 and 87.64% when SiNS was added from 1 to 3% weight. While, the flexural strength of BCP composites is enhanced by 15 to 60% through adding SiNS from 1 to 3%. This is because of the role that SiNS plays as fillers to prevent cracks from growing while also preserving crystalline tissue, which makes it a crucial defence against fracture propagation due to the crystalline form of SiNS which behaves similarly to graphene.

However, as some percentages climbed and others fell in toughness or flexural strength, the results of the samples containing 5% of SiNS started to differ somewhat. When the weight percentage of SiNS increased excessively, they cause additional aberrations in the intergranular fractures' route due to they are concentrated at the substrate's grain boundaries. As a result, these pores function as flaws, causing weaknesses and fractures that lower fracture toughness. The results of this study indicate that the strength and fracture toughness of bioceramic materials and their composites are significantly influenced by (SiNS).

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## NOMENCLATURE

SiNS	Silicon nanosheet
HA	Hydroxyapatite
TCP	Tricalcium phosphate
BCP	Biphasic calcium phosphates
MMT	Montmorillonite clay
$K_{Ic}$	Stress intensity factor
MC	Methylcellulose
$\sigma$	Sample pressure
a	Crack size
f	Crack's geometric factor
$\sigma_f$	Flexural strength
P	Brake force