
ENGINEERING SUSTAINABLE PCL/PLA/HAP-SR/KERATIN FIBRE BIOPOLYMER COMPOSITES FOR BONE TISSUE ENGINEERING APPLICATIONS: MECHANICAL AND CHEMICAL PROPERTIES

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ABSTRACT

Polycaprolactone (PCL) is widely recognised as a promising biomaterial owing to its ease of processing, biocompatibility, and biodegradability. Despite these advantages, its slow degradation rate and insufficient mechanical strength and stiffness hinder its application where enhanced structural performance is required. This study investigates the influence of PLA, strontium-doped hydroxyapatite (HAp/Sr), and keratin fibre reinforcement on the chemical structure and mechanical performance of polycaprolactone (PCL)-based biocomposites. FTIR spectroscopy was employed to examine molecular interactions between the polymer matrix and reinforcing phases. The tensile strength, Young's modulus, hardness and fracture toughness tests were conducted to evaluate mechanical integrity. The spectra show the presence of PCL through characteristic absorption bands at 2947 and 2865 cm⁻¹, attributed to the C-H asymmetric stretching vibration. A slight shift is observed in some peaks toward either a lower or a higher wavenumber, with no new peaks observed in the reinforced biocomposites. This indicates possible weak intermolecular interaction rather than new covalent bond formation. Pure PCL (BC1) exhibited the lowest mechanical performance, with an ultimate tensile strength of 4.55, Young's modulus of 2 MPa, hardness of 22.3, and fracture toughness of 0.5659. Incorporation of reinforcing agents (i.e., PLA, strontium-doped hydroxyapatite (HAp/Sr), and keratin fibre) resulted in a significant increase in hardness and fracture toughness, demonstrating the reinforcing effect of bioactive fillers. The sample BC4 showed the highest overall mechanical performance, with ultimate tensile strength (38.5), Young's modulus (4.05), hardness (56.33), and fracture toughness (1.0534), indicating enhanced stiffness and resistance to crack propagation. These findings suggest that the synergistic combination of PLA, keratin fibre, and bioactive ceramic additives significantly enhances the mechanical behaviour of PCL-based materials, making them more suitable for applications requiring improved strength, stiffness, and durability, such as biomedical scaffolds and load-bearing structures.

Keywords: Polycaprolactone; Polylactic Acid; Keratin Fibre; Hydroxyapatite; Mechanical properties

1.0 INTRODUCTION

Bone tissue serves a number of vital functions in daily life, including safeguarding internal organs, giving the body structural support, facilitating mobility, and controlling the storage and release of minerals (Gharibshahian et al., 2023). Many people suffer from bone diseases brought on by trauma, tumour excision, infection, congenital anomalies, and injuries sustained in accidents (Li et al., 2020). However, defective bone tissue possesses the ability to self-repair or self-regenerate, which occurs in four stages: hematoma, soft callus, hard callus formation and bone remodelling (Monfared et al., 2022). Self-repair and self-regeneration weaken with age or in critical bone defects (i.e., cracks above 2.5 cm). Tissue engineering deploys an innovative approach to repair bone defects (Jung et al., 2022; Yudoh et al., 2023). The approach adopts engineered bone grafting techniques, which aid the healing of the defective bone, while also providing the needed strength that supports the healing process (Menotti et al., 2023). Archunan & Petronis (2021) highlighted that approximately 2.2 million bone grafting techniques are used in orthopaedic surgical processes worldwide, and the number is expected to increase yearly by 13%. However, the current bone grafting techniques (i.e., allograft and autograft) face

different challenges such as risk of disease transmission, limited availability and immune rejection (Gharibshahian et al., 2023).

To overcome the shortfalls from the current bone grafting techniques, bone tissue engineering offers a solution, involving the use of a three-dimensional scaffold made from biomaterials that creates an environment for cell proliferation and regeneration (Biscaia et al., 2022). Biomaterial scaffolds for repairing defective bone are often developed from standalone materials (i.e., ceramics, biodegradable polymers, and metallic materials) or composites (Hernandez et al., 2017). A suitable biomaterial needs to possess good mechanical properties so that it can provide structural support during the healing process (Obada et al., 2021; You et al., 2022). The materials and production methods (i.e., melt extrusion/hot pressing, solvent casting, electrospinning and many others) used in producing the scaffolds influence the mechanical properties.

Biomaterial scaffolds could be produced from biodegradable polymers (either synthesised or sourced naturally) since they have excellent mechanical properties and porous architecture that creates a bioactive environment for cell proliferation and adhesion (Saurav et al., 2024). PCL is known for its great flexibility (Loyo et al., 2024), while PLA is a cost-effective polymer, with good biocompatibility and a low melting point (Zarei et al., 2023). Several attempts have been made to develop a polymer composite by blending PLA and PCL (Aliotta et al., 2023; Min et al., 2024). Vonbrunn et al. (2020) reported that incorporating PCL into the PLA matrix results in a broad surface area for tissue regeneration and host cell structural support. However, PCL/PLA scaffolds have poor hydrophilicity, low cell migration and adhesion. The poor hydrophilicity could result in prolonged degradation (Mohandesnezhad et al., 2020). Also, they lack the optimal combination of mechanical strength, bioactivity and degradation properties necessary for successful bone regeneration.

In this study, scaffolds for tissue engineering rely on the interplay of biodegradable polymers (PLA and PCL), bioactive filler (HAp/Sr), and reinforcement material (keratin fibre). HAp is known for its biocompatibility, osteointegration, osteoconductivity and non-toxicity (Bee & Hamid, 2020). The addition of hydroxyapatite (HAp), enhanced with strontium (Sr), introduces osteoconductive properties that promote bone formation and integration (Obada et al., 2021). Combining these components with biodegradable polymers like polycaprolactone (PCL) and polylactic acid (PLA) creates a scaffold that closely mimics the structure and function of natural bone. Keratin fibres, derived from chicken feathers, are a durable, abundant, and underutilised natural protein with exceptional bioactive and biocompatibility properties, making them a potential reinforcement material. Cheng et al. (2009) reported that keratin fibre obtained from chicken feathers has a special cross-linked and semi-crystalline structure which helps improve the mechanical stress resistance and elastic modulus of polymer-based composites. However, the study utilised PLA rather than PCL; the findings provide the provide for reinforcing capability of keratin fibre derived from chicken feathers. The utilisation of locally sourced materials (i.e., sheep bone and chicken feathers) offers potential cost and sustainability benefits by converting waste into valuable biomaterials. These materials could lower reliance on imported materials while supporting local production; consequently, this supports Nigeria's objectives for industrial growth, creation of sustainable materials and waste management.

Therefore, this study examined the effect of incorporating PLA, HAp/Sr and keratin fibre on the mechanical properties of PCL biopolymer composites. The hardness, fracture toughness, tensile strength and Young's modulus of the developed biopolymer composite were examined. The functional groups in the samples were identified using the Fourier Transform Infrared Spectroscopy technique.

2.0 MATERIALS AND METHODS

2.1 Materials

The polycaprolactone and the polylactic acid powder were purchased from Sino Convoy New Material Co., Ltd. (China) and Guangzhou Bio Plus Materials Technology Co., Ltd, respectively. The hydroxyapatite was obtained from sheep bone, which was sourced from a local abattoir. The collected sheep bones were washed, dried, and carbonised in a charcoal-fired oven. The carbonised bones were calcined in a Muffle furnace at 900 °C for 2 hours. A mortar and pestle were used to grind the calcined bone into a fine powder. A 100 µm mesh was used to sieve the resulting fine powder to get a uniform particle size (Obada et al., 2024). Strontium was incorporated into the HAp powder using the solution

gelation (sol-gel) technique in a proportion suggested by Obada et al. (2021). The keratin fibres were extracted from chicken feathers using the hydrolysis technique.

2.2 Production of the Pure PCL and PCL Biocomposite Polymers

The polymer samples were produced using a solvent-casting technique guided by the process presented by Adamu et al. (2023) and Rinaldi et al. (2024). The PCL and PLA powders were dissolved in chloroform, then stirred till the powder dissolved completely in the solution. Keratin fibres and HAp/Sr powder were added to the solution, followed by stirring to ensure even distribution. The mixture was then poured into a mould and allowed to dry under controlled conditions. The dried samples were subjected to characterisation and testing. The samples were developed using the composition in Table 1, which was selected after optimising the process using Box-Behnken DOE. The functional groups in the developed samples were identified by FT-IR.

Table 1: Composition of the samples

Sample Code	PCL (wt.%)	PLA (wt.%)	HAp/Sr (wt.%)	Keratin fibre (wt.%)
BC1	100	--	--	--
BC2	78	22	--	--
BC3	68	22	10	--
BC4	61	22	10	7

2.3 Hardness and Fracture Toughness

The hardness of the developed sample was measured using a Vickers hardness tester. An indent load of 300 grams was used, and the indent diagonal was measured, which was then used to calculate the hardness. The fracture toughness was calculated using Equation 1 as adopted by Obada et al. (2023), the parameters included the hardness value, diagonal length and the crack length.

$$K_{ic} = 0.016 \times \left(\frac{c}{a}\right)^{-1.5} \times H_v \times a^{0.5} \quad 1$$

Where Hv: Vickers hardness, c is the sum of half of the diagonal length (l), and a is the crack length: $0.5(a + l)$

2.4 Tensile Strength Measurement

The tensile strength measurement was conducted using a universal testing machine. The standard dimensions used for the test are a gauge length of 50 mm and a diameter of 12.5 mm. The maximum load (tensile strength) and elongation at break were recorded as the test proceeded until the specimen breaks. The tensile strength and Young's modulus of the samples were obtained from the data generated by the machine. For each composition, three independent specimens ($n = 3$) were tested under the same experimental conditions.

3.0 RESULT AND DISCUSSION

3.1 FTIR Analysis of the Pure PCL and PCL Biocomposite Scaffolds

The FTIR spectra of the PCL and PCL biocomposite are shown in Figure 1. The spectra show the presence of PCL through characteristic absorption bands at 2947 and 2865 cm^{-1} , attributed to the C-H asymmetric stretching vibration. In the BC2 spectrum, no new peaks were observed, indicating that no chemical decomposition or interaction occurred between PCL and PLA during the development process. Maleki et al. (2021) developed a PCL/PLA composite, and the FTIR spectra of PLA, PCL, and the PCL/PLA blend were reported to be similar, indicating that no chemical reaction occurred. The authors then concluded that this illustrates a proper mixture. However, a slight shift is seen in some peaks to either a lower or a higher wavenumber. For instance, the C=O symmetric vibration was seen at 1716 cm^{-1} , but shifted to 1728 cm^{-1} in the BC2 spectrum. The peak shift is attributed to the weak interaction between the PCL and PLA (Lu & Kazarian, 2020). A similar shift is observed in the BC3 and BC4 sample spectra, with no new peak observed, suggesting that the principal functional groups of the individual constituents were retained following the development of the composite. This aligns with the study conducted by Shojaei et al. (2019).

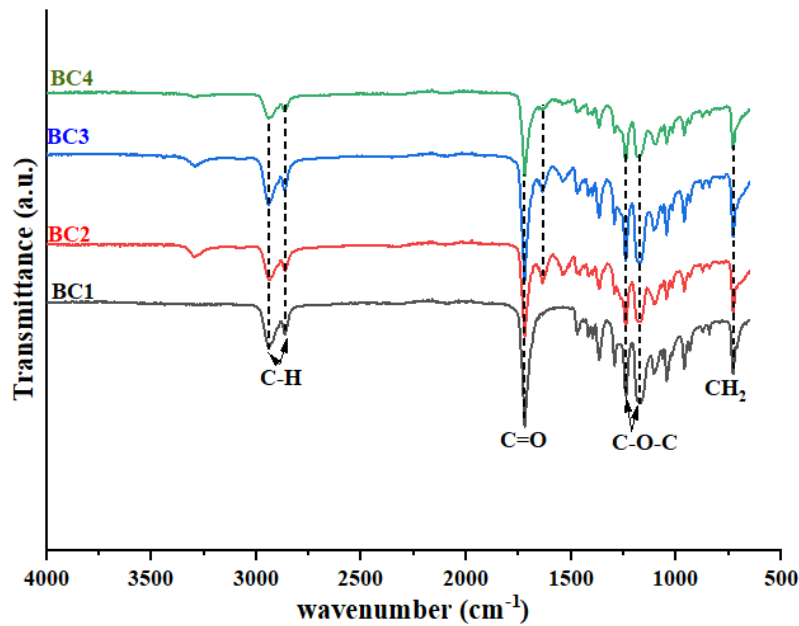


Figure 1: FTIR spectra of the pure PCL and PCL biocomposite

3.2 Mechanical Properties of the Pure PCL and PCL Biocomposite Scaffolds

3.2.1 Hardness Value and Fracture Toughness

The hardness value and fracture toughness of the unreinforced PCL and reinforced PCL are illustrated in Figure 2. The differences observed in samples' hardness and fracture toughness show the effect of both ceramic reinforcement and polymer blending on resistance to crack propagation and deformation. The BC1 had the lowest hardness (22.3 Hv) and fracture toughness ($0.566 \text{ MPam}^{1/2}$), confirming its relatively soft structure (Chen et al., 2018). A slight improvement was observed when PCL was blended with PLA, as seen in BC2, with hardness and fracture toughness increasing to 24.27 Hv and $0.646 \text{ MPam}^{1/2}$, respectively. This indicates that PLA increases the PCL rigidity and its energy absorption capacity during fracture. Homkhiew et al. (2024) noted that increasing the PLA concentration made the composite less flexible, resulting in increased hardness. A more significant improvement was recorded in BC3, where the hardness increased to 33.33 Hv and fracture toughness to $0.664 \text{ MPam}^{1/2}$. This enhancement is attributed to the presence of reinforcing agents (i.e., PLA polymer and hydroxyapatite-strontium particles).

These reinforcing agents could have promoted better stress distribution and energy-absorbing capacity that delays fracture propagation. The sample BC4 had the highest value, with hardness and fracture toughness reaching 56.33 Hv and $1.053 \text{ MPam}^{1/2}$, respectively. When compared with BC1, BC4 gave a substantial increase, increasing approximately by 60.4% for hardness and 46.3% for fracture toughness. This suggests that there is a strong synergistic interaction between the reinforcing agents and the polymer matrix. The improved homogeneous dispersion of the fillers and enhanced interfacial bonding between the matrix and the filler contributed to improved crack formation and resistance against indentation. The hardness value obtained for BC4 (56.33 Hv) falls within the hardness range of trabecular bone (41.8 – 90.8 Hv) (Wang et al., 2006), making the developed composite suitable for cancellous-bone application, since cortical bone requires higher hardness and strength.

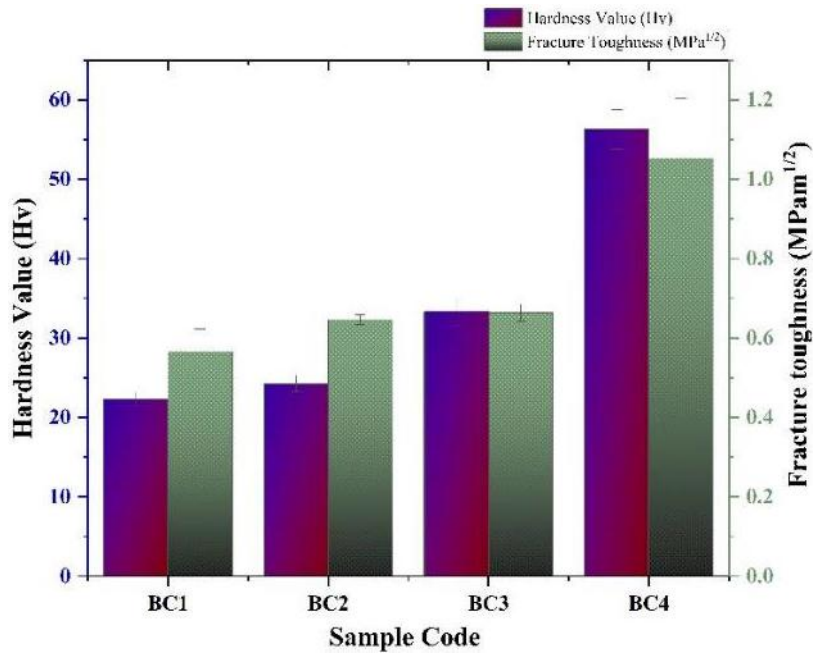


Figure 2: Hardness value and fracture toughness of the samples

3.2.2 Tensile Strength and Young Modulus

Figure 3 presents the ultimate tensile strength and Young's modulus of the pure PCL and the BC4 from section 3.2.1. A low ultimate tensile strength of 4.55 MPa and a Young's modulus of 2 MPa indicate the PCL's ductile, soft and low-stiffness nature. Hashim et al. (2022) noted that the PCL polymer had low modulus and tensile strength. These low values indicate that the BC1 had low resistance to applied stress and possessed a significant tendency for elastic deformation under load, which limits its applicability in structurally challenging situations. A significant increase was observed for BC4, resulting in an ultimate tensile strength of 38.5 MPa and a Young's modulus of 4.05 MPa. This shows that the tensile strength and Young's modulus increase by 88.1% and 50.6%, respectively. This shows a remarkable improvement in the load-bearing capacity and stiffness compared to BC1. The measured values in the study are relatively low, attributed to the production technique used (solvent casting), which can introduce porosity and residual solvent within the composite, thereby lowering the overall mechanical performance.

The improvement suggests that the addition of the reinforcing agents could have fewer structural defects and promote effective load distribution across the matrix. In general, sample BC4 exhibits superior resistance to failure, making it more suitable where structural integrity and mechanical reliability are required.

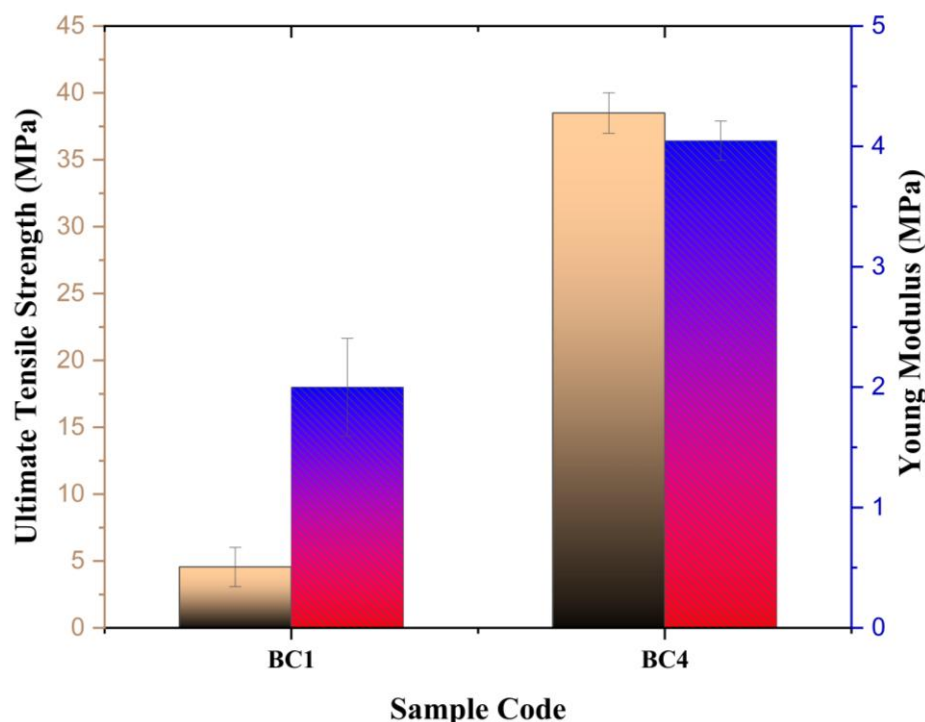


Figure 3: Ultimate tensile strength and Young's modulus of the samples

3.3 Morphological Analysis of the Pure PCL and PCL Biocomposite Scaffolds

The SEM images of the pure PCL and PCL biocomposite scaffolds are illustrated in Figure 4. No surface fractures and a uniform surface morphology were observed in the BC1 scaffold. Additionally, small pores formed as a result of chloroform evaporating during drying were observed. The results are consistent with the research of Adamu et al. (2024), which claimed that the evaporation of chloroform during drying leaves tiny pores in the unreinforced PCL scaffold. The surface of sample BC4 was smoother and more compact, suggesting that the reinforcing agents were evenly distributed throughout the PCL matrix. This could have strengthened the polymer chain, improving the mechanical characteristics, as observed in Figures 3 and 4. According to Matsumoto (2024), the polymer chain is influenced by the surface characteristics and behaviour of the polymer. The physical and mechanical qualities might be impacted by a smooth, compacted surface that limits the mobility of the polymer chains. Additionally, there was a white, bright granule on the surface, indicating that HAp/Sr particles were present in the polymer matrix.

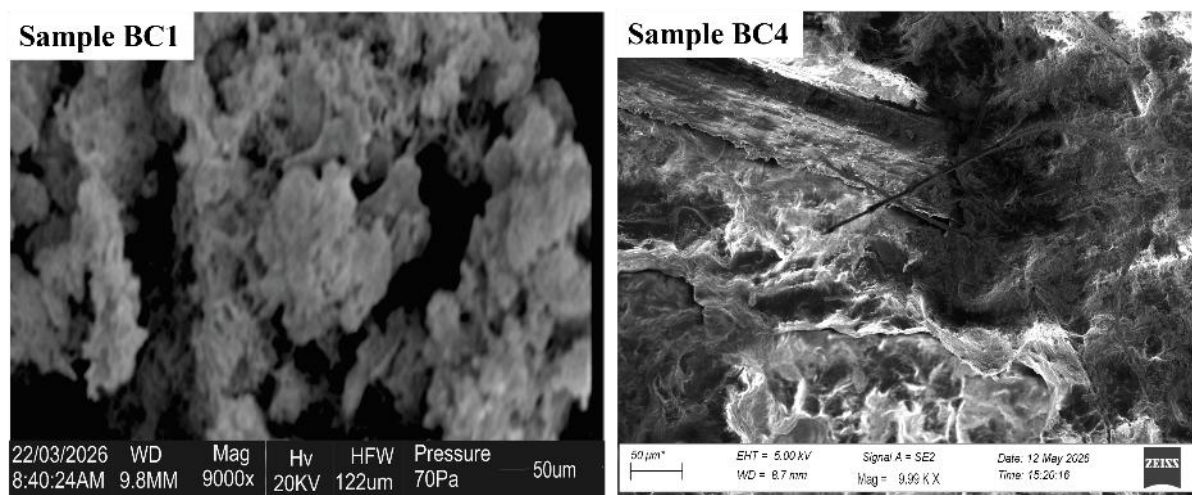


Figure 4: SEM images of the samples

3.0 CONCLUSION

The study demonstrated the successful improvement of the mechanical properties of PCL biopolymer-based composites through material modification. The FTIR spectra show C-H asymmetric stretching vibrations at 2947 and 2865 cm^{-1} , characteristic of polycaprolactone. The addition of reinforcing agents (PLA, HAp/Sr and keratin fibre) resulted in no new peaks, suggesting the materials were properly integrated into the PCL. There is a significant improvement in the mechanical performance resulting from the incorporation of PLA, keratin fibres, and bioactive fillers such as hydroxyapatite and strontium. Sample BC4, which comprises the three reinforcements, exhibited a significant increase in the mechanical properties when compared to BC1. For instance, the addition of the reinforcing agents increases the hardness and fracture toughness by 60.4% and 46.3%, respectively. Also, the tensile strength increased by 88.1%, while the Young's modulus increased by 50.6%. This enhancement is attributed to efficient stress transfer and improved interfacial bonding between the polymer matrix and reinforcing phases. Also, the improvement in both tensile strength and fracture resistance indicates that the developed biocomposites overcome the main limitation of pure PCL, achieving a more balanced combination of durability, toughness and stiffness. In conclusion, the sample BC4 had strong potential as a suitable material in bone repair and regeneration. The findings contribute to the ongoing development of high-performance biodegradable composites, while providing a foundation for further research on structural optimisation.

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