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Effect of Polyethylene Glycol (PEG) & Polyvinyl Alcohol (PVA) Concentrations on the Mechanical Behavior and Microstructure of Sintered Hydroxyapatite for Biomedical Use

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Abstract

The increasing number of road accidents has led to a growing demand for effective bone repair materials. Hydroxyapatite (HAp), a bioceramic that closely resembles the mineral component of natural bone, is widely used for this purpose. To enhance its mechanical properties, water-soluble binders such as polyethylene glycol (PEG) and polyvinyl alcohol (PVA) are commonly incorporated into HAp. This study was conducted with three main objectives: to characterise the fundamental material properties of Hydroxyapatite (HAp), to examine the role of binder composition (specifically PEG and PVA) in the fabrication of HAp composites, and to assess the mechanical properties of HAp composites with varying binder concentrations. Four samples were prepared using dry mixing and compaction methods, with PEG and PVA concentrations ranging from 1% to 5% by weight. Among the prepared samples, Sample 3—containing 3% PEG and 3% PVA—demonstrated the best overall performance. It exhibited high hardness (515.998 HV), moderate porosity (5.6467%), excellent yield strength (16.1356 N/mm^2), and a strong crystalline structure, indicated by an intensity count of 2100. These findings reveal that the ratio of PEG to PVA significantly affects the mechanical strength, porosity, and surface quality of HAp composites, making Sample 3 particularly promising for bone replacement applications.

1. Introduction

Hydroxyapatite (HAp) is a calcium phosphate mineral that plays a crucial role in biomedical applications due to its close chemical similarity to the mineral component of human bones and teeth. As a type of biomaterial, substances engineered to interact with biological systems for the purpose of supporting, repairing, or replacing damaged tissues or organs, HAp is particularly valued for its biocompatibility, osteoconductivity, and bioactivity. While biomaterials come in various forms such as polymers, biopolymers, and ceramics, they are not designed to fully replace the function of entire organs but rather to assist or restore specific functions.

In medical applications, HAp is widely used in orthopaedics and dentistry. Its ability to bond well with bone tissue makes it ideal for use in bone grafts, implant coatings, and other regenerative solutions. In dental care, it is used in implants and restorative treatments due to its natural integration with enamel and dentin. Beyond healthcare,

HAp also has environmental uses, such as in water treatment, where its high adsorption capacity enables the removal of heavy metals and other pollutants.

Current research efforts continue to focus on improving how HAp is synthesized and enhancing its mechanical strength, as well as better understanding how it interacts with the body to further expand its potential in clinical and environmental applications.

2. Biomaterial

Biomaterials are engineered materials designed to interact with biological systems to replace, repair, or enhance the function of tissues and organs. Their biocompatibility makes them essential in modern healthcare applications such as implants, tissue engineering, and regenerative medicine. They can be classified into several types: metallic biomaterials like titanium and stainless steel are strong and corrosion-resistant but may pose stiffness and toxicity issues; polymeric biomaterials such as PLA and polyethylene are lightweight and flexible, though their degradation can be a concern; ceramic biomaterials like hydroxyapatite and β-TCP offer excellent biocompatibility for bone use but are brittle; and biologically derived materials like collagen and chitosan are highly biocompatible yet mechanically weaker than synthetic options [1].

2.1 Bio-Ceramic

Bio-ceramics are ceramic materials designed for medical use, particularly in bone tissue engineering. They are known for their bioactivity, biocompatibility, and strong mechanical properties, enabling interaction with biological tissues to support healing and integration [2]. Bio-ceramics are categorized into three types: bioactive ceramics, such as hydroxyapatite and bioglass, which bond directly with bone; bioinert ceramics, like alumina and zirconia, which provide high strength and wear resistance with minimal biological interaction; and resorbable ceramics, including calcium phosphates, which gradually degrade and are replaced by natural bone [3]. They exhibit high compressive strength and wear resistance, though their brittleness limits tensile strength [4]. Biologically, they are highly biocompatible, support bone regeneration, and elicit minimal immune response. Nano-bioceramics, such as nanohydroxyapatite, offer enhanced bioactivity due to their larger surface area [5] .Their porous microstructure promotes cell adhesion, proliferation, and vascularization—key factors for bone regeneration [6]. Bio-ceramics are widely applied in bone regeneration as scaffolds or grafts, in drug delivery systems using nanostructures for targeted therapy, and in dental and orthopedic implants where bioinert ceramics provide durability and strength . Additionally, they enhance osteogenesis through improved cell-scaffold interaction [7].

2.2 Hydroxyapatite

Hydroxyapatite (HAp), with the formula $Ca_{10}(PO_4)_6(OH)_2$, is a calcium phosphate mineral that constitutes a major component of human bone and teeth. It is highly valued in medical and dental fields for its excellent biocompatibility, bioactivity, osteoconductive, and thermal stability, making it suitable for applications like bone grafts, implants, and drug delivery. Its hexagonal structure and Ca:P ratio of 1.67 closely resemble natural bone, supporting integration and bone regeneration. HAp can be synthesized via methods such as dry, wet, sol-gel, hydrothermal, and hydrolysis, each influencing its properties. Although it has poor mechanical strength for load-bearing uses, research is ongoing to improve this limitation. HAp exists in two forms: natural HAp, derived from sources like mammalian and fish bones or shells after organic removal, and synthetic HAp, chemically produced and free from trace elements. Despite similar composition, synthetic HAp is often preferred due to its lower biodegradability [8].

2.3 Binder

Binders play a vital role in the fabrication of hydroxyapatite (HAp)-based bioceramic scaffolds for bone tissue engineering by enhancing particle cohesion, shaping, and mechanical strength. They also influence key scaffold properties like porosity, surface roughness, and degradation rate, which are essential for cell attachment and bone regeneration. Common binders include natural polymers such as polyvinyl alcohol (PVA), gelatin, and chitosan, and synthetic ones like PEG and PLGA. PVA, in particular, is widely used due to its water solubility, biodegradability, and ability to improve surface roughness and osteoblast attachment when combined with PEG. The binder content typically ranges from 5% to 20%, with 10% often used to balance structural integrity and porosity. Choosing the right binder type and concentration is crucial for ensuring optimal scaffold performance, biocompatibility, and gradual degradation during bone healing [9].

2.3.1 Polyethylene Glycols (PEG)

Polyethylene glycol (PEG) is a widely used binder in hydroxyapatite (HAp)-based bio-ceramic scaffolds due to its ability to enhance mechanical strength, structural integrity, and biocompatibility. It improves particle dispersion and contributes to scaffold stability, as demonstrated in nanoparticle formulations containing PEG, HAp, and hyaluronic acid. PEG also supports controlled drug release, as seen in studies involving Zoledronic acid-loaded composites. When combined with polymers like polylactic acid or polycaprolactone, PEG improves flexibility, ductility, and printability, making it ideal for load-bearing and 3D-printed bone scaffolds. These properties make PEG valuable in improving scaffold performance, drug delivery, and bone regeneration [10].

2.3.2 Polyvinyl Alcohol (PVA)

Polyvinyl alcohol (PVA) is a versatile, water-soluble, and biodegradable polymer widely used in biomedical and environmental applications. Its film-forming ability and compatibility with other polymers make it valuable for drug delivery systems, tissue engineering, and eco-friendly packaging. In bone tissue engineering, combining PVA with hydroxyapatite (HAp) improves scaffold strength and porosity, aiding bone regeneration. PVA also plays a key role in forming water-resistant films for packaging and medical use, particularly when blended with natural polymers. Its biodegradable and non-toxic nature further enhances its suitability for sustainable applications in both medical and food industries [11].

3. Methodology

The flowchart in the figure depicts the entire process of completing the project from initiation to completion. This experiment utilizes hydroxyapatite and binders, with mechanical properties evaluated throughout the procedure.

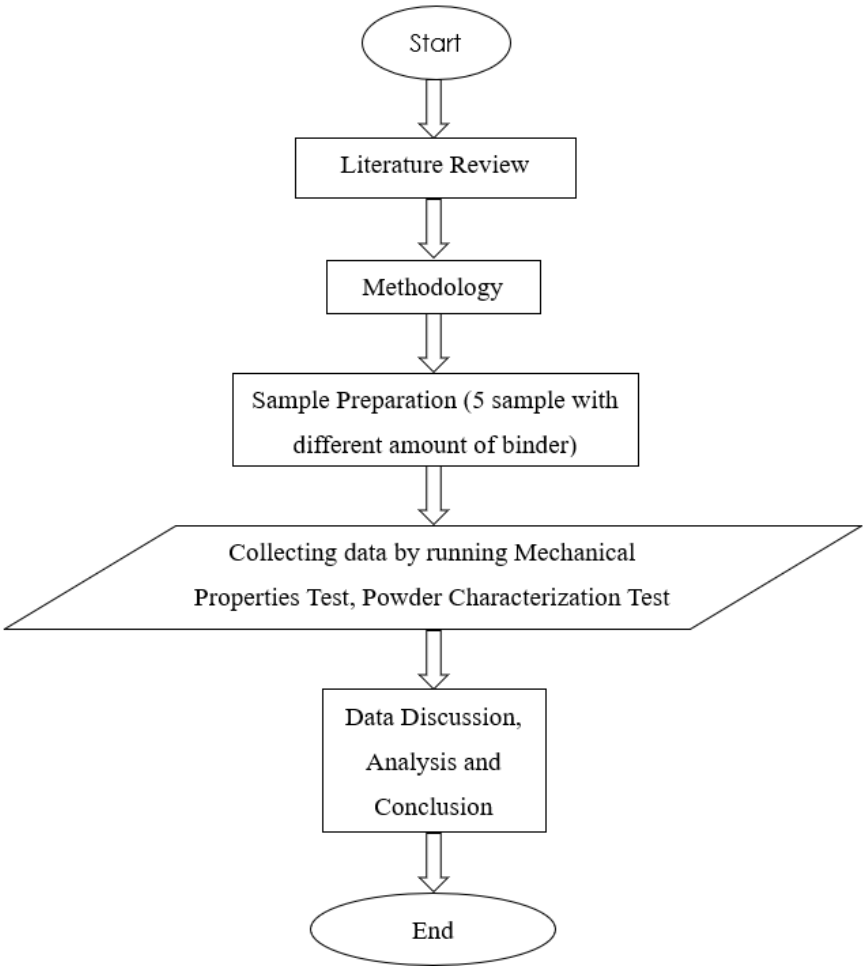

Fig.1 Experiment Flowchart

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4. Result & Discussion

Two test categories have been successfully conducted. The first focuses on the characterization of hydroxyapatite powder as an initial assessment, while the second, performed post-machining, delves into further analysis. The characterization of hydroxyapatite powder involved a series of tests, including X-ray diffraction, scanning electron microscopy (SEM), modulus of rupture (MOR), surface roughness, density, porosity, and evaluations of sintering shrinkage. All collected data were meticulously examined to support discussion and conclusions.

4.1 Shrinkage

A shrinkage test was carried out to gain insight into the composite's behavior and improve its mechanical strength and bioactivity. The process commenced with preheating the HAp sample at 56°C for 24 hours to eliminate water content, followed by sintering at 1200°C with a holding time of 120 minutes. Shrinkage was measured by recording the sample's length before and after sintering using vernier calipers, which offer an accuracy of ±0.005 mm. The recorded measurements, summarized in Fig.3, were then utilized to determine the percentage of shrinkage.

Table 1 Hydroxyapatite sample dimensions

Mixture Ratio		Weight (g)		Length (mm)		Width (mm)		Thickness (mm)	
PEG	**PVA**	**Before**	**After**	**Before**	**After**	**Before**	**After**	**Before**	**After**
1%	5%	14.3278	9.7550	102	80.109	10	8.142	7.965	6.253
2%	4%	16.7682	11.7371	102	80.021	10	8.001	8.289	6.232
3%	3%	9.1098	6.9840	102	70.789	10	7.822	6.112	4.001
4%	2%	12.3221	8.1883	102	70.923	10	7.438	7.466	5.114
5%	1%	11.985	9.3540	102	80.112	10	7.244	8.533	6.012

4.1.2 Modulus of Rupture (MOR)

The Modulus of Rupture (MOR) test was conducted to determine the maximum stress a material can endure before fracturing under bending loads. This test provides insight into the mechanical properties of the HAp sample. The table below presents the recorded maximum stress values obtained from the testing.

Table 2 Max Stress and Strain obtained from testing

Sample		Max Stress (N/mm^2)	Max Strain (%)
PEG	PVA		
1%	5%	12.7399	0.45772
2%	4%	28.5292	1.26642
3%	3%	26.3833	0.69510
4%	2%	-	-
5%	1%	-	-

4.1.3 Hardness Test

Vickers hardness measures a material's hardness by analyzing the size of the indentation left by a diamond pyramid-shaped indenter under an applied load. The HAp samples underwent three indentations at optimal points using a constant load of 1.961N (HV 0.2). The mean Vickers hardness was then derived from these trials. The data obtained from the experiment is presented in the table below, with the mean hardness value summarized in Fig.3.

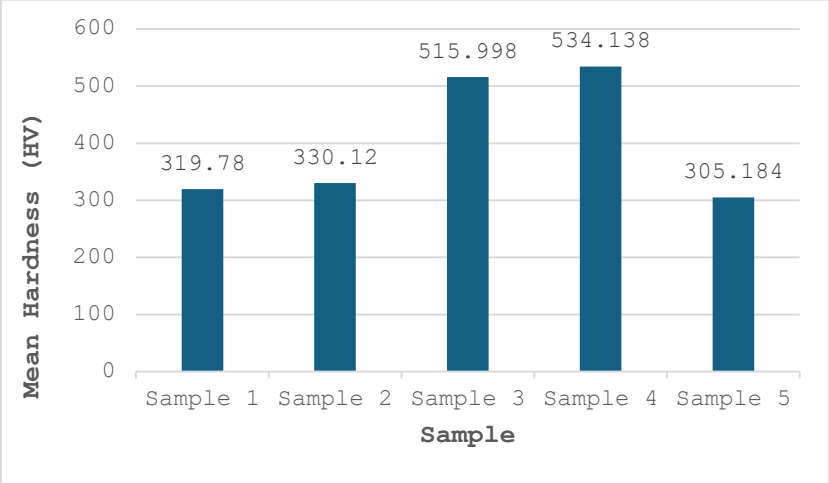

Fig.2 Graph Mean Hardness Result

4.1.4 Flexural

The Modulus of Rupture (MOR) test was performed to determine the maximum bending stress a material can endure before fracture, thereby evaluating the mechanical properties of the hydroxyapatite (HAp) samples. Table 3 presents the recorded maximum stress and load values obtained from the testing. Sample 3, with 3% PEG and 2% PVA provides the optimal composition for maximum yield strength.
However, for the samples containing 4% PEG and 5% PEG, the MOR test could not be conducted successfully, as all prepared specimens fractured prematurely during handling or preparation, preventing reliable measurements.

Table 3 Yield Strength of HAp samples

Sample		Yield Strength
PEG	PVA	(N/mm^2)
1%	5%	8.7396
2%	4%	12.4831
3%	3%	16.1356
4%	2%	-
5%	1%	-

4.1.5 Porosity & Density

The table shows how different ratios of PEG and PVA binders affect the porosity percentage in five samples. Samples with low PEG and high PVA (1% PEG, 5% PVA) resulted in the highest porosity (18.33%), indicating poor compaction. In contrast, samples with higher PEG content (2% PEG, 4% PVA and 5% PEG, 1% PVA) achieved the lowest porosity (1.54%), suggesting better binding efficiency. A balanced 3% PEG and 3% PVA mix led to moderate porosity (5.65%), while a 4% PEG and 2% PVA combination showed slightly lower porosity (4.52%). The data indicates that increasing PEG while reducing PVA generally reduces porosity, with the optimal ratios being either 2% PEG/4% PVA or 5% PEG/1% PVA for minimal porosity.

Table 4 Percentage Porosity of HAp

Sample	Binder		Percentage of Porosity %
	PEG	PVA	
1	1%	5%	18.3342
2	2%	4%	1.9792
3	3%	3%	5.6467
4	4%	2%	4.5211
5	5%	1%	1.5390

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4.1.6 Scanning Electron Microscope (SEM)

A microstructure analysis of hydroxyapatite (HAp) was performed to investigate both the surface and internal structure of the material in detail. In this study, HAp samples were prepared using different binder percentages, with the powder sieved before being compacted into samples. The sintered HAp body was examined using scanning electron microscopy (SEM) at a magnification of 1500x, enabling a detailed assessment of the pores formed after sintering. The main objective of this experiment was to compare the porosity observed in the SEM micrographs with the porosity percentages derived from previous measurements. This comparison serves to validate whether the prior porosity data accurately reflect the visual pore distribution within the microstructure.

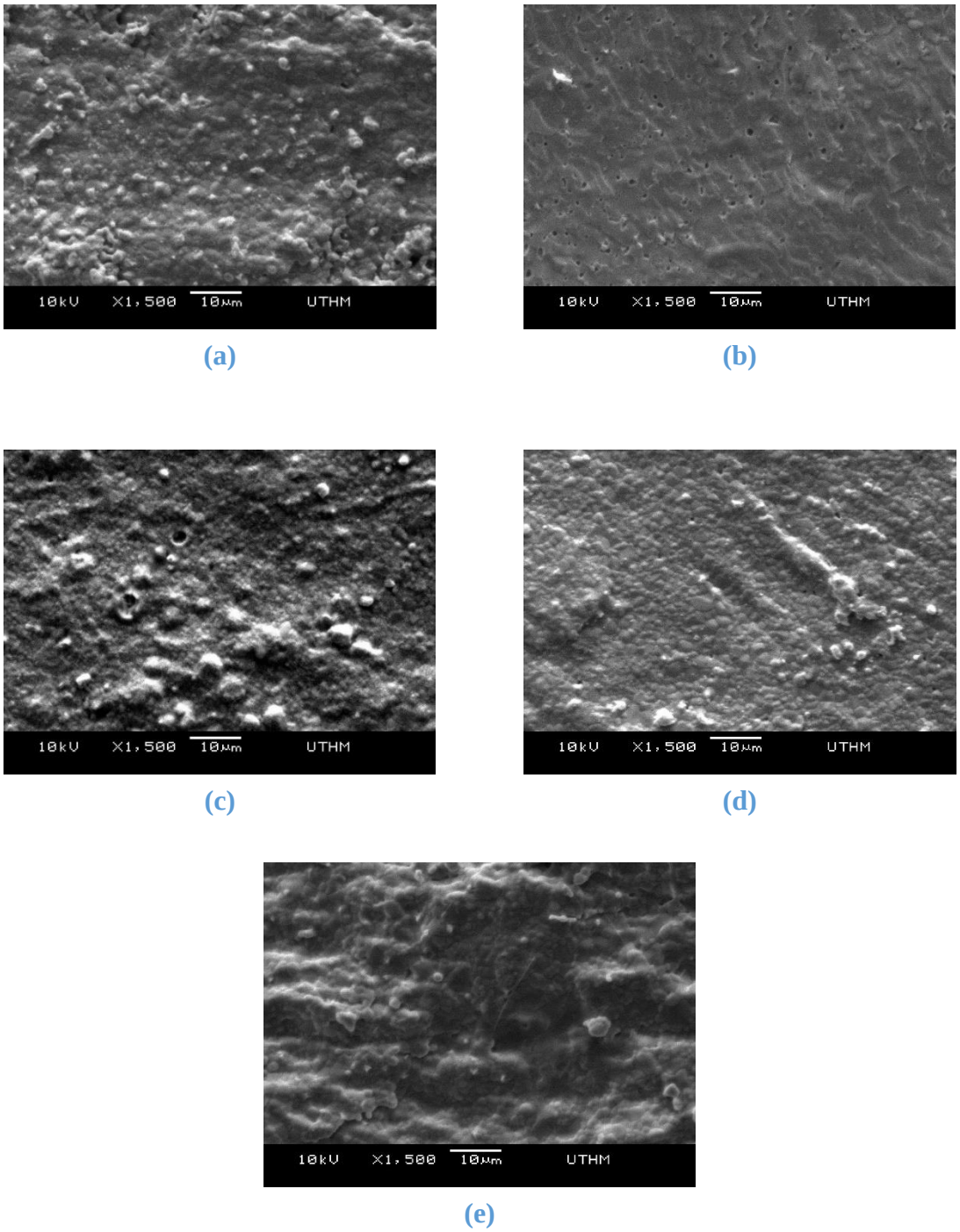
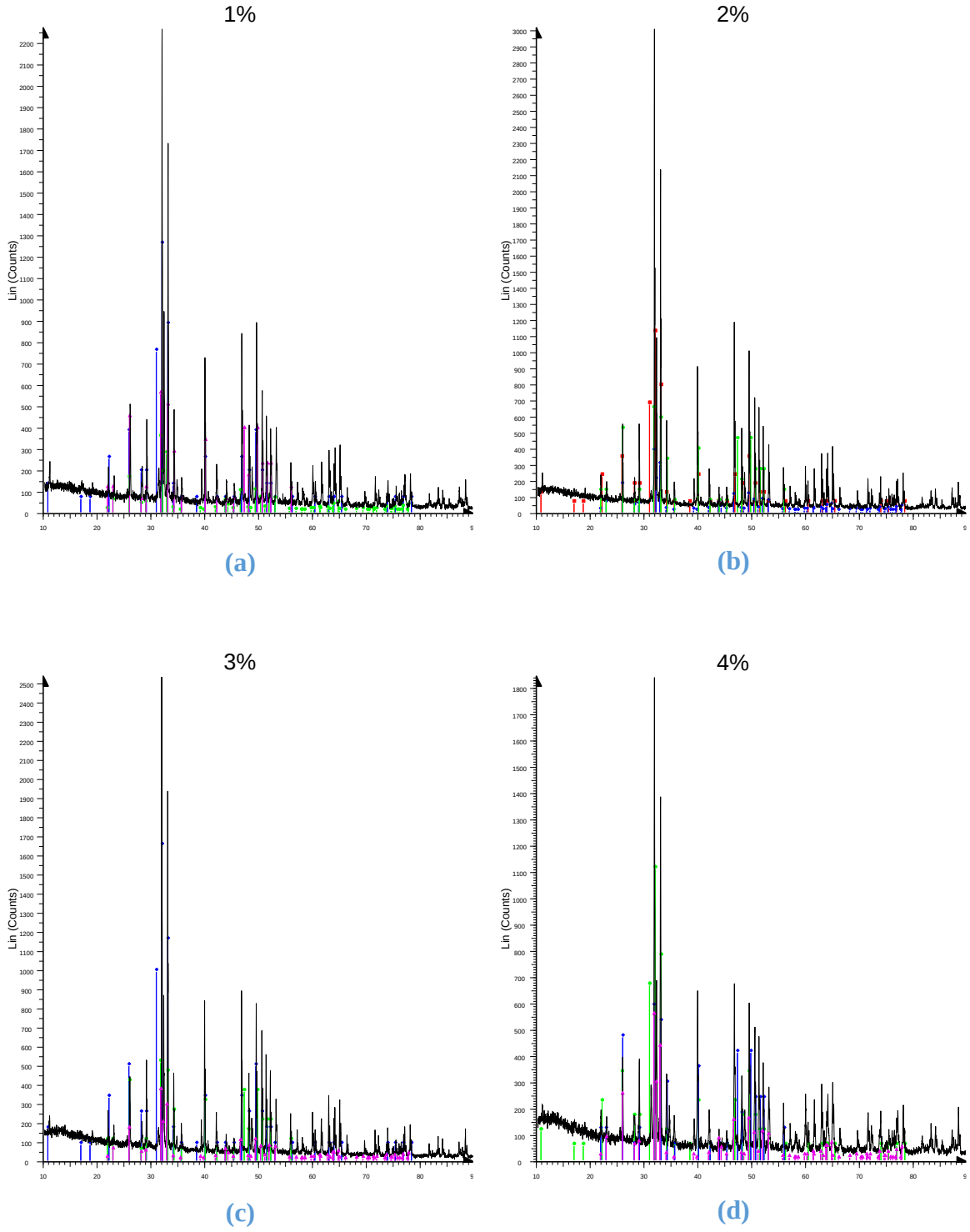


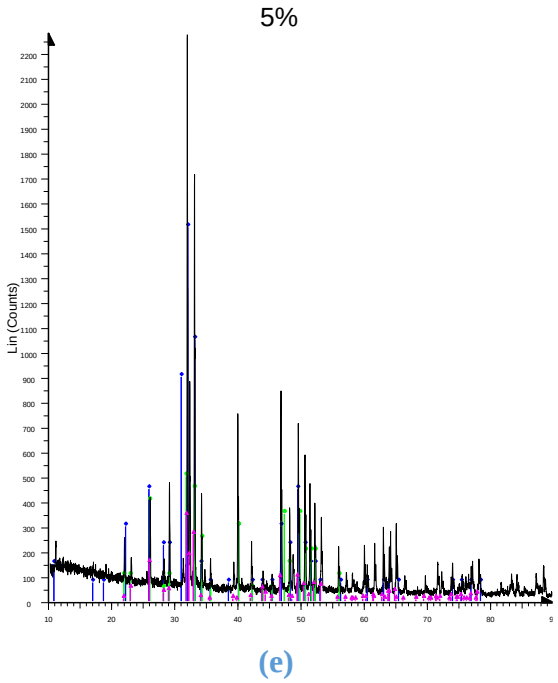
Fig.3 *Hydroxyapatite surface SEM micrograph (a) 1(PEG 1 PVA 5); (b) 2 (PEG 2 PVA 4); (c) 3(PEG 3 PVA 3); (d) 4(PEG 4 PVA 2); (e) 5(PEG 5 PVA 1).*

4.1.7 X-ray Diffraction (XRD)

X-ray Diffraction (XRD) analysis was carried out to determine the highest peak in the hydroxyapatite (HAp) samples. The findings revealed that all samples exhibited the same nanocrystalline structure of HAp. The analysis indicated that the HAp powder, regardless of the binder ratio, possesses a hexagonal crystal structure with a space group of P63/m (No. 176). The measured lattice parameters were a = b = 9.41890 Å and c = 6.88270 Å, with z = 1.

Further calculations determined the molecular weight of the HAp sample to be 1004.64 g/mol, along with a detected volume of 528.80. The phase identification was performed over a 2θ range of 10° to 90°, matching the peak patterns with the reference JCPDS card 00-055-0592. The results confirmed that all synthesized samples maintained the nanocrystalline form of stoichiometric hydroxyapatite, $Ca_{10}(PO_4)_6(OH)_2$.





(e)

Fig.4 *X-ray Diffraction Pattern (a) 1(PEG 1 PVA 5); (b) 2 (PEG 2 PVA 4); (c) 3(PEG 3 PVA 3); (d) 4(PEG 4 PVA 2); (e) 5(PEG 5 PVA 1).*

5. Conclusion

The surface quality of machined bioceramics is highly dependent on the binder composition used in their fabrication. This study investigates the influence of polyvinyl alcohol (PVA) and polyethylene glycol (PEG) binders on the surface roughness and mechanical properties of hydroxyapatite (HAp) samples processed through mixing, compaction, sintering, and end mill tungsten machining. Various binder concentrations (1-5 wt%) were evaluated, with comprehensive characterization including shrinkage analysis, mechanical strength testing, microscopy, hardness measurements, and porosity/density assessments.

Results indicated that binder formulation critically affects HAp's performance characteristics. The optimal composition was achieved with Sample 3 (3% PEG and 3% PVA), which exhibited superior properties including: minimal surface roughness, high hardness (515.998 HV), low porosity (5.6467%), and excellent yield strength (16.1356 N/mm^2). These properties were complemented by a well-developed crystalline structure (2100 intensity count), confirming the material's suitability for biomedical applications requiring precise surface finish and mechanical reliability.

This study demonstrates that careful optimization of PEG-PVA binder ratios can significantly enhance the surface quality and mechanical integrity of machined HAp bioceramics. The findings provide valuable guidelines for developing high-performance bone graft materials with tailored surface characteristics for specific clinical applications.

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