

Chapter 2

Background and Literature Review

2.1 Introduction

The presented chapter briefly reviews research work reported by different researchers in the development field, ATZ, SiO₂ (RHA waste derived), and Hydroxyapatite (AWB waste derived) based biomaterials for tissue engineering applications. The chapters review different processing techniques employed in the fabrication of ATZ and ceramic HAP/SiO₂-based composite, along with their advantages, disadvantages, and applications. Thus, this chapter explains ceramic-based biomaterial processing.

Zirconia (ZrO₂) is a versatile bioceramic widely used in medical implants due to its exceptional mechanical strength, hardness, and wear resistance. Common applications include dental crowns, bridges, and implants in orthopedic and dental prosthetics [1]. The crystallographic phases of zirconia under atmospheric pressure- monoclinic (m), tetragonal (t), and cubic (c)-are crucial. The tetragonal-to-monoclinic (t-m) transformation at around 1000°C results in volume expansion and shear strains, impacting its use as a structural material [2]. Yttria (Y₂O₃) addition stabilizes the t phase, enabling stress-induced phase transformation and enhancing toughness [3]. Y-TZP (yttria-stabilized tetragonal zirconia polycrystal) is widely used, especially in hip prostheses. However, surface exposure to water can lead to hydrothermal aging or Low-Temperature Degradation (LTD), causing roughening and microcracking [4]. Studies reveal a slow t-m transformation in humid conditions, initiating microcracking and strength loss. This phenomenon contributed to a notable failure in 2001 [5].

Several Strategies have been attempted to prevent Low-Temperature Degradation (LTD), including exploring alternative ceramic materials, such as alumina–zirconia and zirconia–alumina composites, and reducing grain size or increasing yttria content in the starting powder, etc. [6,7]. However, these approaches reduce zirconia toughness, diminishing its mechanical appeal. Another strategy involves the addition of dopants, such

as alumina, silica, copper oxide, cerium oxide, iron oxide, etc., which has proven effective in slowing down Low-Temperature Degradation (LTD) [8]. Alumina toughened zirconia (ATZ) is a preferred choice in contemporary orthopedic surgery due to its similar mechanical properties and reduced sensitivity to aging [9]. Schneider (2008) observed that an ATZ-ceramic composite (80 wt% Y-TZP and 20 wt% alumina) demonstrated decelerated aging in a humid atmosphere at body temperature over 50 years [10]. Li et al. (1997) suggested that Al_2O_3 suppresses water-induced phase transformation by interacting with H_2O , forming a protective hydroxyapatite layer on Al_2O_3 -dispersed particles [11]. This shields the sample surface from further interaction with H_2O and prevents LTD transformation. Accumulation of alumina in the first 5–10 monolayers significantly stabilizes zirconia and improves Low-Temperature Degradation (LTD) resistance. Vleugels found the highest toughness contribution at an alumina fraction of 2.5 vol%, while a further increase (> 10 vol%) leads to alumina toughened zirconia, a material with exceptional strength [12]. As alumina addition also enhances wear resistance, ATZ becomes an intriguing material for biomedical implants. Kern et al. (2012) attempted to improve the mechanical properties of Y-TZP/alumina composites by substituting coprecipitated 3Y-TZP with tougher and more transformable 2.5Y-TZP produced through powder coating [13].

Silica emerges as a compelling dopant for enhancing the stability of 3Y-TZP. The exploration of silica's impact on the hydrothermal aging of YSZ samples dates back to 1986 when Lange et al. investigated its effects on YSZ samples, revealing that the aging phenomenon stems from water vapor, with intergranular silicate glassy phases playing an insignificant role [14]. In 1987, McCartney studied how an amorphous second phase changes the qualities of Y-TZP when silica is used. It was observed that samples with a higher glass phase content experienced less degradation, refuting the notion of stress

corrosion cracking as the primary cause of degradation [15]. This study supports the idea that glass reduces residual stresses from thermal expansion anisotropy. A decade later, Tan and co-workers demonstrated that the use of silica encourages the formation of intergranular glassy layers, fortifying the anti-degradation resistance of Y-TZP [16]. In 2000, Gremillard et al. illustrated the effects of small silica additions on the microstructures and mechanical properties of 3Y-TZP ceramics [17]. Silica was identified at triple junctions, resulting in a rounded microstructure and reduced internal stresses, thereby significantly enhancing Low-temperature degradation (LTD) resistance without impacting slow crack growth (SCG) behavior [18]. Further insights from Takigawa et al. in 2009 highlighted that 0.1 mol% SiO₂ doping retards the t-m phase transformation of 3Y-TZP [19]. The presence of silicon ions segregating along grain boundaries alters the grain boundary diffusivity of hydroxyl ions, influencing phase transformation behavior. Nakamura et al. 2011 demonstrated that silica doping led to ceramics with reduced monoclinic content, enhanced resistance to degradation, and minimal strength reduction compared to conventional Y-TZP [20,21]. In 2015, Samodurova et al. conducted a comprehensive study on silica doping, both with and without alumina co-doping in 3Y-TZP [22]. The findings indicated that silica concentration at multiple grain junctions contributed to the rounding of zirconia grains without affecting fracture mode. Co-doping with alumina and silica showcased distinct mechanisms decelerating LTD in 3Y-TZP, and their combined use increased resistance to aging without compromising material fracture toughness. Recent research by Alves et al. and Ramos et al. has further emphasized the protective role of a silica glass layer in preventing LTD and antagonistic wear [23,24]. Additionally, this layer enhances bondability to resin cement when applied to the inner surface. Collectively, these studies underscore the efficacy of silica, whether used as a dopant or as an outer layer material in Zirconia-based ceramics, as an exceptional means

of reducing LTD. Notably, while various forms of silica exist, including crystalline and amorphous, there remains a gap in understanding the optimal form for mitigating LTD in Zirconia-based bioceramics.

In addition to traditional methods utilizing additives and dopants for enhancing the resistance to Low-Temperature Degradation (LTD) and other properties of bioceramics, there is a growing interest in exploring alternative solutions that not only tackle these challenges but also bring about economic advantages while reducing waste [25].

Silicon dioxide, one of the most commonly used materials in the scientific fraternity, is found in multiple forms in nature. Silica has both amorphous and crystalline forms, with quartz, tridymite, and cristobalite as their most stable crystalline forms[26]. The potential application of silica is not limited to structural refractories; they are also widely used by semiconductor and insulator industries and are excellent adsorbents [27,28]. The several intrinsic properties of silica make it a suitable candidate to be used as the matrix for incorporating other functional components and to be used as a reinforced material for different matrix phases [29]. The properties like water solubility, thermal stability, chemical and magnetic field inertness, excellent biocompatibility, and cheap availability give silica an extra edge toward being used for various applications [30]. Composites developed with the help of silica as a matrix or reinforcement phase possess unique mechanical, thermal, magnetic, optical, and electric properties [31]. Silica-based materials are also found in advanced applications like radome [32]. Apart from dense silica, porous silica has been widely used in various applications [33]. Fu et al. have successfully utilized a porous silica-based composite to remove phosphorus [33]. Similarly, Kobayashi studied the effect of porous silica on removing tar components and found it very efficient [34]. Wu et al. have shown good adsorption and separation behavior of porous silica towards Y(III) and Sr(II) in acid solution [35]. Silica, mostly available as quartz in the silica sand, is the

Cheapest source of silica; it is also found in various rocks in tridymite and cristobalite forms. However, some other typical chemical routes can be used to synthesize silica. In the past few decades, silica from Rice Husk (RH) and agricultural waste has emerged as the cheapest source of silica [36]. Both amorphous and crystalline forms of silica are available through RH under suitable conditions [37].

Millions of tons of rice are produced globally per annum. According to the literature, around 200 kg of RH is produced per metric ton of rice. Disposal of RH is a great challenge associated with rice production due to its large surface area and carbon content, causing a pollution threat to the environment. Efforts have been made to utilize RH properly without affecting the environment. Some researchers used RH directly to reinforce it with other components, while most of them have used it as ash to utilize its high silica content [38]. Hydrated Silicon, cellulose, and lignin are the major constituents of Rice husk [39]. Various approaches, including chemical and thermal treatment, have synthesized silica from RH [40]. However, thermal treatment is the most preferred one. The silica produced requires minimum grinding due to the highly reactive nature of silica particles, which can be considered the main advantage of this process. Carbonaceous constituents of the rice husk provide the energy required for its pyrolysis, which produces highly porous silica with a large surface area [41]. During the whole process, 20 wt.% of rice husk remains as the burnt-out residue, of which 95 wt.% contains silica. It has been studied earlier that the quality of silica produced from rice husk ash depends on sintering temperature and time. These sintering conditions are also responsible for the structural transformation of silica [42]. RH sintered at a lower temperature of 800°C produces amorphous silica, and crystalline silica is found above 900°C [43]. The most widely recognized types of crystalline silica are quartz and cristobalite. The tridymite phase may also be evident in some cases, depending on the percentage of impurity content in RHA [44]. Many studies

have demonstrated the efficiency of using RHA-based silica precursors for ceramic/metal/polymer-based composite production to improve their mechanical properties [45-47]. Santos et al. investigated the effect of RHA-kaolin-based composite reinforced with corrugated steel fibers [48]. The results showed that the composite has a high degree of deformation at the point of rupture and better thermal shock resistance. The effect of reactive silica from RHA has been investigated on the mechanical and microstructural properties of metakaolin/volcanic ash-based geopolymer [49]. The higher mechanical strength of geopolymer is obtained due to the enhanced compactness provided by RHA silica. Mechanical and wear behavior is analyzed on aluminum composites using alumina, RHA, and graphite as reinforcement, and it is found that tensile strength becomes higher when 50% RHA is used [50]. Similarly, the effect of RHA silica reinforcement on copper matrix and their wear behavior is analyzed by Dinaharan et al., who found a decreased wear rate of Cu/RHA composite [51,52]. The role of RHA silica on polymer-based composites is also widely studied. Ayswarya et al. studied the role of RHA on epoxy composites and found that nano-silica obtained from RHA gives better tensile strength and thermal stability to epoxy composites [52]. RHA-based silica is also used to fabricate a variety of silicate ceramics and silicon-based non-oxide ceramics like SiC and Si₃N₄ [53,54]. The above investigations provide enough evidence for using RHA-based silica with different materials, showing its importance in improving the mechanical and other properties of metal/ceramic/polymer-based composites. Both amorphous and crystalline forms of silica are available through RHA. Researchers have extensively used these forms of silica to get anticipated results. However, none of the research has been focused on the selective study of these two different forms of RHA-based silica as well as their composites. Thus, we herein report a systematic study on the various available forms of silica obtained through the thermal treatment of RH, including their microstructural and mechanical

properties. Thermal treatment of RH has been carried out in various forms. Four different temperatures treated RHA silica, both in amorphous and crystalline form, are used to fabricate silica compacts. Sintering is done between 1450°C-1550°C for the final consolidation of silica compacts. Effects of silica structure and phase percentage on compaction behavior, green density, sintered density, microstructure, and mechanical properties have been analyzed. XRD and SEM analysis is used to study different phases of silica and their microstructure, respectively. Finally, a comparative analysis of these forms on the mechanical and microstructural properties of silica compacts has been assessed.

The current socioeconomic conditions of the developing world have presented significant challenges, such as ensuring food security, maintaining water quality and availability, mitigating global warming, managing limited energy resources, and promoting environmental conservation [55]. Unfortunately, many of the proposed solutions to these challenges can create further obstacles. For instance, food waste is a persistent issue that affects both food security and economic prosperity. One such source of food waste is animal waste bone (AWB), with estimations suggesting a staggering annual contribution of approximately 20 million metric tons on a global scale [56]. AWBs constitute a significant byproduct of the food industry and waste from homes, with an estimated annual global production exceeding 130 million metric tons [57]. This surplus stems from a diverse array of sources, such as slaughterhouses, eateries, and even everyday households. Efficiently handling this waste presents a formidable obstacle as it carries the risk of infectious disease transmission, compounded by the unpleasant odor accompanying direct ground disposal [58]. Furthermore, the improper disposal of such waste onto agriculturally productive and fertile land can lead to soil contamination and the impairment of land fertility. Improperly managing the disposal of animal bone waste originating from restaurants and slaughterhouses not only incurs additional costs in terms of capital and

resources for appropriate disposal [59] but also results in potential revenue loss. Conversely, harnessing the potential of these by-products in a resourceful manner can yield substantial economic benefits for a nation. Failing to address this waste mismanagement not only undermines environmental conservation but also hampers the pursuit of sustainable development goals.

In addition to traditional approaches, there is a growing interest in exploring alternative solutions that not only address these pressing issues but also generate economic benefits while minimizing waste [60]. The circular economy, or more specifically, the bioeconomy, has emerged as a promising approach advocated by global experts to tackle these complex and interconnected problems [61]. The paradigm of biocircularity represents a holistic approach to the sustainable production of renewable biological materials, prioritizing their extended usage, optimal reuse, and recycling [62]. Researchers have delved into the exploration of animal waste bone (AWB) for diverse applications, capitalizing on its inherent composition, particularly hydroxyapatite (HAp). Simple transformation processes enable the conversion of HAp into tricalcium phosphate (TCP) [63]. The catalytic, thermal, and chemical stability exhibited by HAp makes it an attractive candidate for biodiesel production [64-66]. AWB derived from different animal species possesses varying elemental compositions, resulting in different catalytic properties. For instance, Khan et al. developed a low-cost biodiesel catalyst from ostrich waste bone, while Chingakham et al. synthesized a heterogeneous catalyst for biodiesel transesterification by calcining and hydrothermal reaction of AWB [67,68]. In a similar vein, Prabu et al. undertook the synthesis of a magnetic absorbent derived from lamb bone, showcasing its efficacy in the removal of heavy metals from aqueous solutions [69]. Amiri et al., on the other hand, harnessed ostrich waste bone to effectively eliminate cobalt from wastewater and activate peroxymonosulfate for dye degradation [70]. The versatility of AWB extends

to its composition, as it serves as a rich source of essential amino acids, minerals, and vitamin B12, making it a suitable candidate for the preparation of animal feed products [71]. Moreover, Gendy et al. successfully synthesized a green nano-biocatalyst, fluorapatite, from waste bone to purify wastewater generated by the petroleum industry [72].

However, the most profound AWB utilization comes from biomaterial production due to the abundance of hydroxyapatite content [73]. Hydroxyapatite (HAp) is a critically crucial bioactive material found abundantly in natural hard tissues, such as bones and teeth, constituting approximately 70% of their structure [74]. Its remarkable biocompatibility and bioactivity make HAp the most suitable ceramic material for prosthetic applications, including artificial bones and teeth [75-78]. Non-stoichiometric hydroxyapatite, obtained from diverse sources such as fish, bovine bones, eggshells, and chicken bones, has exhibited remarkable biological characteristics surpassing those achieved through synthetic means [79]. This superiority can be attributed to the presence of essential ions like Cl, F, K, Mg, Na, SO₄, and Sr, which contribute to advantageous properties facilitating accelerated bone regeneration [80]. Among these minor elements, magnesium (Mg) is present at a higher concentration, contributing to the formation of the Whitlockite phase [81,82]. The inorganic component of human bone consists predominantly of hydroxyapatite, with a secondary presence of whitlockite, in a ratio of 3:1 [83]. Heat treatment of animal bones can produce a composite material called HAp/TCP/Whitlockite, which possesses excellent biocompatibility, bioactivity, and osteoconduction properties [84]. However, high-temperature treatment induces the transformation of β -TCP into α -TCP, a less biocompatible material than β -TCP [85]. Nonetheless, in the presence of magnesium, the sintering process of TCP phases allows for higher densification. Furthermore, the presence of magnesium during the cooling process leads to a reversible

transition from β to α , indicating that Mg^{2+} promotes the kinetics of α to β conversion upon cooling or slows down the kinetics of the β to α transition upon heating, thereby increasing the time required to reach equilibrium [86]. Emerging research has highlighted the enhanced bone healing potential of composite materials composed of hydroxyapatite and other calcium phosphate phases. These mixtures, specifically biphasic or biphasic/triphasic materials, have been shown to outperform single-phase materials in addressing defect areas and promoting effective bone regeneration [87,88].

Despite the advantages of calcium phosphate-based biocomposites, their degradation caused by rapid revascularization has resulted in weakened pore structures unsuitable for load-bearing applications [89]. Consequently, developing mechanically robust scaffolds based on calcium phosphate is of utmost importance in replicating the properties of natural bone [90]. To achieve this, such composites' mechanical properties, particularly the strength and fracture toughness, need improvement. One such approach is the intrusion of either high-strength material into Calcium phosphate composites or the doping of calcium phosphate into high-strength composites. Ceramic materials such as zirconia and alumina have emerged as excellent biomaterials, overcoming the limitations of calcium phosphate with their higher strength and improved mechanical properties [91,92]. Alumina exhibits excellent biocompatibility and mechanical strength, making it suitable for load-bearing implants and orthopedic devices. However, its brittleness can be a disadvantage, potentially leading to fractures under high stress [93]. Conversely, Zirconia demonstrates exceptional biocompatibility, mechanical properties, and resistance to wear and corrosion. Both alumina and zirconia have certain drawbacks: alumina is hard to machine and prone to brittleness. In contrast, zirconia can be difficult to sinter and undergoes a phase transformation at high temperatures, causing weakness [94]. Alumina toughened zirconia (ATZ) has been developed as a composite material to address

these limitations. Alumina toughened zirconia (ATZ) combines the properties of alumina and zirconia by dispersing alumina particles in a zirconia matrix. This composite enhances resistance to crack propagation, reducing the risk of catastrophic failure. Alumina toughened zirconia (ATZ) offers improved toughness and reliability compared to pure alumina or zirconia [95]. Consequently, Alumina toughened zirconia (ATZ) has gained popularity in biomedical applications, including dental crowns, bridges, implants, artificial joints, and bone plates, where higher toughness and reliability are required [96,97].

This study aims to determine the best amorphous/crystalline silica addition for Alumina toughened zirconia (ATZ) biocomposites to improve LTD resistance. The research also incorporates Rice Husk waste-derived silica to promote biocircularity in biomaterial synthesis. Amorphous and crystalline silica from Rice Husk Ash (RHA) were compared for hydro-aging, mechanical performance, and other properties. This research improves bioceramics and promotes sustainability by using agricultural waste to make biomaterials. The preceding studies demonstrate the importance of RHA-based silica in improving mechanical and other properties of metal/ceramic/polymer-based composites. RHA produces amorphous and crystalline silica. Researchers have frequently used these silica forms to achieve expected outcomes. No research has selectively studied these two RHA-based silica forms or their composites. Thus, we present systematic research on thermally treated RH silica's microstructural and mechanical properties. RH has been thermally processed into several forms. Silica compacts are made from amorphous and crystalline RHA silica treated at four temperatures. Silica compacts are consolidated by sintering at 1450°C-1550°C. The impact of silica structure and phase % on compaction, green density, sintered density, microstructure, and mechanical characteristics was examined. XRD and SEM analyze silica phases and microstructure. Finally, these shapes' mechanical and microstructural effects on silica compacts were compared.

A HAp/TCP/Wk-ATZ biocomposite is being developed by adding waste-derived calcium phosphate phases into an alumina toughened zirconia (ATZ) composite to promote biocircularity in biomaterial synthesis. This composite improves bioactivity while maintaining mechanical characteristics. The bioactivity of the ATZ composite can be increased by adding animal waste bone (AWB) to strengthen bone tissue connections. The major inorganic component of natural bone, HAp/TCP/Wk, is bioactive and promotes bioactive interfaces with adjacent bone tissue. This improves osseointegration and implant stability. Waste-based dopants and inexpensive materials like alumina can be used to make a zirconia-based biocomposite with enhanced bioactivity.

2.2 Background

2.2.1 Bioceramics and Biocomposites

Most of the work that goes into orthopedic surgery depends on the progress made in biomaterials, which are used to fix and repair damaged body parts. The body is susceptible to injury in many ways that are painful and can't be fixed, such as dislocations, strains, and fractures. Fractures are breaks in bones that are usually caused by forces that are stronger than the bone tissue. Surgery is done to put in extra material that can support the body weight so that the broken bone can heal. The implanted materials are usually called biomaterials, and they should have good mechanical properties, like an elastic modulus, a yield strength, and an ultimate tensile strength, so that they can handle different biomechanical forces. In addition, the implanted materials should have low density, good biocompatibility, and great resistance to corrosion and wear. Because of these qualities, it may be used in rods, bone plates, screws, joint replacements, wires, cardiovascular stents, and oral implants, as well as other load-bearing devices. Some implant materials don't last long because they wear out, biological factors like illness, implants coming loose, and the

material's low strength and inability to match the bone or other body parts' elastic modulus. Because of this, the need for a second treatment becomes clear. There are a lot of problems with revision surgery, like how much it costs, how painful it is, and how often the inserted part works. Choosing the right material for a medical device can be hard because of how it behaves mechanically, biologically, and thermally [98,99]. For good mechanical force transfer, these implant devices must be able to handle big torques and forces resulting from shear and compression when they are loaded normally. Because of this, it is necessary to make implant materials that are tough, bioactive, safe, hard, corrosion-resistant, aging-resistant, and able to provide good longevity. Biomaterials for implants are usually made of plastics, metals, ceramics, and mixtures of these. The biocompatibility and osseointegration of clay materials with the host tissues are very high because they are similar to the mineral parts of bones.

Bioceramics are ceramics used to make implants and other orthopedic devices meant to fix and replace broken or fatigued body parts, like teeth, bones, joints, and skeletons. [100,101]

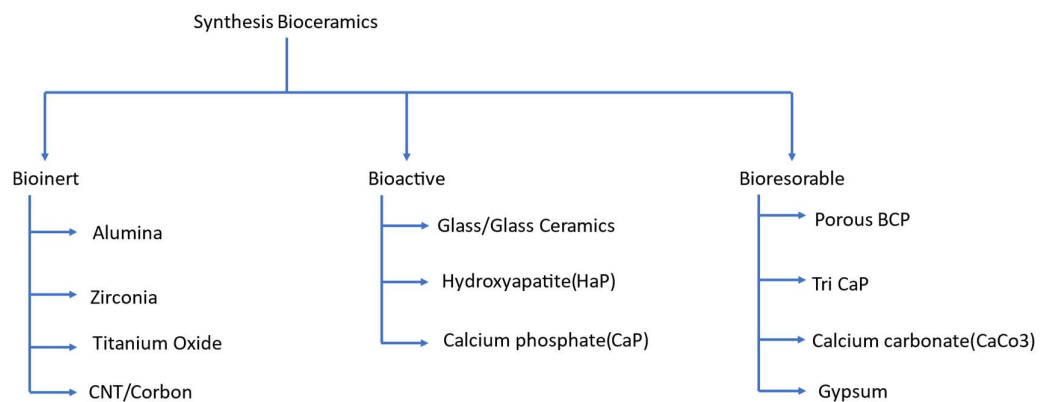


Figure. 2.1 Synthesis of bioceramics.

Traditional Ceramics as Bioceramics

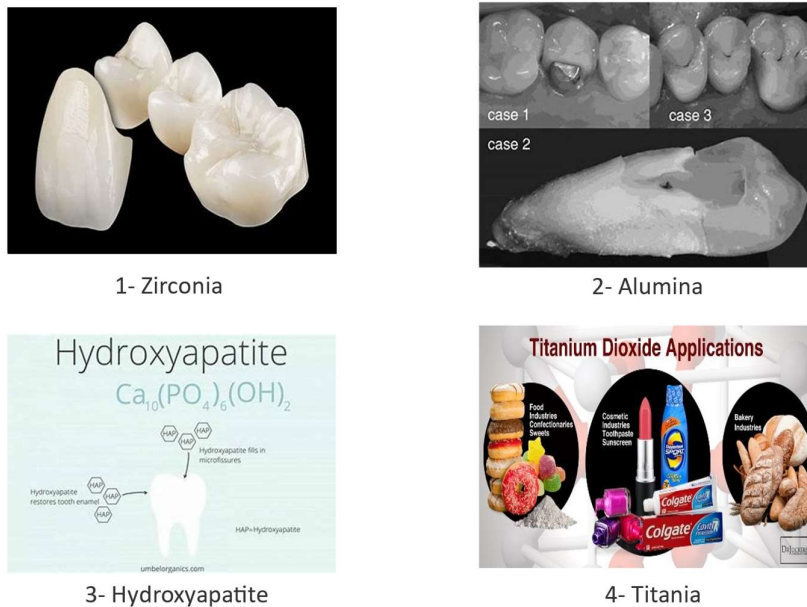


Figure. 2.2 Traditional ceramics as bioceramics [102,103,104]

2.2.2 Bioceramics and Biocomposites: properties and applications

There are two main types of implants: hard and soft tissue implants. Soft tissue implants are used to restore function in the skin, liver, blood vessels, heart tissue, ligaments, kidneys, and cartilage. Because of this, they need to have the appropriate tensile strength, elastic stiffness, and flexural strength. This suggests using polymers and new metals. Hard tissue implant materials are used to fix broken teeth, shoulders, bones, knees, and hips so people don't have to deal with pain and immobility. These materials must be very hard, tough to break, have the right amount of elasticity, resist wear and corrosion, and last long [102]. Different techniques can be used to make bioceramic implants. Which one to use relies on the properties that you want the bioceramic to have. How the bioceramic is made and what it is treated after it is made depends on what it will be used for as a biomaterial product. Understanding the link between the implant and the tissue is also important because it

greatly impacts the patient's life and the implant material's choice, design, and production [103]. Following the reaction between the tissue and the implant, bioceramics are usually put into three groups: (i) bioactive, (ii) bioresorbable, and (iii) bioinert [104]. Most bioinert ceramics are made of alumina (Al_2O_3) and zirconia (ZrO_2), which are also known as biotolerant materials because they don't help bone and implants stick together [105].

Bioceramics have been shown to establish interfacial interactions with human bones and induce modifications in bone composition. Bioceramics, including bioactive glass, Al_2O_3 , ZrO_2 , calcium phosphate, and hydroxyapatite (HAp), are frequently used in modern orthopedic applications due to their exceptional osteo-conductivity, biocompatibility, and resemblance to the inorganic constituents of bone. In addition to their advantageous characteristics, bioceramics have several drawbacks in load-bearing implant applications because of their brittleness, weakness, and poor workability [104]. To address these constraints in bioceramics, researchers have proposed using different fillers as reinforcement and surface remodeling methods. The existing literature has extensive coverage of research that specifically analyzed the incorporation of various fillers and techniques for surface modification and coating to improve the functionality of bioceramic implants. The discipline of bioceramic applications has made substantial progress over the past century. This implies that the clinical application of bioceramics has predominantly concentrated on their mechanical properties, with biocompatibility being a critical requirement for these applications. Consequently, this review aims to comprehensively examine the most recent research on bioceramics and their composites intended for orthopedic applications. The review offers a comprehensive analysis of bioceramic composites' microstructural, mechanical, and biological properties and the effects of additives, reinforcing materials, and processing techniques. Finally, it provides suggestions for future research to further the development of bioceramic implants that are longer-

lasting.

The main reason for hip joint implant failure is the early deterioration of materials with insufficient mechanical properties (such as Young's modulus, hardness, and fracture toughness) when subjected to sudden impacts. The human hip joint, which is the second largest load-bearing joint after the knee, endures repeated stresses during everyday physical activities such as walking, running, exercising, and jumping.[106] Along with these physically demanding activities, there are also common chronic conditions affecting the joints, such as osteoarthritis, avascular necrosis, rheumatoid arthritis, cancer, and femoral neck fractures. Sports injuries and road accidents are other significant causes that may necessitate hip surgery. While the normal bone undergoes a stress level of 40 MPa during routine activities, the hip joint bears three times the body weight. Conversely, during strenuous activity such as jumping and sprinting, the hip joint undergoes stress that is 10 times more than the body weight. The key characteristic of stress in this context is its repetitive and fluctuating nature, which depends on various activities such as climbing, jogging, standing, stretching, and sitting. Therefore, the selected biomaterial must possess high fatigue strength, excellent wear and corrosion resistance, a large elastic modulus, and biocompatibility. Meeting these requirements for the final implant material necessitates substantial advancements and innovations in biological research, materials science, and manufacturing technologies.[107].

Inert ceramics such as Al_2O_3 (alumina) and YSZ (yttria-stabilized zirconia) are commonly used as implant materials for hip joint prostheses due to their bioinert properties, enhanced mechanical performance, and wear resistance. However, Al_2O_3 has limitations, including average strength and fracture toughness, which are key factors contributing to failure in orthopedic applications. Researchers have extensively studied the effects of adding various ceramic stabilizers like yttrium oxide (Y_2O_3), magnesium oxide (MgO),

chromium oxide (Cr_2O_3), and cerium oxide (CeO_2) to Al_2O_3 , ZrO_2 , and silicon nitride (Si_3N_4) matrices to evaluate their effectiveness in biomedical uses. This study focuses on the role of t- ZrO_2 (tetragonal zirconia) and m- ZrO_2 (monoclinic zirconia) particles in improving the fracture toughness of Al_2O_3 - ZrO_2 composites. The analysis revealed that the composites' toughness was enhanced through micro-cracking and transformation toughening, with micro-cracking being the dominant mechanism. Zirconia-toughened alumina (ZTA) ceramics were produced by Homerin et al. using two distinct methods: milling and hot pressing and electrochemical dispersion, slip casting, and pressure-less sintering. The study evaluated the performance of the composites after incorporating ZrO_2 and Y_2O_3 , as well as the impact of processing parameters on the material's mechanical properties. The results demonstrated that adjusting the composition and processing techniques significantly improved the strength and toughness of the final composite. Trabelsi et al. aimed to explore the relationship between the mechanical properties and wear resistance of Al_2O_3 - ZrO_2 composites. Their findings showed that adding ZrO_2 particles improved fracture behavior but reduced wear resistance. This increase in toughness was attributed to a toughening mechanism that counteracted the reduction in hardness, thus mitigating the impact on wear resistance. Orange et al. [110] studied the influence of microstructure and temperature (room and high) on the mechanical properties—such as fracture strength, toughness, and slow crack growth resistance of ZTA ceramics, comparing them to pure Al_2O_3 . Their research confirmed that the combined processing of Al_2O_3 - ZrO_2 composites resulted in superior mechanical properties compared to pure Al_2O_3 , with enhanced wear and thermal fatigue resistance.

Orthopedic implants and devices used in the human body. The idea came from Ref. [101].

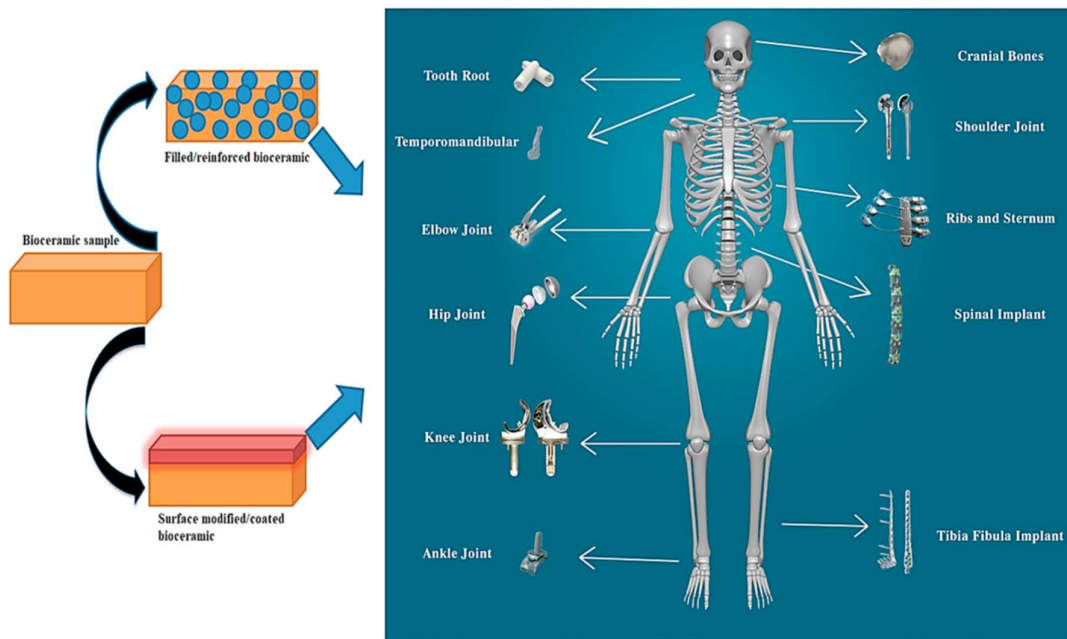


Figure. 2.3 Orthopedic implants and devices used in the human body.

2.2.3 Wastes as Bioceramics

The ceramic industry provides favorable conditions for manufacturing byproducts or wastes. The ceramic industry has conducted various studies in the previous two decades to achieve this goal. Thus, rice husk ash (RHA) [111,112], fly ash (FA) [113], blast furnace slag (BFS) [114], waste marble powder [115], oil production waste [116], paper-processing residues [117], polished tile waste [118], bottom ash [119], water treatment sludge [120], petroleum waste [121], and glass waste [122] are potential materials for ceramics.

Waste materials like FA, RHA, BFS, water treatment sludge, polished tile waste, and red mud can be used as substitutes for natural raw materials in ceramic production. The chemical components of these wastes are listed in Table 1. During ceramic manufacturing, environmental conditions and process parameters significantly influence these wastes' properties and chemical composition.

Table 2.1. The chemical composition of certain wastes.

	SiO ₂	CaO	K ₂ O	Fe ₂ O ₃	MgO	Na ₂ O	TiO ₂	P ₂ O ₅	other	LOI	Al ₂ O ₃
Coal fly ash [123]	55.57	5.12	1.22	6.8	2.97	3.42	-		0.6	2.83	21.47
RHA [124]	91.48	0.36	1.4	0.05	0.32	0.04	0.01		5.24	3.5	-
Water treatment sludge [125]	53.7	14.4	3.2	5	3.6	0.4	0.7		3.2	-	15.8
Fish bones [126]	0.07	55	0.18	0.08	2.13	5.65	-	35.6	.14+	-	0.35
Cattle bone ash [127]	< .01	43.26	< .01	< .01	0.54	< .01	-	44.67	0.09	2.36	<.01
Calcinated bone powder [128]	1.34	52.45	0.3	0.25	1.3	1.6	< .01	36.85	0.84	1.2	0.35
Cow bone char powder [129]	0.89	49.8	-	-	0.78	0.96	-	32.90	1.11	13.13	0.44

Table.2.2 The types of waste materials and other substances used to produce synthetic wollastonite, along with the details of the process.[130-133].

Wastes & other ingredient	Synthesis temperature(°C)	Process	Application
commercial silica and Eggshells	1100	Microwave heating	Biomedical
rice husk Ash and Eggshells	850	Sol-gel	Biomedical
Rice straw ash and calcium nitrate	1450	Sol-gel	Biomedical
Rice husk ash and cement kiln dust	1100	Solid-state reaction	Ceramic basic material

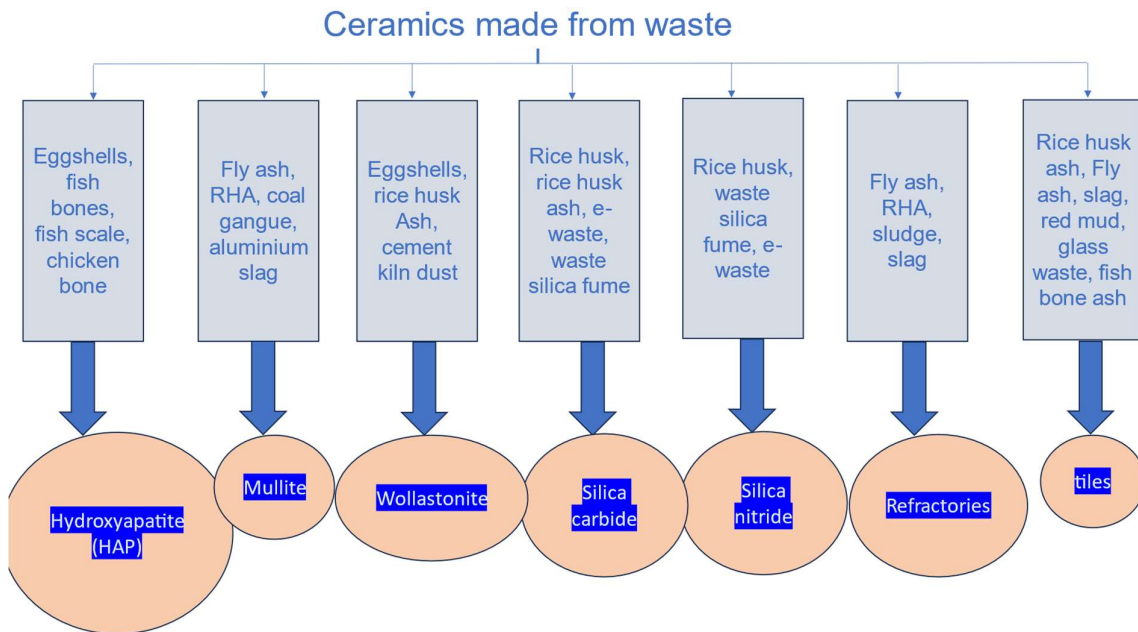


Figure. 2.4 Various wastes are used to derive ceramics [134].

2.2.4 Rice Husk Ash (RHA) in Bioceramics

Researchers have endeavored to utilize RH effectively by directly reinforcing it with other components or utilizing it as ash to harness its high silica content [135]. Rice husk consists of major constituents such as hydrated silicon, cellulose, and lignin [136]. Various methods, encompassing chemical and thermal treatments, have been employed to synthesize silica from RH, with thermal treatment being the preferred method [137,138]. The silica produced from RH necessitates minimal grinding owing to the highly reactive nature of the silica particles, representing a noteworthy advantage of this process.

One popular way to improve the mechanical properties of scaffolds is to add SiO₂ particles. Because it is biocompatible, bioactive, resistant to rust, and an antioxidant, SiO₂ is often used as a reinforcement phase [139]. For tissue engineering, the most important thing about any scaffold construction is that it should be made of biocompatible materials that help bone cells attach and differentiate on its surface [140]. RH is utilized for various purposes during the manufacturing of the scaffold, the most important of which is to serve as a space holder material. Additionally, it is a low-cost source of silica [139]. The circular economy, especially within the bioeconomy framework, has emerged as a promising strategy endorsed by global experts to address the intricate and interconnected issues in this domain [141]. In recent decades, silica derived from Rice Husk (RH), an agricultural byproduct, has garnered attention as a cost-effective source of silica [142]. Both amorphous and crystalline forms of silica can be derived from RH under suitable conditions [143]. Given that millions of tons of rice are produced globally annually, resulting in approximately 200 kg of RH generated per metric ton of rice [144], the proper disposal of RH presents a considerable challenge due to its expansive surface area and carbon content, which can contribute to environmental pollution

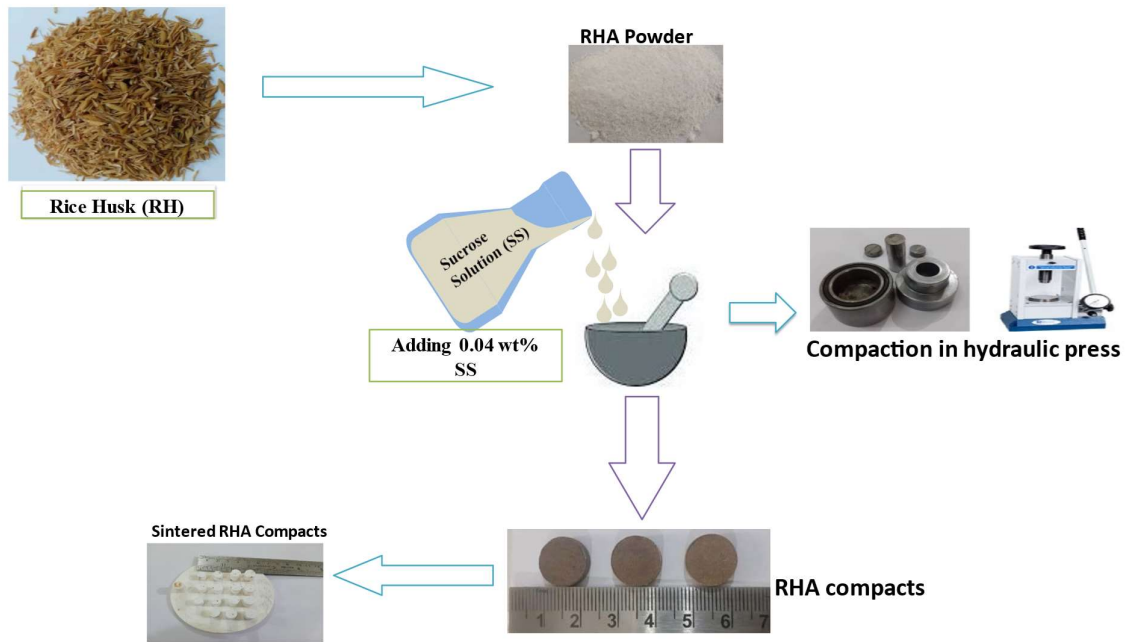


Figure. 2.5 (a) Synthesis of green Rice Husk Ash (RHA) to sintered RHA silica.

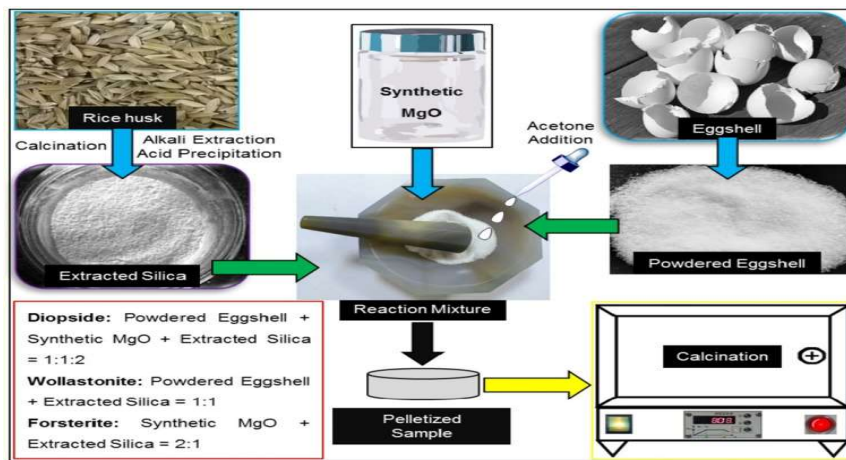


Figure 2.5 (b). Silica and Hydroxyapatite (HAP) through Rice Husk Ash (RHA) and Eggshells

2.2.5 Animal Waste Bone (AWB) in Bioceramics

The objective is to utilize AWBs' environmental and economic costs most effectively before their permanent disposal in landfills. Animal waste bones are classified as soft or hard based on their hardness. For easy use, small animals like fish and birds produce soft bones. Hard bone wastes come from pigs, goats, and cattle. [145]

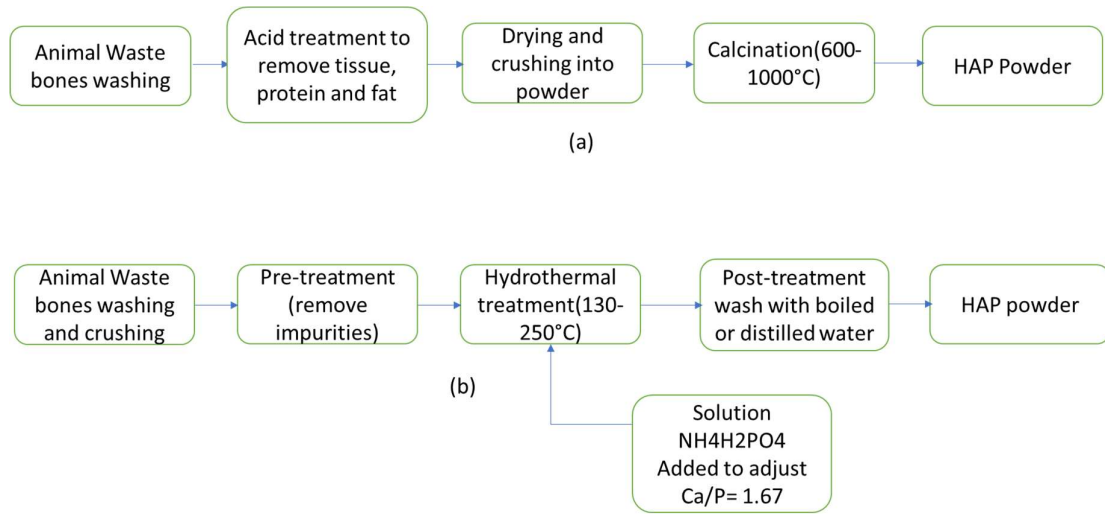
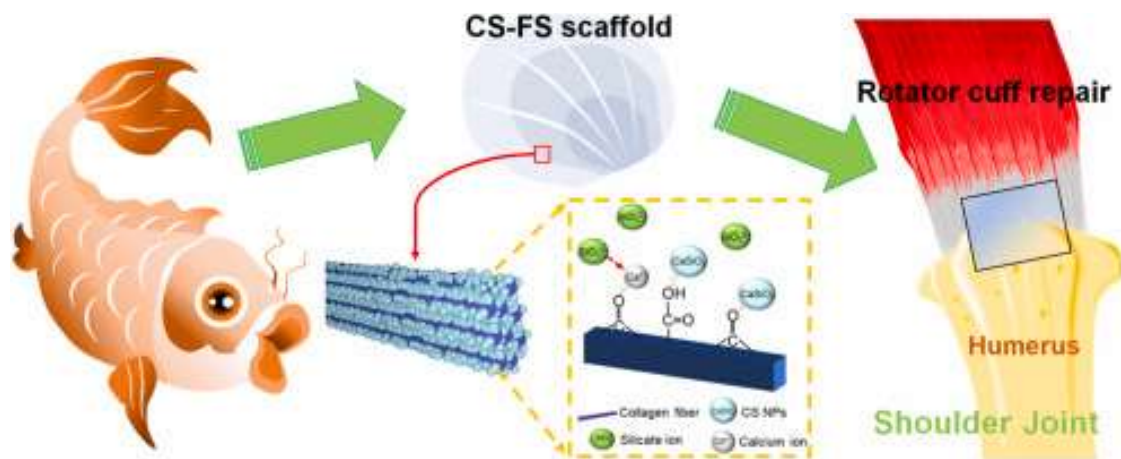


Figure 2.6. A step-by-step guide for making hydroxyapatite powder from used animal bones: (a) the calcination method and (b) the geothermal method.



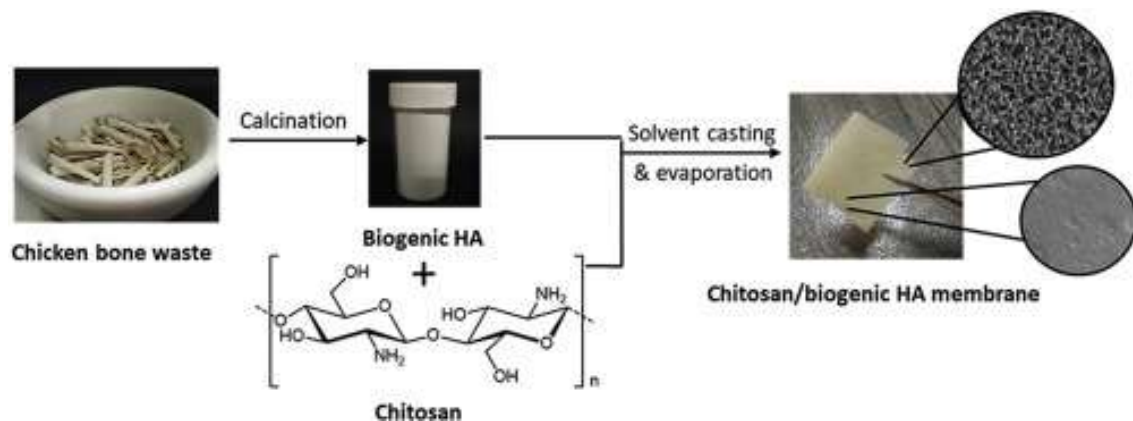


Figure 2.7 (a). Fishbone and chicken bone waste are used as Hydroxyapatite (HAP)

Hydroxyapatite, HAP $[\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2]$, is the major mineralogical component of bones. Based on the Fourier-transform infrared spectroscopy (FTIR) technique, the bone natural apatite contains trace elements (Na^+ , Mg^{2+} , and K^+) and major functional groups, such as hydroxyl (OH), carbonate (CO_3^{2-}), and phosphate (PO_4^{3-}) in its complex molecules.[146,147] But calcium carbonate and carbonate apatite are the two phases of carbonate found in bones. Between 60 and 70 wt% of bones are composed of HA, depending on the animal and the type of bone. Specifically, its biocompatibility allows it to be widely used as a biomaterial in tissue engineering and drug delivery agents.[148,149] Other applications of this material include bioceramics, adsorbents, catalysts, and catalyst support, powder carriers, chromatographic lighting materials, and chemical sensors.[150]

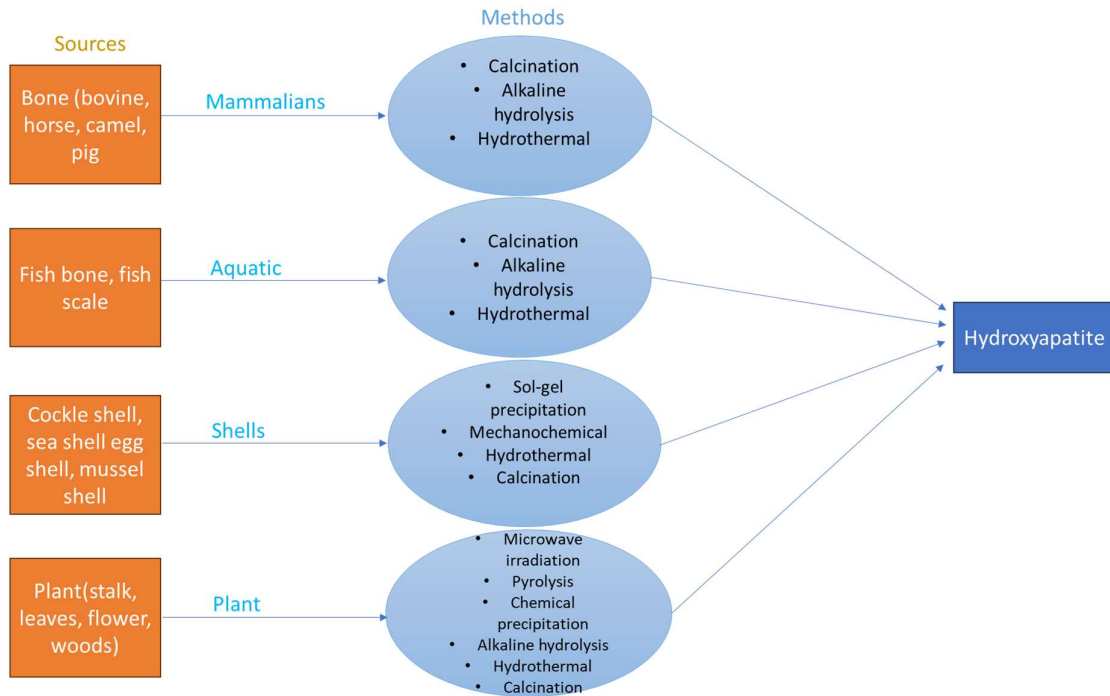


Figure. 2.7 (b) An overview of the processes and sources used to synthesize Hydroxyapatite (Hap). [151].

HAp ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$), a ceramic material similar to human bones and teeth, is extensively explored because of its bioactivity, biocompatibility, osteoconductivity, chemical composition, and thermodynamic stability in bodily fluid [152,153]. Bone augmentation and defect repair can be achieved with HAp without generating inflammation or toxicity [154]. Therefore, HAp has been used in orthopedic, dental, odontology, medication delivery, and non-bioactive implant coating [155]. HAp exhibits strong ion exchange, limited water solubility, and stability in oxidizing and reducing conditions [156]. HAp is an absorbent material in wastewater and soil treatment facilities and adsorbs heavy metals [157]. However, numerous methods for making HAp with customizable physiognomies have been extensively researched. Simpler methods for synthesizing HAp with cheap calcium and minimal energy are worthy of study. Lately, high-calcium wastes like mussel shells [159], oyster shells [158], kina shells [160], abalone shells [161], fish bone [126], fish scale [162], eggshells [164], chicken bone [163], and phosphogypsum

waste [165] have been used to make HAp.

2.2.6 Environmental threat of wastes

The development of science and technology is being pursued to discover the most suitable method of utilizing waste to manufacture products with added value. This is being done considering environmental, ecological, and economic concerns. Considering these considerations, ceramic researchers also consider the possibility of recycling industrial wastes or by-products to manufacture ceramics. As a result, many study articles have been published in recent years. On the other hand, ceramics that are manufactured in factories using waste materials have not yet reached their full maturity. On the other hand, some tile industries have begun integrating waste into their tile production [122].

As waste management has grown in size and value, disposal methods have changed to comply with rules and laws. Waste collection, recycling, reuse, treatment, and disposal are basic waste management methods; however, using recyclables for energy and substances is more sustainable. [166] Innovation, policies, and laws, especially landfill and carbon fees, transform waste management from landfill disposal to waste treatment, reduction, recycling, reuse, and energy and resource recovery. Underusing AWBs increases disposal costs and environmental pollution.

However, these numbers are eager due to the fact that numerous conditioning features, such as the compatibility between natural raw materials and wastes, the characteristics of the final product, the continuous availability of the product, the costs of transportation, the constant chemical compositions, and the pre-treatments of wastes, limit the applications. hence, a more in-depth examination is required in order to facilitate the transfer of technology from the academic sector to the industrial sector for the purpose of

commercializing ceramics that are created from waste. The transfer of technology is becoming a challenge from various viewpoints, including those pertaining to ethics, knowledge, and the potential for unsustainable outcomes. Because of this, there is a need for further support to be given to industrial manufacturing in order to recycle waste. The creation of laws and regulations by the government is another potential way in which it can contribute to a rise in interest. Production that is sustainable not only benefits the ceramics industry but also protects both the environment and society from the negative effects of pollution.

The generation of toxic and hazardous wastes from various sources is on the rise, contributing to environmental pollution and disposal challenges. By repurposing and upgrading these wastes as substitutes for primary natural resources, it is possible to reduce resource extraction (conserving resources), lower processing energy consumption (making it more cost-effective), and minimize pollutant emissions (enhancing public health and safety). Since ceramics rely heavily on natural raw materials, recycling waste is particularly beneficial. Even small-scale waste disposal in high-volume ceramics production can lead to substantial waste absorption. Over the past two decades, significant research has been conducted into waste utilization in ceramics, although commercialization remains limited. Some wastes have great potential to substitute natural resources in ceramics manufacturing.

2.3 Literature Review

2.3.1 Bioceramics and Biocomposites

Table. 2.3 Properties and Application of Bioceramics [30], [101,102], [167]

Types of Bioceramics	Bioceramics	Properties				Applications
		Compressive Strength (MPa)	Hardness (HV)	Density (g/cm ³)	Fracture Toughness (MPa.m ^{1/2})	
Bioinert	Al ₂ O ₃	4000	2000	3.97	4-5	Femoral head implants
	ZrO ₂	2000	1400	6.53	10	Endosseous implants
	Y-TZP	3000-4000	1200	6.05	6-12	Replacement of diseased or fractured
	ZTA	4000	1800	4.4	6-10	hip, shoulder, elbow, wrist, elbow, Part of knee, tooth etc
	ZrO ₂ -3Y-TZP	2200	1200	6.06	8	
	ATZ	2500	1400	5.4	5	
	Bioactive	HAp	600	350	3.1	0.6-1
glasses		40-60	600	2.6	0.5-1	bone defect fillers, Bone grafts
Biodegradable	CaP	20-900		3.05	1	Bone grafts, Replacement of the surrounding tissue

2.3.2 Zirconia-based Biocomposites

Zirconia implants osseointegrate, as do titanium implants. Zirconia creates fewer phlogistic reactions in tissue and less bacterial colonization than titanium. However, particles from the degradation of zirconia at low temperatures (LTD) or from the manufacturing process can be released, promoting an immune localized inflammatory reaction.[168] Aging or low-temperature degradation is the spontaneous, slow transformation of the metastable tetragonal phase to the more stable monoclinic phase in the absence of any mechanical stress, occurring over time at low temperatures. It is exacerbated in the presence of water, steam, or fluids. LTD is based on the same mechanism as PTT (Phase Transformation Toughening); that is, it requires the presence of t-ZrO₂ grains in thermodynamic metastability, which can undergo t-m transformation [169].

HAp-ZrO₂ composites were synthesized by dispersing the materials in an aqueous polyacrylic acid and glutamic acid solution. After achieving stable slurries, the green samples with higher density were processed using slip casting. To further enhance density, pressure-less sintering was performed on the green samples at 1450°C for 2 hours. The study results indicated that the proposed method of colloidal processing combined with pressure-less sintering is highly effective for producing HAp-ZrO₂ composites with excellent mechanical and biological properties, making them suitable for orthopedic applications. In a similar study conducted by Sung et al. [170] one year later, They synthesized a homogeneous blend of HAp and YSZ nano-ceramics through chemical coprecipitation, followed by drying, calcination and hot pressing at 1100°C for 1 hour under a vacuum atmosphere. The addition of YSZ to the HAp matrix significantly improved the mechanical properties of the resulting nanocomposites, particularly in terms of fracture toughness (2.1 MPa·m) and flexural strength (155 MPa). The successful creation of HAp/YSZ nanocomposites highlights their potential for load-bearing orthopedic

applications. This is evident from the enhanced properties of the 25 wt% YSZ nanocomposites, including flexural strength (155 ± 16 MPa), elastic modulus (135 ± 15 GPa), hardness (7.2 ± 0.6 GPa), and fracture toughness (2.1 ± 0.2 MPa·m). Furthermore, Gain et al. [54] Porous HAp-ZrO₂ nanocomposites were produced by incorporating polymethyl methacrylate powders as a pore-forming agent during manufacturing. The polymethyl methacrylate powders helped create a random distribution of pores in the final composite. The HAp-ZrO₂ nanocomposites exhibited superior performance compared to monolithic HAp. The compressive strength of monolithic HAp was measured at 31.4 MPa, while the HAp-ZrO₂ nanocomposites achieved a compressive strength of 34.9 MPa. The elastic moduli were found to be 19.6 GPa for HAp and 23.4 GPa for HAp-ZrO₂, both comparable to human cortical bone's properties.

Dudnik and colleagues [171] attempted to A nanocrystalline powder composed of ZrO₂-rich ZrO₂-Y₂O₃-CeO₂-Al₂O₃ was synthesized. The fracture toughness values of the powders, sintered at temperatures ranging from 400°C to 1300°C, varied between 6.4 and 16.8 MPa·m, depending on the processing conditions. A separate group of researchers [57,172] aimed to assess The fracture toughness, strength, and slow crack propagation properties of a ceria-stabilized ZrO₂-Al₂O₃ nanocomposite were evaluated for potential biomedical applications. The research results showed that the threshold values (4.5 MPa·m) and fracture toughness (8.8 MPa·m) were superior to those of currently used ceramics, such as Al₂O₃ and Y-TZP. The material demonstrated a cyclic threshold value of 4.5 MPa·m, exceeding the performance of established biomedical-grade materials like Al₂O₃ and ZrO₂. Arin and colleagues [58][173] synthesised and characterised ZrO₂ ceramics and composites with different concentrations of Y₂O₃, CeO₂ (2-3 mol.%), and Al₂O₃. They also produced TiN-reinforced ZrO₂ composites with a material content ranging from 35 to 60 vol%.

The main factor contributing to transformation toughening was the toughening mechanism. The hardness and strength decreased because to the larger particle size of TiN and the concurrent increase in TiN concentration. The toughness decreased due to lower transformation toughening during the transition from t→m (tetragonal to monoclinic) ZrO₂ phase structure. However, the elastic modulus directly correlated with the increasing titanium nitride (TiN) concentration. The objective of Mazzocchi et al.[174] was to examine three distinct series of Si₃N₄-based ceramic materials for orthopedic implants. This investigation focused on these materials' processing, microstructure, mechanical properties, and cytotoxicity. The findings indicated that the use of appropriate sintering aids or secondary phases in the production of these ceramics would result in improved mechanical characteristics and the absence of intergranular phases after development. This indicates efficient bonding between the elements, which in turn ensures effective stress transfer. Nath et al. [174] examined the obtained mechanical, microstructural, and tribological properties of ZrO₂ ceramics doped with calcium and sintered using microwave technology. Analysis revealed a theoretical density of 97.5% for 8 mol.% calcium-doped ZrO₂ and 91.6% for calcium-doped ZrO₂. The hardness of the material rose as the sintering temperature increased. The hardness values achieved were 10 GPa for 8 mol.% calcium-doped ZrO₂ and 9 GPa for 16 mol.% calcium-doped ZrO₂. 8 mol.% calcium-doped ZrO₂ ceramics had the highest toughness value (6 MPa–m) compared to other ceramics.

Bartuli et al. [175] aimed to produce compact and cellular Y-TZP ceramics using the sol-gel casting method and analyze the ensuing mechanical properties and characterization. Post-gel casting sintering of Y-TZP ceramic powders yielded an elastic modulus of 170 GPa and a modulus of rupture of 400 MPa. The overall quantity of pores produced directly impacts the mechanical characteristics of the manufactured ceramics. Rascon and colleagues undertook an assessment of the mechanical characteristics of

ceramic composites based on alumina-toughened zirconia (ATZ) and zirconium titanate (ZTA) by altering the amounts of aluminum oxide (Al_2O_3) and zirconium dioxide (ZrO_2). The primary objective of this work was to evaluate the impact of sintering on the hardness and fracture toughness of ATZ and ZTA. The obtained relative density was around 94%, the achieved hardness ranged from 9.5 to 21.9 GPa, and the fracture toughness was measured to be 3.6 $\text{MPa}\cdot\text{m}$. In contrast to pure oxides, our work demonstrated that ATZ and ZTA composites can be considered more promising materials for implant manufacturing. Bernal et al. [176] investigated the mechanical characteristics of Al_2O_3 ceramics within the context of sinter and sinter-HIP processes. The samples generated by the hot isostatic pressing (HIP) sintering technique obtained relative densities over 98%, hardness of 19 GPa, and fracture toughness of 5.2 $\text{MPa}\cdot\text{m}$. A further study by Oungkulsolmongkol et al. [177] aimed to investigate the impact of integrating ZrO_2 and SrO (strontium oxide) additives on the mechanical properties (hardness and toughness) and features of composites based on Al_2O_3 . The results indicated that the combined inclusion of SrO and ZrO_2 in the Al_2O_3 matrix did not improve hardness. However, it did provide increased fracture toughness compared to SrO- Al_2O_3 and ZrO_2 - Al_2O_3 compositions. Meanwhile, Maiti and Sil [178] aimed to establish a correlation between the fracture toughness properties and the structure of Al_2O_3 ceramics produced by the sintering technique. A primary aim of the study was to examine the impact of sintering temperature (1500, 1600, and 1700 °C) and soaking time on the fracture toughness of Al_2O_3 ceramics. The samples sintered at 1700°C exhibited lower fracture toughness values of 4.6 and 5.0 $\text{MPa}\cdot\text{m}$, with a predominant transgranular fracture-type mode. Crack deflection is the dominating phenomenon responsible for the enhanced fracture toughness in samples sintered at temperatures of 1500 and 1600 °C. A study conducted by Echeberria et al. [179] examined the microstructure and fracture toughness values of ZTA nanocomposites

reinforced with sintered and hot isostatic pressed multi-wall carbon nanotubes (MWCNT). In both cases, including MWCNT, there was a decrease in hardness metrics.

2.3.3 Alumina and Zirconia-Based Biocomposites

Researchers have developed many composites in which zirconia is used as the structural framework, and alumina is included in the framework. In order to enhance the mechanical properties of the tetragonal zirconia matrix, the purpose of this integration is to enhance the resistance of low dynamic strength (LTD) and to make greater use of the exceptional attributes that these ceramics possess [180-185]. On the other hand, alumina must be added to the matrix in order to create a hard structure that locks the zirconia particles in place. It is possible for tetragonal zirconia grains to change from the tetragonal phase to the monoclinic phase as they cool down from the sintering temperature. This way of looking at it, alumina, keeps the zirconia grains in a metastable state, which stops them from fully changing into the monoclinic phase. The noted rise in the hardness of the ceramic material is due to the retention of the tetragonal phase [184,186,187].

The pros and cons of both Y-TZP and alumina can be found in the literature. Since both ceramics have some problems, combining them to make alumina-zirconia composites seems like the best way to get around those problems and make the most of their individual strengths. This is why many experts have been studying it so much. Because of this, this study shows what we know about alumina-zirconia composites now. The general information about alumina/zirconia ceramics is summed up after this introduction, focusing on ATZ ceramics. It also emphasizes the important factors that affect these materials and their qualities, and how different sintering conditions change these properties. This review also summarizes what almost all papers written on ATZ in the last 30 years have found.

Comparing ATZ and ZTA composites, Sequeira et al. [180] found that ZTA

samples were harder than ATZ samples. This difference was due to the increased alumina content in the ZTA composite. minor alumina concentration fluctuations (80%-90% in ZTA samples) did not significantly impact hardness. In contrast, ATZ samples showed a modest drop in hardness as alumina levels decreased from 20 to 10 wt%.

The catastrophic failure of the zirconia and alumina femoral heads sparked discussion among medical professionals, researchers, and industrialists. Zirconia ceramic was unsuccessful as a result of the low-temperature degradation-induced transition from a tetragonal phase to a monoclinic phase, which was followed by both macro and microcracks [188], whereas alumina ceramic failed because of its low fracture toughness. Consequently, a lot of time and energy have gone into creating composite ceramics that use zirconia and alumina. These composites aim to improve slow fracture growth resistance by combining zirconia's strength and toughness with alumina's high hardness and stiffness [182,189].

Alumina Toughened Zirconia, frequently known as ATZ, is a composite material that exhibits high toughness and strength. It is composed of a matrix of tetragonal zirconia that is reinforced with alumina particles[180,190,191]. Alumina, which is a minor phase, contributes to an increase in the overall stiffness of the matrix because its elastic modulus is twice as high as that of zirconia. Tetragonal zirconia is the principal phase that has been incorporated into this composite. Incorporating secondary hard particles, such as alumina, resulted in the production of fracture deflection, also known as micro-crack toughening, which contributed to an increase in the overall toughness of the composites[192].

Compared to the other two forms of alumina-zirconia composites, the ZTA composite has been the subject of significant research. It has been noticed time and time again that the transition toughening of zirconia in this composite results in an increase in

fracture toughness and strength. This is the reason for this. Various processing methods and formulations have been evaluated for ZTA to improve the properties of the composites. These include adjusting the quantities of zirconia, adjusting the sintering conditions, and incorporating sintering additives [180,192]. On the other hand, the ATZ composite has not been as successful in attracting attention as ZTA. A limited number of studies have been conducted to improve the properties of ATZ by experimenting with different sintering temperatures and methods, according to the review of the current literature, which suggests that relatively few of these studies have been carried out.

2.3.4 Alumina-toughened zirconia (ATZ) based composites

ATZ materials have a bending strength of up to 1900 MPa. Including alumina initially generates tensile residual cooling stress in the zirconia matrix, promoting phase change[67]. Adding ~2.5–3 mol% of yttria stabilizes the tetragonal phase, creating Y-TZP, a strong and robust material. Adding alumina to TZP has numerous effects. Alumina and zirconia are immiscible, although TEM shows that Y-TZP's grain border contains alumina. Alumina buildup in the first 5–10 monolayers stabilized zirconia and increases LTD resistance[193], [194] A further increase in the alumina fraction that is greater than 10 vol%, results in the formation of alumina toughened zirconia, which is a material that possesses exceptional strength when the addition of alumina is present [195,196] As alumina addition also enhances wear resistance [194]

There have been many studies on different types of ATZ compositions, with alumina making up to 49.9 wt% of the composite. However, only a few of these compositions have become commercially viable, large-scale products. As a result, the most successful composition was found to be 20 wt% alumina and 80 wt% zirconia (stabilized with 3mol% Y₂O₃).

To get a ceramic with no flaws, consider the sintering temperature, the sintering method, the sintering holding time, and the zirconia powder supplier. Following this, different studies showed that adding alumina to zirconia had various effects. The researchers believe that this is because they used different starting materials and processing methods, which changed the end microstructure [188,197]

On the other hand, improving mechanical and physical qualities depends on the size of the grains in the ceramic composite. So, using nanoparticles, often found in ceramics, has been a big part of improving the mechanical features. Experiments in the lab showed that the grain size is affected by many things, including the powders' initial size, the amount of pressure used during the pressing process, the sintering temperature, the dwell time, and the sintering method [198]. So, for the ATZ composite to have the best physical and mechanical qualities, the alumina nanoparticles need to be spread out among the zirconia matrix. Adding nano alumina to the zirconia matrix can stop matrix cracks and make the material more complicated and less likely to break [181].

Nevarez-Rascon et al. [188] used powder pressing to create alumina-Y-TZP composites in various quantities. The alumina powder was highly pure (>99%) and contained 0.025 wt% MgO as a grain development inhibitor. The zirconia powder was high-purity, commercially available 3 mol% Y-TZP made using co-precipitation. Researchers used a sintering process to include green bodies into a ZrO₂ and Al₂O₃ powder bed. Sintering began with a temperature increase to 1200°C at 10°C/min, followed by a final step at 1500°C at 6°C/min. After 2 hours at this temperature, the samples were cooled to room temperature. Results show that the ATZ composite with 20 wt% Al₂O₃ attained a relative density of 97%. Comparatively, samples containing 30 and 40 wt% Al₂O₃ had 96.3% and 88.6% relative densities.

Research indicates that increasing alumina content decreases relative density. The relative density difference is due to the different densities of zirconia and alumina particles. Based on particle densities and diameters, ZrO₂ particles are 5.13 times heavier than Al₂O₃ particles. When the Al₂O₃ level approaches 40 wt%, the volume occupied by both Al₂O₃ and ZrO₂ particles equalizes. When ZrO₂ and Al₂O₃ weights are balanced, a significant rise in density occurs. This behavior is often seen in spherical particle mixtures with a particle size ratio nearing unit [188].

The study by Boniecki et al.[199] examined the characteristics of ATZ with alumina content of 20 wt% utilizing the hot press (HP) process. Sintering occurred at 1400 °C for 1 hour in an inert atmosphere at 30 MPa pressure. The researchers found that applying pressure during sintering achieved a high level of densification in ATZ material, reaching 99.6% of theoretical density. This shows that pressure-assisted sintering can greatly improve the density and quality of ATZ composites.

In their study, Gil-Flores et al. [183] examined the density of ATZ nanocomposites made using pressure-less rapid microwave sintering. The study found that the sintering method produced high-density ATZ nanocomposites. Successful densification was achieved as the samples reached a density close to the theoretical value. The study examined the impact of sintering temperature and duration on nanocomposites' density. Higher sintering temperatures resulted in higher densities due to improved particle rearrangement and bonding. Extended sintering periods led to denser samples due to increased particle diffusion and compaction. The density differences were caused by changes in microstructure, including grain development and pore removal.

Studies confirm that sintering temperature affects the density of alumina-toughened zirconia (ATZ) composites. Higher sintering temperatures typically result in

higher ATZ composite densities. However, the impact of sintering time on ATZ composite density is unclear, with some research indicating higher densities and others showing no meaningful effect.

Boniecki et al. [200] found that 20 wt% Al₂O₃ was most effective in refining the microstructure of alumina-doped Y-TZP composites. The ATZ (20 wt% + 80 wt% Y-TZP) had reduced tetragonal and alumina grain sizes (0.22 ± 0.06 and 0.25 ± 0.07 μm , respectively) compared to the Y-TZP and alumina monolithic (0.41 ± 0.23 and 0.78 ± 0.21 μm , respectively). Kirsten et al. [201] examined subcritical crack growth in oxide ceramics, including ATZ (20 wt% Al₂O₃ + 80 wt% Y-TZP), and found that zirconia pinned alumina grain growth, confirming the reduction in grain size.

Freitas et al. [202] created an alumina toughened zirconia (ATZ) composite with 80 wt% TZ-3YSB/20 wt% Al₂O₃ composition through mechanical alloying in a planetary ball mill under an argon environment. Researchers examined the tetragonal phase stability of the composite using X-ray diffraction analysis. The powders initially had monoclinic phases but underwent a complete change into tetragonal phases after sintering. The authors attribute this result to Y₂O₃ oxide, stabilizing the tetragonal ZrO₂ phase at ambient temperature. In their investigation, Sequeira et al. [180] created alumina-toughened zirconia (ATZ) composites with different quantities of Al₂O₃ (10-20 wt%) and 3YSZ (90-80%) via powder mixing. The composite powders underwent a cold isostatic procedure and were sintered at 1400 °C for 3 hours in a laboratory furnace. X-ray diffraction (XRD) analysis was used to assess the phase composition of sintered composites. According to the study, the composites achieved a tetragonal matrix after sintering. This implies good stabilization of the tetragonal phase in ATZ composites. In line with Zhang et al. [203], all ATZ composites with varying quantities of Al₂O₃ (5, 10, 15 wt%) sintered at 1500 °C for 5 h showed a completely tetragonal phase. Another study found that sintering duration

impacts the change of ATZ composites from monoclinic to tetragonal. In their study, Bocanegra-Bernal et al. [204] sintered ATZ composites with TZ-3YE, 3 mol% Y_2O_3 stabilization, and varying quantities of Al_2O_3 (5, 15, and 30 wt%) in a graphite powder bed. Sintering occurred at 1520 °C for 0-10 h at a heating rate of 10 °C/min in air. Using XRD Rietveld analysis, the monoclinic phase increased with longer sintering times. At a holding duration of 0 h, no monoclinic phase was seen in the ATZ samples. The authors suggested that a protective alumina layer during sintering in a graphite powder bed environment prevented the breakdown of the tetragonal zirconia into the monoclinic phase.

Several studies have found monoclinic and/or cubic phases in ATZ composites during sintering. Boniecki et al. [205] researched tetragonal phase stability in ATZ composites with TZ-3YE and Al_2O_3 content from 20 to 80 wt%. All polished samples after sintering showed a monoclinic phase, regardless of alumina addition, as validated by XRD examination. Researchers found that the transformability of the tetragonal phase diminishes with higher alumina concentration, as seen by the low monoclinic phase composition on the composite fracture surface, supporting Kern and Gadow's [194] findings on ATZ. As Al_2O_3 content grew in the composite, Y-TZP matrix transformability decreased. The formation of ATZ was achieved by hot-pressing 2.5Y-TZP with Al_2O_3 at 1400 and 1500 °C/1 h under 50 MPa pressure. The scientists showed that alumina added compressive stresses to the composite matrix, counteracting the tetragonal granules' transition toughening. Therefore, tetragonal grains became more stable and less sensitive to external stress [206].

A study by Meena et al. [207] examined the tetragonal phase stability of an ATZ composite (10 vol% Al_2O_3 + 90 vol% 3YSZ) using conventional (CS) and microwave (MW) methods. Samples were sintered at 1600 °C for 6 h with CS and 1 h with MW. Compared to the CS, the MW-sintered ATZ composite dramatically reduced the

monoclinic phase in the matrix after sintering. The scientists found that the CS technique's increased sintering duration reduced composite tetragonal phase stability.

In alumina-toughened zirconia (ATZ) composites, alumina particles reinforce and increase hardness. Alumina prevents the tetragonal phase from becoming monoclinic by creating a stress field that stabilises it. Consequently, ATZ composites show better crack propagation resistance than pure zirconia. Numerous research have shown that several elements affect zirconia's stability. The impact of these parameters varies by production process and sample sintering. In some cases, increasing the alumina content in a composite can improve the stability of zirconia's tetragonal phase, while surpassing this limit may have the reverse effect. Numerous studies suggest that alumina content of 20 wt% is ideal for obtaining tetragonal phase stabilisation in ATZ composites. The size of alumina particles affects tetragonal phase stabilization, with smaller particles being more efficient than bigger ones. The stability of the tetragonal phase depends on the distribution of alumina particles, which must be equally distributed throughout the composite for optimal performance. In contrast, processing factors affect tetragonal phase stability. Sintering at high temperatures can destabilize the tetragonal phase. Microwave sintering reduces monoclinic zirconia compared to conventional sintering, which affects tetragonal phase stabilization. Research indicates an inverse link between sintering holding time and tetragonal phase stability, with duration decreasing stability. In summary, zirconia's tetragonal phase stability in ATZ composites depends on various parameters. Understanding these parameters is crucial for optimizing ATZ composite characteristics and performance across various applications. Multiple parameters are involved, such as sintered body density and grain size. The density of sintered bodies significantly impacts ceramic material hardness. Denser structures are more compact and tightly packed, leading to increased plastic deformation resistance and hardness. To optimize the hardness of ATZ

composites, high density during sintering is a desirable objective [206,208].

Meena et al. [207] compared microwave and conventional sintered ATZ samples for hardness change. The study examined the impact of adding 10 vol% Al_2O_3 as a minor phase in the 3Y-TSZ matrix during microwave sintering at 1600 °C/1 h. Results indicate a considerable increase in microwave-sintered ATZ hardness, reaching 18.7 GPa. The standard sintered ATZ composite, sintered at 1600 °C/6 h, had a hardness of 17.3 GPa. The authors attributed the hardness improvement to inhibiting grain development and increasing density using microwave sintering. The scientists discovered that microwave-sintered ATZ had a grain size of 0.39 μm and a relative density of 99.1%. Conventional sintered ATZ has a grain size of 0.51 μm and a relative density of 98.6%.

It is important to note that the alumina content present within ATZ composites is directly related to their hardness. Zirconia is a substance that is harder than alumina, which means that alumina has an inherent influence on the composite's hardness. Because of this, an increase in the amount of alumina in the composite material results in a drop in the composite's hardness because zirconia's contribution is proportionally reduced.

A material's fracture toughness is a crucial mechanical parameter that affects its resistance to crack propagation and stress-induced fracture. Alumina-toughened zirconia composites are popular for their superior fracture toughness compared to pure zirconia. The fracture toughness of ATZ is affected by parameters such as composition, sintering temperature, processing procedures, transformation toughening, and crack deflection mechanism [206,209].

In another work, Zhang et al. examined the impact of adding alumina (Al_2O_3) to Y-TZP on fracture toughness. Researchers examined composites with up to 50% alumina content. Fracture toughness increased with alumina concentration, reaching 6.9 $\text{MPa}\sqrt{\text{m}}$

at 50 vol% Al₂O₃. The scientists ascribed the increased fracture toughness to multiple reasons. Initial transformation toughening of the tetragonal zirconia matrix significantly enhanced composite toughness. The toughening mechanism involves stress-induced transition of tetragonal zirconia to monoclinic, resulting in crack closure and energy dissipation, preventing crack development. Second, the composite fracture mode was mostly transgranular, indicating crack propagation within grains rather than along grain borders. This transgranular fracturing behavior enhanced fracture toughness [210].

Boniecki et al. [200] studied composites with 20-80% Al₂O₃ and 3Y-TZP (3 mol%) in varied weight percentages. The composites were manufactured using planetary ball milling and sintered at 1480 °C for 2 hours in an air environment. The study aimed to examine how zirconia and alumina content affect composites' subcritical fracture development behavior.

Authors discovered that increasing zirconia percentage significantly enhanced composites' fracture toughness (K_{Ic}), bending strength, and subcritical crack resistance. This was due to the transformability of the tetragonal zirconia matrix. XRD analysis of fracture surfaces showed that the 20 wt% Al₂O₃ + 80 wt% Y-TZP composite had a similar monoclinic phase (20.5%) to the monolithic YTZP (25.2%), which had the highest toughness among the tested samples. The monoclinic phase decreases when alumina content exceeds 20 wt%, leading to lower fracture toughness (K_{Ic}) values. Fracture toughness was correlated with lower monoclinic phase levels on these composites' fracture surfaces. Researchers found that combining zirconia and alumina in the composite significantly affects subcritical crack development behavior. A sufficient proportion of zirconia facilitates the production of a monoclinic phase, enhancing composite toughness. Excessive alumina content can limit monoclinic phase development and reduce fracture toughness [194].

2.3.5 Sintering of green sample

Sintering is a heating process that joins particles that touch each other into a solid. It is very important to know the particle size and how they are packed in the green body because they affect how fast it sinters and how dense it becomes. Sintering is a key step in forming advanced ceramics because it changes the end microstructure of the sintered body, which in turn changes its mechanical, optical, biological, and electrical properties [211]. Nevertheless, it is very important to ensure that the proper steps are taken when making the powder and adding additives or binders because getting a good ceramic product depends a lot on controlling the ceramic microstructure by changing process parameters during the sintering stage. So, picking the correct sintering method, holding time, heating profile, and other factors is important for making the sintering process work best. The process of sintering can be broken down into two main groups:

1) Sintering in a solid state. 2) Sintering in a liquid phase.

When two solid particles are heated to a high enough temperature (below the melting point of the particles), solid-state sintering (SSS) takes place without any liquid phase present. In the early stages of SSS, Johnson says that two particles next to each other can join by vapor transport, lattice-diffusion, surface-diffusion, grain boundary-diffusion, and plastic deformation via dislocation migration. It is thought that surface diffusion, lattice diffusion, and vapor transfer of matter from the particle surface to the neck area cause the neck to form and grow without getting denser. It gets denser through lattice diffusion and grain boundary diffusion from the grain boundary between particles to the neck region. It also gets denser through plastic deformation caused by dislocation motion in response to the capillary forces from the curved neck surface. Matter will fill the space and bond the particles together during these processes. Regarding SSS, diffusion won't happen until the temperature drops below a certain level, and it takes enough time for diffusion to complete.

2.3.6 Alumina toughened zirconia Applications

The mechanical properties of alumina-toughened zirconia (ATZ) ceramic composites are known for their remarkable versatility, including high strength, fracture toughness, elasticity, hardness, and wear resistance. These characteristics render ATZ composites highly suitable for various applications in several sectors. The exceptional biocompatibility and mechanical performance of ATZ ceramics make them highly promising for potential biomedical applications. The potential of these ceramics to improve patient outcomes and stimulate progress in medical technology is considerable. Ongoing research efforts are actively investigating new uses for ATZ ceramics and refining their characteristics to meet the changing requirements of the medical sector [212,213]. The exceptional characteristics of ATZ ensure that these ceramics are well-suited for a wide variety of biomedical applications.

In dentistry, ATZ ceramics are essential to developing long-lasting and biocompatible dental implants, such as crowns and bridges. Their exceptional strength, resilience to fracture, and resistance to wear guarantee long-term stability, providing a dependable tooth replacement option and enhancing patient outcomes in dental applications. Dental crowns and bridges made of ATZ ceramics offer long-lasting, aesthetically beautiful ways to replace lost or damaged teeth. The restorations will last many years because of the material's strength and resistance to wear. ATZ ceramics can also be created to imitate real teeth' color, making them indiscernible [200,209,214].

ATZ ceramics are widely used in orthopedic applications, such as hip and knee replacements, due to their biocompatibility and resistance to mechanical stress. These implants are favored for orthopedic surgery due to their better wear resistance and strength, resulting in durability and reduced risk of adverse responses. Alumina toughened zirconia ceramics are being investigated for use in artificial joints. This research aims to

use their unique features to reduce wear on articulating surfaces, improving joint replacement function and longevity [180,182].

Another use of ATZ ceramics is as bioceramic coatings on metal implants, demonstrating their flexibility. Enhancing biocompatibility reduces implant rejection and unfavorable tissue responses, enhancing the effectiveness of implant-based therapies. Expanding into tissue engineering, ATZ ceramics demonstrate potential as bone scaffolds. Their biocompatibility and strong mechanical qualities promote bone formation and regeneration, promising advancements in tissue engineering [192,215].

The aircraft sector uses ATZ ceramics for structural components such as turbine blades, engine parts, and heat shields. In aerospace applications, their excellent thermal stability and mechanical strength are crucial for reliability under harsh conditions [207,216]. Excellent electrical insulation qualities make ATZ ceramics useful insulators in high-temperature and high-voltage situations. These components are valuable in different electrical and electronic applications. Industries requiring high wear resistance often use ATZ ceramics for pump parts, nozzles, and valves. The ceramics' ability to reduce wear extends equipment service life and lowers maintenance costs [198,216].

ATZ ceramics are commonly used in structural applications across sectors due to their remarkable mechanical qualities and thermal stability. ATZ ceramics are used in production as inserts and drills for precise machining. Strong hardness and wear resistance enable the successful shaping and cutting of materials like metal alloys. Ceramics are used as bearing components in industries where steel bearings may not be sufficient. Low friction coefficient and high hardness make them appropriate for heavy loads and corrosive environments, improving bearing reliability and performance [198,202,217].

Overall, alumina-toughened zirconia ceramics are very useful for many different structures because they are strong, tough, resistant to wear, and stable at high temperatures.

ATZ ceramics are used in dental implants, such as crowns and bridges, in the field of dentistry, and orthopedic applications, such as hip and knee replacements, due to their biocompatibility and resistance to mechanical stress and also used in cutting tools, bearings, ballistic armor, aerospace parts, electrical insulators, and equipment that doesn't wear down easily. They continue to show their worth in fields that need high-performance materials that can handle harsh circumstances.

2.3.7 Additives used in Alumina and zirconia

In their study, Patil and Mutsuddy [218] highlighted the need for meticulous processing of raw ceramic powders, followed by a two-stage sintering process. This approach can mitigate the use of expensive methods such as hot pressing or hot isostatic pressing, which are often chosen to achieve greater densification in the final ceramic composites. Full densification was achieved by sintering the samples in air and in the presence of gas, hence eliminating pores. The hardness and fracture toughness values achieved at room temperature were 17.8 GPa and 4.8 MPa·m, respectively. The enhanced hardness and toughness values were seen as a result of the change in toughness mechanism induced by the inclusion of small-sized ZrO₂ particles in the final composites. Another investigation was conducted by Piconi et al. to assess the stability of Y-TZP (yttria-stabilized tetragonal zirconia polycrystal) by in vitro and in vivo aging of yttria-coated powders (140°C, 0.2MPa water pressure) in rabbits. The strength degradation of Yttria-coated particles was measured to be lower than that of co-precipitated powders. Repeated steam sterilization did not impact the wear resistance of yttria-coated powder. Kong and colleagues introduced an alternative technique for producing ceramic composites by combining hydroxyapatite (HAp) with tetragonal zirconia polycrystal (TZP) powder that is covered with aluminum oxide (Al₂O₃). The coating was applied by dispersing TZP powder in a boehmite suspension and then modulating the pH to cause hetero-coagulation. Integrating ZrO₂ (15 vol%) and

Al₂O₃ (30 vol%) resulted in strength values (300 MPa) and fracture toughness (3 MPa–m) that were almost three times greater than those achieved with pure HAp. The application of a coating helped to mitigate the harmful reaction between HAp and ZrO₂.

This study by Riu et al. [220] examined the effects of incorporating Cr₂O₃ into the Al₂O₃ matrix by studying the alterations in the microstructure and their impact on the mechanical characteristics of the Al₂O₃. The experimental findings showed that the diffusion of Cr ions led to modifications in the microstructures of Al₂O₃, which improved the mechanical characteristics (hardness and elastic modulus) of the final specimens. Conversely, the fracture strength decreased when Cr₂O₃ particles were added above 2 mol.%. Hodgson and Cawley adopted an alternative method, where Y-TZP was doped with TiO₂ particles. The results indicated that maintaining a titanium oxide (TiO₂) concentration below 0.25% yielded Y-TZP material with increased density, which could be smelted at reduced temperatures. The addition of TiO₂ also resulted in an increase in transformation toughening, which was discernible in the microstructure after high-temperature treatment. Their study also indicated that the decrease in mobility caused by the inclusion of TiO₂ may be advantageous for ZrO₂-based composites, particularly when retaining porosity and/or considering surface activity. Similarly, Zhigachev et al. [221] investigated the impact of integrating TiO₂ on the mechanical characteristics and phase composition of Ca-TZP (calcium-stabilized tetragonal zirconia) ceramic. The investigation revealed a decrease in the hardness metric of TiO₂-doped Ca-TZP from 12.2 GPa (undoped Ca-TZP) to 11.3 GPa (0.75 mol.% TiO₂-doped Ca-TZP). In contrast to undoped Ca-TZP (5 MPa-m), the ceramics containing TiO₂ in the 0.5-0.65 mol.% exhibited the most outstanding fracture toughness value of 9.1 MPa–m. Two series of ZrO₂-HAp composites (40 and 60 vol% of ZrO₂) were manufactured by Silva et al. [222]. The sintering behavior, changes in microstructural features, and adjustments in the mechanical properties of the

composites were studied. The raw powders were compressed by uniaxial force at 700 MPa and then sintered in air at temperatures ranging from 1200 to 1500 °C for 3 hours. The observed relative densities varied from 89% to 91% of the theoretical values. Quantitative tests assessed density, micro-Vickers hardness, Young's modulus, Poisson's ratio, and ultimate compression strength. These results were then compared to the mechanical characteristics of human tissues and biomaterials. Findings indicated that composites with 60 vol% ZrO₂ concentration showed the most improved characteristics. Enhanced characteristics in the series were mainly attributed to the higher ZrO₂ phase composition. However, the series sintered at 1300 °C and/or 1400 °C achieved exceptional mechanical qualities. Conversely, all the composites sintered at 1500 °C presented the worst properties, primarily due to large-scale micro-cracking.

Tanaka and colleagues [223] presented a novel nanocomposite, Ce-TZP/Al₂O₃, and conducted a comparative analysis of its biocompatibility, phase stability, and wear properties with the currently used conventional ceramics (Al₂O₃ and Y-TZP) in complete joint prosthesis. The mechanical properties of sintered samples were analyzed for elastic modulus (247 GPa) using the resonance vibration method, flexural strength (941 ± 34 MPa) using 3-point bending, hardness (11.71 ± 0.03 GPa) using Vickers indentation, and fracture toughness (20.05 ± 0.22 MPa.m) using the indentation-fracture method. The evaluated nanocomposites had superior mechanical characteristics to Al₂O₃ and Y-TZP ceramics, indicating their potential suitability for hip joint replacement material. A study by Maneshian and Banerjee [224] found that extended ball milling (mechanical alloying) followed by sintering can enhance the fracture toughness, hardness, and densification of Al₂O₃/ZrO₂ composites. A study conducted by A. H. De Aza et al. [181] focused on assessing the influence of processing parameters, crack propagation, microstructures, and their effects on the mechanical characteristics of next-generation Al₂O₃, ZrO₂, and ZTA

bioceramics produced using the colloidal processing technology. The slow crack-growth behavior experiments of the chosen three ceramics showed increased dependability for orthopedic applications. These ceramics were able to withstand stresses twice as high as monolithic Al₂O₃ without experiencing any failure. Fracture toughness and hardness of Ce–Y–TZP (ceria-yttria stabilized tetragonal zirconia) ceramics produced by precipitation, milling, isostatic pressing, and sintering were assessed by Lin and Duh [225]. This study investigates the direct correlation between the composition of Ce-Y-TZP ceramics and their hardness. Furthermore, the study found that fracture toughness rose as the initial temperature of the m→t transition and tetragonality increased. A study conducted by Kim et al. in the same year investigated the impact of including calcium fluoride (CaF₂) on the densification, sinterability, and mechanical characteristics of HAp-10, 20, 30, 40 ZrO₂ composites (0, 5, and 10 vol% CaF₂) for biomedical purposes. This work proposed that among the composites produced at a temperature of 1350 °C and treated for 1 hour, the composites containing 2.5 vol% CaF₂ exhibited the highest flexural strength value of around 180 MPa, compared to the other composites produced without including CaF₂ (40 MPa). As the concentration of CaF₂ increased beyond 2.5 vol%, the flexural strength value significantly declined. Furthermore, the composites containing 2.5 vol% CaF₂ exhibited the highest fracture toughness value of approximately 2.3 MPa.m. In their study, Mangalaraja et al. [226] examined the relationship between the addition of ceria content (1-4%) and the physical, mechanical, and thermal properties of Y-ZTA (yttria stabilized zirconia toughened alumina) composites. The findings revealed a reduction in the composites' mechanical properties (hardness, fracture toughness, modulus of elasticity, and modulus of rupture) as the ceria content varied from 0 to 5 vol%. The decrease in mechanical characteristics was ascribed to increased apparent porosity made possible by the solid-state mixing of powders resulting from the addition of ceria. The work is

undertaken by Celli et al.[227] aimed to examine the fractal architecture of the fractures formed in Al₂O₃-ZrO₂ composites. An empirical relationship was identified between fractal dimension and fracture toughness, indicating that intergranular fracture occurs in pure ZrO₂, pure Al₂O₃, and composites with a larger proportion of Al₂O₃.By analyzing the fractal energies assessed using both the conventional and fractal approaches, it was shown that composites with greater ZrO₂ concentration exhibited a transgranular fracture mechanism. In addition, this work explored the possibility of elucidating the intricate fracture mechanisms of brittle materials in order to understand their fracture behavior. Gaining knowledge about the transition toughening process has made significant progress, but customizing and adjusting the toughness of ceramics continues to be a significant obstacle in the current situation. Therefore, Basu et al. [228] attempted to modify the toughness of ZrO₂-Al₂O₃ composites by meticulously designing the ZrO₂ matrix and using a mixing method with identical sintering conditions. This proposed approach demonstrated that the fabricated TZP-Al₂O₃ composite attained a toughness of around 10 MPa-m, which was twice as high as that of commercially available ZrO₂-20 wt% Al₂O₃ ceramics. The same group conducted a separate investigation to assess the alterations in the microstructure and mechanical characteristics of Y-TZP ceramics with different yttria concentrations. Y-TZP ceramics reinforced with 20 wt% Al₂O₃ were synthesized using powder mixing and hot pressing methods under vacuum conditions at 1450°C for 1 hour. The produced ceramics exhibited fracture toughness within the range of 2 MPa-m to 10 MPa-m due to the controlled accumulation of m-ZrO₂ particles using the proposed approach. An improved toughness value of 10 MPa-m and an increased flexural strength of around 1250 MPa were achieved for mixed grade 2 mol.% yttria produced by hot pressing. An investigation of the microstructure using electron probe microanalysis revealed the presence of intentionally introduced ZrO₂ particles without yttria in the initial

powder mixtures. This likely caused a redistribution of yttria during the sintering process, eventually enhancing the transformability. This work proposed that to increase toughness values in the region of 2 to 10 MPa-m, the composition of the initial powder in yttria-doped ZrO₂ was adjusted by the controlled addition of ZrO₂ particles to 3 mol.% Y₂O₃ [229]. Silva and colleagues investigated the toxicity and efficacy of employing Si₃N₄-based ceramics for load-bearing purposes. Ceramics were synthesized using two distinct compositions of sintering aids: ytterbium, yttrium, and Al₂O₃. These ceramics were produced by sintering in a carbon-resistant furnace under a nitrogen atmosphere. The created ceramics were assessed for their fracture toughness (5 MPa-m) and hardness (13 GPa). The exceptional mechanical properties and non-toxic nature of Si₃N₄-based ceramics make them a promising option for heavy load-bearing applications in the human body, such as knee and hip joints. Moraes et al. [230] introduced a new variation of Y-TZP composites by modifying the ZrO₂ concentration from 5 to 80 wt%. The study's findings indicated that including ZrO₂ in composites enhanced numerous favorable mechanical characteristics, including density, flexural strength, and fracture toughness. Furthermore, they stated that these composites exhibited a flexural strength of 93% and a fracture toughness of 29% compared to pure Al₂O₃ ceramics. Ahn and colleagues [231] conducted a study to assess the impact of ZrO₂ reinforcement on the microstructure and mechanical characteristics of nanocomposites based on HAp. The performance of 1.5 wt% ZrO₂ demonstrated the most favorable outcomes in terms of Vickers hardness. Using 1.5 wt% of ZrO₂ particles enhanced the bending strength of HAp-ZrO₂ nanocomposites from 183 to 243 MPa. The predominant phase of HAp grains was around 100 nm in size, which suggests its remarkable ability to provide enhanced bioactivity.

Moreover, Magnani and Brillante conducted an experimental study to examine the influence of different compositions and sintering methods on the mechanical

characteristics and residual stresses in ZrO₂-Al₂O₃ composites. Nanoparticles of chromium oxide (Cr₂O₃) and magneto plumbite were added to ZTA under various fabrication circumstances. Incorporating platelet reinforcements (magneto plumbite) suppressed the t-ZrO₂ to m-ZrO₂ conversion during the stress-induced transformation toughening mechanism, resulting in the highest fracture toughness achieved at 0.5 wt% Cr₂O₃ and 2 mol.% Y₂O₃. Furthermore, this investigation revealed that the stabilizer content significantly influenced the transformability of Zhang and his colleagues [232].

Silica was found at triple junctions but neither at grain boundaries nor in the lattice. Undoped zirconia ceramics exhibited faceted grains and significant internal stresses, while doped zirconias showed a much more rounded microstructure and a lower level of internal stresses. Low-temperature degradation (LTD) and slow crack growth (SCG) measurements were conducted on the different batches. The addition of silica strongly increases LTD resistance without affecting the SCG behavior. The presence of a glassy phase does not degrade the crack resistance but greatly improves the LTD resistance [71]. To avoid degradation, the stability of the tetragonal phase can be increased by either decreasing the grain size or increasing the yttria content. However, both solutions reduce the phase transformation toughening, resulting in a reduced crack propagation resistance. Other approaches have been proposed, among them the addition of dopants such as silica and alumina. A limited amount of literature indicates that silica additions should increase the LTD resistance without providing further explanations. Thus, the present study aimed to understand better the effect of SiO₂ on the microstructure of 3Y-TZP ceramics and on SCG and LTD in order to investigate whether LTD resistance can be increased without decreasing mechanical properties [71]. The lifetime of tetragonal zirconia ceramic implants is controlled by their resistance to slow crack growth and low-temperature degradation (LTD). A decrease in the grain size is often proposed to decrease the effect of LTD on

zirconia; however, this decrease also reduces the resistance to crack propagation. An alternative approach is reported here; such an approach consists of introducing a silica glass phase only at triple grain junctions without changing the grain size. Adding a small amount of silica (0.5 wt%) decreases the internal stresses, improving the resistance to LTD without affecting the resistance to crack propagation [72]. Tetragonal zirconia ceramics that have been stabilized with yttria (so-called Y-TZP) exhibit high toughness (KIC) (up to 7 MPam^{1/2}) and strength (up to 2 GPa). Such values are due to both small grain size and a phase-transformation toughening mechanism. More than 350,000 zirconia femoral heads have been implanted, and there has been good clinical success [233].

Zirconia ceramics can exist as two polymorphs at room temperature: a tetragonal, metastable phase (the t-phase, which can be obtained using stabilizers such as yttrium or cerium oxides) and a monoclinic, stable phase (the m-phase). A cubic phase exists in zirconia with higher concentrations of stabilizers. The t-phase is generally obtained after sintering yttria-stabilized tetragonal zirconia polycrystal ceramics with 2-3 mol% Y₂O₃; however, this phase can transform to the m-phase [72,234]. Increasing the grain size of TZP improves the resistance to slow crack growth (SCG). At the same time, increasing grain size decreases the stability of tetragonal zirconia, thus favoring the isothermal transformation of tetragonal grains to the monoclinic phase at the surface of zirconia. This mechanism is known as low-temperature degradation (LTD) [234].

The volume increase associated with the t-m transformation induces microcracking and an increase in the surface roughness. Such an increase in roughness with time may increase the wear rate. The volume increase associated with the t-m transformation induces microcracking and surface roughness. Such an increase in roughness with time may increase the wear rate.[234].The different experimental features of the LTD in TZP are as follows

- The transformation is slow at room temperature and shows a maximum rate at 200°-300°C.

- The transformation is accelerated in humid media (in the presence of water or water vapor).
- The transformation starts on the surface and extends into the bulk

2.3.8 Additive used in Alumina Toughened Zirconia

Minimal amounts of copper oxide, manganese oxide, and stainless steel facilitate the low-temperature densification of ATZ using the microwave method. It was found that the addition of sintering additives was beneficial in aiding the densification of ATZ without causing grain coarsening. In addition, it was revealed that regardless of dopant addition, all sintered samples exhibited a dual microstructure comprising tetragonal zirconia (major) and alumina (minor) phases. There was no monoclinic phase formation after sintering, thus indicating that the stability of the tetragonal phase was not disrupted by the dopant addition, alumina addition, and microwave sintering method. The addition of these dopants was beneficial in enhancing densification, Vickers hardness, and fracture toughness of ATZ regardless of sintering temperatures. In particular, the addition of 0.2 wt% CuO was most effective in aiding the low-temperature sintering of ATZ with a relative density of over 99%, a hardness of above 13 GPa, and a fracture toughness of higher than 7 MPam^{1/2} when sintered at 1500°C. In contrast, the undoped ATZ required a high temperature to obtain results similar to those of the CuO-doped ATZ samples. The statistical analysis revealed that the CuO-doped ATZ sample exhibited the highest mean values for both hardness (H) and fracture toughness (K_{Ic}) among all the tested samples. This indicates that the CuO-doped ATZ sample is highly significant and effective in improving H and K_{Ic} across all temperature conditions.[213]

2.4 Research Gap

Although strides have been made in utilizing Rice Husk Ash (RHA) and Animal Waste Bone (AWB) in producing biomaterials, there is still a significant lack of comprehensive studies dedicated to exploring the concurrent application of RHA and AWB for biomaterial fabrication.

While advancements have been made in the field of alumina, zirconia, and, more specifically, Alumina Toughened Zirconia (ATZ), there is still a discernible research gap regarding the role of Rice Husk Ash (RHA) and Animal Waste Bone (AWB) in the fabrication of ATZ-based biomaterials.

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