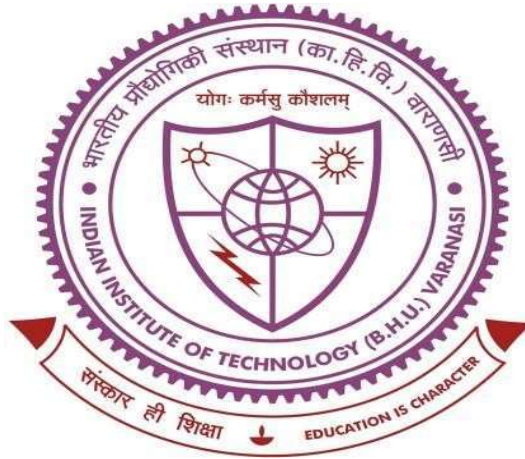


Synthesis and physico-mechanical Characterization and
Biocompatibility Evaluation of Waste Derived ATZ
Composites



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Chapter 8

Conclusions & Future Directions

This chapter offers the key findings obtained from the study carried out in this thesis.

In addition, the study examines several unsettled matters that demand further analysis and, therefore, suggests recommendations for future research.

8.1 Conclusion

This chapter contains an overview of the findings and conclusions that were derived from the research study. As a result of all of the research that has been covered up to this point, it is possible to determine that the primary objective of the research was to find ways to make agricultural waste and waste animal bone more valuable by developing the possibility of using them as a source of biomaterials. The most important conclusions that can be taken from all of the prior research work are as follows:

The development of silica compacts using rice husk ash-derived amorphous and crystalline silica provided these primary conclusions:

- RHA silica is in the amorphous form up to 800°C, which ultimately converts to crystalline after 900°C.
- The temperature interval between 800°C and 900°C is the conversion stage in which RHA silica transforms from amorphous to crystalline.
- The cristobalite phase is the main dominant phase at higher temperatures, with some traces of tridymite.
- Tridymite concentration depends on the quantity of impurities like CaO in RHA. Tridymite to cristobalite inversion occurs at 1470°C. But here, instead of transitioning from tridymite to cristobalite conversion, RHA directly undergoes phase transformation to cristobalite.
- The obtained silica compacts possess bulk density and compressive strength in the range of 1.59 g/cm³-1.97 g/cm³ and 20 MPa–52 MPa, respectively.

- Samples prepared using amorphous silica show a lower green density than crystalline silica samples.
- However, the sintered densities and mechanical properties of samples prepared using amorphous powder are better than those of crystalline powder samples.
- So, it can be concluded that amorphous RHA obtained at lower temperatures is better used to make silica compact for better strength. Still, if porosity is the concern, crystalline RHA at higher temperatures will be the preferred one.

RHA-derived amorphous and crystalline silica doped Alumina toughened Zirconia biocomposite provided these primary conclusions:

- The comprehensive investigation into the impact of silica doping on the properties of the developed ATZ biocomposite has provided valuable insights into the intricate relationships among microstructure, physico-mechanical attributes, and biological behavior.
- Rice Husk Ash (RHA) as a silica source has been established as a reliable means to enhance the material's characteristics. The study highlights that doping silica up to 5 wt% in ATZ contributes to increased grain growth of zirconia particles; however, higher wt% of silica leads to reduced grain growth in both alumina and zirconia particles, adversely affecting the mechanical properties of the biocomposite.
- Notably, the comparison between amorphous and crystalline silica reveals that amorphous silica demonstrates superior grain growth and densification of ATZ samples. The significance of the silica doping level is evident, with physical and mechanical properties experiencing enhancement up to 1 wt% doping. Beyond this threshold, a notable and significant decrease in these properties is observed. Hydrothermal ageing resistance is improved with silica doping, particularly with amorphous silica, showing superior resistance compared to crystalline silica. The optimal results for aging resistance are obtained with up to 1 wt% silica doping, beyond which a decline is noted. Furthermore, the increased hydroxyapatite layer underscores the positive influence of silica doping on bioactivity.

- In summary, the research demonstrates that RHA is a suitable source for obtaining silica, which can be effectively utilized as an additive, particularly in the amorphous form derived from RHA, to enhance the mechanical and aging properties of ATZ biocomposites. These findings contribute to the ongoing exploration of advanced biomaterials, offering promising avenues for developing improved biocompatible materials with enhanced performance in biomedical applications.

Triphasic calcium phosphate to fabricate Alumina toughened Zirconia biocomposite provided these primary conclusions:

- The bioactivity of a biocomposite based on alumina-toughened zirconia (ATZ) has been significantly improved by incorporating AWB as a dopant, reducing processing costs.
- The abundance of calcium and phosphorus elements in AWB promotes the formation of calcium phosphate phases within the composite.
- A precise thermal treatment process is employed to consolidate the ATZ particles and induce the formation of various calcium phosphate phases (HAp/TCP/Wk), thereby enhancing the biocompatibility, osteoconduction, and osteointegration properties of the ATZ composite.
- Magnesium's small yet significant presence in AWB facilitates the conversion of the Whitlockite phase, a primary constituent found in human bone.
- The XRD results revealed that AWB converts to HAp, which partially decomposes to TCP phases. With the increase in AWB concentration, these phases (HAp, TCP, and Wk) are more evident with a small portion of CaZrO₃, which comes out due to a reaction between decomposed CaO from HAp and Zirconia. Transformation of tetragonal Zirconia to cubic and tetragonal forms is also favored by HAp decomposition.
- The FTIR data also supports the XRD results, confirming the presence of triphasic calcium phosphate in the form of HAp, TCP, and Wk, along with Alumina and Zirconia phases. In vitro proof-of-concept results have demonstrated that the

incorporation of AWB leads to an increased formation of an apatite layer, confirming the enhanced bioactivity of the composite.

- Including triphasic structures in the form of HAp, TCP, and Whitlockite confers superior biological properties to the biocomposite compared to a single-phase monolith. This study reveals that AWB is a promising "waste-to-resource" raw material suitable for bone graft substitutes in bone repair and regeneration.
- Furthermore, using ATZ as the main matrix imparts sufficient mechanical strength to the biocomposite for load-bearing applications.
- The ATZ-AWB biocomposite exhibits bulk density, apparent porosity, hardness, and bending strength within the ranges of 3.7-5.2 g/cm³, 3.6-17%, 2-9 GPa, and 620-1000 MPa, respectively.
- In terms of bioactivity (due to the presence of triphasic calcium phosphate-HAp/TCP/Wk) and cost-effectiveness, the present ATZ-AWB biocomposite surpasses zirconia-based biocomposites doped with synthetic materials. Altogether, the utilization of AWB not only amplifies bioactivity but also curtails the expenses associated with zirconia-based biomaterials, thus fortifying the principles of biocircularity and bioeconomy through a truly sustainable approach.

The porous scaffold's physical, mechanical, and biological properties are comparable to those of real bone. Thus, the study mentioned above can conclude that agricultural waste and animal bone can be used to construct scaffolds suited for tissue engineering and dental applications.

8.2 Future scope of the work

Based on what we learned from this study, the next steps for the work will be as follows:

- Potential future avenues include investigating the benefits of integrating these waste materials (Rice husk and Animal Waste Bone) into other biomaterials.

Such exploration may increase their bioactivity while simultaneously reducing production costs.

- It is possible to create other combinations of these ceramic-based samples using advanced manufacturing methods, such as 3D printing.
- Defect-free samples can be prepared using advanced sintering techniques such as Spark plasma sintering and microwave sintering.
- Animal testing and systematic analysis might follow in Vivo testing. If the in vivo results are positive, the Porus scaffold can be suggested for clinical study per medical criteria.

List of Publications

- A comparative study on the physico-mechanical properties of silica compacts fabricated using rice husk ash-derived amorphous and crystalline silica. **Ashutosh Gupta**, Vaibhav Pandey, Mayank Kumar Yadav, Kalyani Mohanta, Manas Ranjan Manjhi. **Ceramics International**, <https://doi.org/10.1016/j.ceramint.2022.07.098> (First Author)
- Strengthening bio-circularity by reinforcing waste-derived Triphasic calcium phosphate to fabricate alumina-toughened Zirconia biocomposite with enhanced bioactivity. **Ashutosh Gupta**, Vaibhav Pandey, Manas Ranjan Majhi. **Sustainable Materials and Technologies**. (First Author)
- Microstructural characterization and hydrothermal ageing resistance of RHA-derived amorphous and crystalline silica-doped Alumina toughened Zirconia biocomposite. **Ashutosh Gupta**, Vaibhav Pandey, Manas Ranjan Majhi. **Journal of Sustainable Technology** (under review). (First Author)
- Synthesis of graphene oxide and green properties of dry pressed alumina compacts with the small addition of graphene oxide/graphite” Vaibhav Pandey, Kalyani Mohanta, Ajay Kumar, Mayank Kumar Yadav, **Ashutosh Gupta**. **Journal of the Australian Ceramic Society**, 56, 1367-1375 (2020), <https://doi.org/10.1007/s41779-020-00487-9> (Co-Author)
- Synthesis, Morphological and Thermomechanical Characterization of Light Weight Silica Foam via Reaction Generated Thermo-Foaming Process. Vaibhav Pandey, Mayank Kumar Yadav, **Ashutosh Gupta**, K. Mohanta, S. K. Panda, V. K. Singh. **Journal of European Ceramic Society**. (2022) (Co-Author)
- Tensile and Dry Sliding Wear Properties of Compo-Cast SiO₂ Coated ABOw/Al-319 Composites Neeraj Pandey, S.S. Kasana, Abhishek Kumar Singh, **Ashutosh Gupta**, Manas Ranjan Majhi Springer Nature B.V. 2023(Co-Author)