Mechanical Reliability and Modeling of Hydroxyapatite Scaffold

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Research Article

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Abstract

Significant contributions on the improvement of the mechanical properties of hydroxyapatite (HAp) have been widely reported. However, failure analysis (mechanical reliability) and modeling are missing. This article filled the gap by conducting Two-parameter Weibull distribution assisted by modeling to investigate the mechanical reliability of HAp. The employed HAp was characterized under SEM/EDS analysis. The results revealed the characteristics of HAp and also the nature of the synthesis route employed through its irregular morphology. The Two-parameter Weibull distribution analysis was conducted on the hardness and compressive strength of HAp scaffold. The characteristic hardness and compressive strength, coupled with their corresponding bounds, failure rates, and correlation coefficients were been presented. The Weibull analysis with the assistance of modeling revealed HAp fabricated under 10 KN compaction load and sintered at 1100 °C as the most reliable sample under hardness condition, while HAp fabricated under 15 KN compaction load and sintered at 1000 °C gave the most reliable characteristic under compression. However, 15 KN compaction load and 1100 °C sintering temperature showed the best reliability on the overall mechanical (hardness and compressive strength) reliability. Future study is recommended on the reliability of HAp scaffolds considering other mechanical properties that are essential for biomedical application.

1.0 Introduction

Bioceramics have been widely used in bone recovery. Its wide application in bone recovery has added value to human's well-being [1]. There are different classes of bioceramics available for orthopedics. Its choice depends on the severity or nature of the defects to be treated. The mechanical integrity of bioceramic is designed to suit different orthopedic applications [1–2]. Resorbable bioceramic is a class of bioceramic that has great capability to transform tissue engineering. They are used as a scaffold and allows bone recovery in the natural tissue repair phenomenon. It requires no surgery because of its resorbability and its decomposed products are not toxic [1–3]. Hydroxyapatite (HAp) is a resorbable bioceramic and has been widely used in bone repairs [4]. However, because of its poor mechanical integrity, it is limited for load bearing application in orthopedics. Researchers have improved on the mechanical integrity of HAp. Few out of many researches are as mentioned next. Martin & Brown [5] employed particulate solid reactants technique to produce calcium-deficient and carbonated HAp, and evaluated their mechanical properties at different liquid-to-solid weights ratio. They discovered that calcium deficient HAp exhibited superior mechanical properties compared to carbonated HAp. Suchanek et al. [6] reinforced HAp fine crystals with HAp whiskers using hydrothermal synthesis. It was established that the reinforcement improved HAp toughness without depleting its biocompatibility. Abifarín [7] optimized sintering parameters for better multiple mechanical properties of HAp. He discovered that fabrication of HAp with better mechanical properties is feasible with little or no forming pressure. Noviyanti et al. [8] doped HAp with different concentrations of La particles. Their findings showed that an increase in the concentration of La increase the hardness of HAp. Obada et al. [9] studied the effect of sintering temperature of HAp under low-cold forming pressure. They discovered that an increase in

sintering temperature increased the mechanical properties of HAp. Obada et al. [10] in another study reinforced HAp with 15 wt% of kaolin. It was observed that the HAp composite exhibited higher compressive strength compared to its non-reinforced counterparts. Abifarin et al. [11] furthered worked upon 15 wt% kaolin reinforced HAp by employing multi-objectives optimization on fabrication parameters for better mechanical properties of the composite. Recently, in another study, Abifarin et al. [12] employed numerical analysis to enhance the mechanical properties of HAp scaffold. It was discovered that compaction load employed in their study contributed immensely to the mechanical enhancement of the fabricated HAp scaffold.

Despite significant contribution of several researches on the mechanical improvement of HAp, failure analysis (reliability) and modeling of HAp are missing. However, many researches have been conducted on failure analysis of other materials for high performance. Few out of many are: Wolf et al. [13] used strength measurement and fractography to investigate failure of alumina-glass dental composites. They discovered that the strength and fracture toughness of the developed composite were ~ 2 times higher than the then dental ceramics. Aarseth & Prestlkken [14] employed Weibull analysis in the assessment of the physical properties of pelletized feed. It was discovered that Weibull analysis helped to have more homogeneous feed. Prakash et al. [15] investigated the mechanical reliability of 3D printed HAp reinforced polylactic acid (PLA) porous scaffolds. It was stated in their work that Weibull distribution showed that the printed porous scaffolds were mechanically reliable. Pereira et al. [16] employed Weibull analysis on the mechanical properties of three yttrium-stabilized zirconia (YSZ) materials with different translucent properties. The analysis unveiled the sample with the highest characteristic strength. Roitero et al. [17] employed Weibull analysis on the mechanical properties on the surfaces of dental zirconia patterned with Nd:YAG laser interference. They concluded that the samples with the enlarged critical defects, had the defects at the origin of the decrease in Weibull characteristic strength and the decrease in Weibull modulus. Guilardi et al. [18] investigated the structural reliability of an yttrium-stabilized tetragonal zirconia polycrystalline ceramic. They discovered that Weibull analysis showed a significant increase in the characteristic strength after grinding (Coarse=Xfine>Ctrl), while aging did not lead to any deleterious impact. Gruber et al. [19] investigated the effect of architecture and loading conditions on the mechanical reliability of ceramic-based substrates. The results showed the significance difference in characteristic strength of the modeled ceramic sub rates under different architectural features, loading types, and or environments. Sglavo & Bellettati [20] improved the mechanical reliability of two different ceramic laminates composed of porous alumina and alumina/zirconia layers by tailoring the residual stress profile and corresponding fracture toughness of the material. It was discovered that the mechanical performance were solely related to the specific architecture of the laminate.

Having discussed the state of the art of the subject matter, this study conducted a research on mechanical reliability and modeling of hydroxyapatite scaffold as a continuation of the study conducted by Abifarin et al. [12], in which they identified the gap this study filled under their conclusion and future studies. Two-parameter Weibull analysis was employed to investigate the mechanical reliability, while modeling and interaction studies of the manufacturing parameters were done using Origin 2019

software. The analyzed mechanical properties of HAp in this study are hardness value (HV) and compressive strength (MPa).

2.0 Materials And Methods

2.1 Materials characterization and data curation

The HAp powders were obtained from the work of Abifarin et al. [12] and characterized with SEM/EDS analysis. The mechanical properties (hardness value and compressive strength data) were obtained from the work of Abifarin et al. [12] as well. The mechanical data were collected in five (5) replications for the analysis according to the description in Table 1.

Table 1
Sample and acronyms description

Codes	Description
C1	HAp under 5KN compaction load at 900 °C sintering temperature
C2	HAp under 10KN compaction load at 900 °C sintering temperature
C3	HAp under 15KN compaction load at 900 °C sintering temperature
C4	HAp under 5KN compaction load at 1000 °C sintering temperature
C5	HAp under 10KN compaction load at 1000 °C sintering temperature
C6	HAp under 15KN compaction load at 1000 °C sintering temperature
C7	HAp under 5KN compaction load at 1100 °C sintering temperature
C8	HAp under 10KN compaction load at 1100 °C sintering temperature
C9	HAp under 15KN compaction load at 1100 °C sintering temperature
HV	Hardness value
CS	Compressive strength

2.2 Methods

2.2.1 Mechanical reliability analysis

In this analysis, Two-parameter Weibull distribution analysis [21] was employed and the mechanical (hardness and compressive strength) probabilities were investigated. For any arbitrary random variable x , the cumulative distribution function of the Weibull distribution is given as Equation 1:

$$F(x) = 1 - \exp\left(\frac{-x}{\alpha}\right)^\beta \quad (1)$$

Where x is the mechanical strength (hardness or compressive strength), β is the shape parameter and it is a function of failure rate (Weibull modulus or strength reliability), α is the scale parameter and it is the characteristic mechanical strength (hardness or compressive strength).

2.2.1 Mechanical regression modeling and interaction plot

Regression analysis was employed to model the mechanical (hardness and compressive strength) reliability of HAp at different fabrication conditions using Minitab 16 software [38]. A is denoted as the sintering temperature, while B is denoted as the compaction load.

Interaction plot was employed using Origin 19 software to examine the effect of sintering temperature and compaction load on the mechanical reliability of HAp. It was also done to understand and to tailor the fabrication conditions for the best mechanical reliability. Fig. 1 shows the overview of the work done in this study.

3.0 Results And Discussions

3.1 SEM/ EDS analysis

The EDS analysis shows the elemental compositions present in the HAp powder. Fig. 2 shows the elemental composition of the HAp powder. The result confirms the presence of calcium and phosphate and they are the prominent elements present in the bulk powder. This agrees with the characteristic nature of HAp [4, 9–10, 22–23].

The surface morphology of the HAp specimen reflected in three different magnifications (A : 100 μm , B : 20 μm , and C : 10 μm) are shown in Fig. 3. The images reflect the white colored-nature of HAp powders, which is a typical color of HAp powder [24–33]. It is also important to note that the SEM images reflected irregular morphology. This is attributed to the synthesis conditions, as the HAp was synthesized from bovine bones, crushed and sieved, posited by Abifarín et al. [12].

3.2 Hardness reliability and modeling

3.2.1 Hardness reliability analysis

The failure rate analysis was conducted on the HAp samples based on its hardness property. The replicated hardness data was ranked from lowest to highest as displayed in Table 2. The rank was done to enable the reliability analysis.

Table 2
Hardness value (HV) replication data for HAp samples

Rank/Samples	HVC1	HVC2	HVC3	HVC4	HVC5	HVC6	HVC7	HVC8	HVC9
1	81.78	68.38	65.98	88.79	66.89	68.87	89.88	88.78	85.79
2	82.5	70.5	66.99	89.95	67.67	68.89	98.56	91.11	87.65
3	87.99	72.11	68.73	89.99	73.27	70.93	98.77	92.88	88.78
4	88.92	77	71.09	92.44	73.44	71.2	104.89	94.39	89.99
5	90.11	78.78	72.9	96.33	75	74.22	111.22	96.45	90.37

Having analyzed the hardness data of HAp samples, Fig. 4 shows the Two-parameter Weibull plots and the characteristic hardness value of all the HAp samples. The Weibull distribution plots of all the samples displayed that the hardness data obtained were within the 95% confidence bound. This shows the possible repeatability and reliability of the experiment. However, samples C1, C3, C7, C8, and C9 displayed that almost all the replicated hardness data aligned with their true or expected hardness value. Even though all the samples are reliable under hardness conditions, but the aforementioned samples are more reliable than the rest of the samples. Emphatically, the most reliable sample is sample C8, but sample C7 exhibited the highest characteristic hardness value (See Fig. 4).

To elucidate the above qualitative analysis of HAp reliability, Table 3 reflect the quantitative analysis of the Weibull analysis. The results showed that all the samples scale parameters (characteristic hardness) and the shape parameters are within the 95% bounds. The correlation coefficients of all the samples range from 0.874 to 0.995. The lowest correlation coefficient being the sample C4, while the highest correlation coefficient is the sample C8, as supported by the distribution plot (Fig. 4). The value of the shape parameter displayed is greater than 10. This could mean the high wear susceptibility of the samples. It also reflects the ceramic nature of all the samples. On the other hands, it can also mean a very high censored hardness data with a very little failure. The high shape parameter of all the samples means, when the environmental conditions or noise become more severe at critical time, the HAp can fail at the accelerated rate. The interpretations given to the shape parameters are adopted from the work of O'Connor & Kleyner [34]. Although, sample C7 reflected the highest characteristic hardness value (113.27 HV), however, sample C8 having characteristic hardness value of 97.37 HV is the most reliable material under hardness testing or conditions. Hence, it is safer and reliable to employ sample C8 when the most important mechanical property to be considered for orthopedic is hardness and the required hardness is not beyond 97.37 HV. The sample C8 is a promising candidate for load bearing biomedical application, as the hardness of human bone is generally not up to its exhibited characteristic hardness value [35].

Table 3
Two-parameter Weibull characteristics for HAp hardness value

Samples	Characteristic hardness value, α	95% Confidence band for α	Shape parameter, β ,	95% Confidence bound for β	Correlation coefficient, ρ
C1	91.97	84.41-95.47	26.7	14.55-61.28	0.937
C2	80.52	75.86-85.19	17.7	9.64-40.63	0.956
C3	73.83	70.8-76.83	25.09	13.67-57.61	0.96
C4	96.36	93.1-99.5	30.71	16.73-70.5	0.874
C5	76.68	73.29-80.03	23.32	12.71-53.54	0.932
C6	74.34	72.01-76.60	33.25	18.12-76.34	0.891
C7	113.27	105.07-121.64	14.02	7.64-32.19	0.976
C8	97.37	94.44-100.24	34.46	18.78-79.12	0.995
C9	91.25	89.59-92.85	57.44	31.30-131.86	0.993

3.2.2 Hardness reliability modeling

Figure 5 shows the effect of sintering temperature and compaction load on hardness reliability (correlation coefficient) of HAp. The results revealed inconsistent hardness reliability with change in sintering temperature and compaction load. Interestingly, sintering temperature at 1100 °C gave the best hardness reliability, while 10 KN compaction load gave the best hardness reliability. Meaning, to produce HAp with the most hardness reliability, HAp should be conditioned under 10 KN compaction load at 1100 °C sintering temperature. But note that this finding is not the same when optimizing fabrication conditions for the best characteristic hardness value. These fabrication conditions reflects sample C8, which validates the findings displayed in Weibull distribution analysis (see section 3.2.1).

The interaction of the fabrication parameters on hardness reliability is reflected in Fig. 6. The contour plot shows the fabrication settings for possible hardness reliability. The plot shows that is possible to achieve hardness reliability greater than 0.95 within the range of 900-920 °C sintering temperature under 10-15 KN compaction load. It also shows that the lowest reliability (< 0.89) can be achieved around 980-1020 °C sintering temperature when compaction load is less than 6 KN and greater than 13 KN. Importantly, the best hardness reliability (>0.98) can be achieved around 1080-1100 °C sintering temperature from 6 KN compaction load and above. Having observed the qualitative model, the mathematical model of hardness reliability is generated using Minitab 16 software, and it is shown in Equation 2. The experimental and modeled hardness reliability are presented in Fig. 7. From Fig. 7, it is seen that the experimental hardness reliability for all samples except for C4, C5, and C6 followed the pattern of the modeled reliability. This explains the level of confidence on the model equation.

Hardness reliability = $0.742 + 0.000185A + 0.0019B$ (2)

3.3 Compressive strength reliability and modeling

3.3.1 Compressive strength reliability

Similar to the analysis done for hardness reliability, Two-parameter Weibull analysis was conducted on compressive strength of HAp samples. The replications were ranked in ascending order as shown in Table 4 for the analysis.

Table 4
Compressive strength (MPa) replication data for HAp samples

Rank/Samples	CSC1	CSC2	CSC3	CSC4	CSC5	CSC6	CSC7	CSC8	CSC9
1	27.88	31.2	34.98	36.58	39.76	39.77	41.95	44.87	49.79
2	28.77	32	35.78	36.77	40.12	41.27	42.22	45.1	50.11
3	29.87	33.76	36.45	37.88	40.99	43.22	44.34	45.99	54.33
4	34.42	34.88	36.87	39.87	41	45.02	44.76	47.46	59.89
5	35.65	39.55	37.66	39.94	42.33	46.2	45.22	49.33	60.21

Two-parameter Weibull plots of the compressive strength of all the HAp samples are displayed in Fig. 8. Similar to hardness reliability analysis, the Weibull distribution plots show that the actual data are within the 95% confidence bound. This shows the efficacy of the performance or reliability of the HAp samples under compression loading. Interestingly for compressive strength reliability, all the experimental data aligned very well with the ideal data. The only sample showing a little deviation was sample C8, yet its compressive strength data fitted well with the expected data. It can be concluded from the graphs that all the HAp samples are reliable under compressive strength condition. However, C9 exhibited the highest characteristic compressive strength, while C1 exhibited the lowest characteristic compressive strength. This findings are supported by the findings of Abifarin et al. [12].

To substantially understand the observations made on the Weibull distribution plots, Table 5 displays numerical data from Weibull distribution analysis. The results showed that all the samples scale parameters (characteristic compressive strength) and the shape parameters (failure rates) are within the 95% bounds. The correlation coefficients of all the samples range from 0.908 to 0.993. Sample C8 showed the lowest correlation coefficient, while C3 showed the highest correlation coefficient. This numerical findings elucidate the Two-parameter Weibull plots (Fig. 8). All the shape parameter values are greater than 10, except for sample C1 (9.43), however, they all showed a high value of shape parameter. The implication of this discovery has been elaborated in section 3.2.1 [34]. Essentially, sample C9 exhibited the highest characteristic compressive strength (63 MPa), however, sample C3 with characteristic compressive strength of 37.97 MPa gave the best reliability and goodness of fit under compression. Hence, sample C3 is recommended when orthopedic requirement for compressive strength

is below 37.97 MPa. This shows that samples C3 is a promising implant for human trabecular bone, having the required compressive strength range of 2-12 MPa [36–37].

Table 5
Two-parameter Weibull characteristics for HAp compressive strength

Samples	Characteristic compressive strength, α (MPa)	95% Confidence bound for α , (MPa)	Shape parameter, β ,	95% Confidence band for β	Correlation coefficient, ρ
C1	37.13	33.21-41.26	9.46	5.16-21.73	0.919
C2	39.68	35.99-43.51	10.82	5.90-24.84	0.910
C3	37.96	36.94-38.94	38.99	21.24-89.51	0.993
C4	40.77	39.13-42.39	25.64	13.97-58.87	0.915
C5	42.41	41.39-43.39	43.50	23.70-99.87	0.933
C6	47.32	44.62-50.03	17.95	9.78-41.22	0.986
C7	45.94	44.55-47.30	37.24	18.66-78.60	0.938
C8	49.63	47.59-51.65	25.07	13.66-57.55	0.908
C9	63.00	57.17-68.51	11.92	6.50-27.37	0.919

3.3.2 Compressive strength reliability modeling

The effect of sintering temperature and compaction load on mechanical reliability (correlation coefficient) of HAp is presented in Fig. 9. As was observed for hardness reliability, there is inconsistency on the mechanical reliability with an increase in sintering temperature and compaction load. Contrary to the hardness reliability, compressive strength reliability showed 1000 °C sintering temperature as the best compressive strength reliability, and 15 KN compaction load gave the best compressive strength reliability. This means that HAp fabricated under 15 KN compaction load sintered at 1000 °C sintering temperature would give the best compressive strength reliability. However, it does not mean the fabrications conditions would give the best characteristic compressive strength as indicated in Table 5. The discovered fabrication conditions to have the most reliable HAp under compression described HAp sample C6, which is different from the initial observation. A little difference shows the gap between the experimental correlation and the modeled correlation.

Figure 10 presents the interaction between sintering temperature and compaction load over compressive strength reliability. The contour plot shows the fabrication settings for possible compressive strength reliability. The plot shows that to have better reliability greater than 0.95, compaction load should be set above 10 KN compaction load with sintering temperature in the range of 900-1000 °C. The best compressive strength reliability ≥ 0.98 can be achieved when compaction load is ≥ 14 KN within 900-1000 °C sintering temperature. Despite a little difference in the observed fabrication conditions of the

model relative to the experimental, the interaction plot shows that sample C3 is also a good candidate for compression reliability. Equation 3 presents the mathematical model of compressive strength reliability as generated using Minitab 16 software. The experimental and modeled compressive strength reliability are presented in Fig. 11. The graph shows that the experimental compressive strength reliability aligned with the modeled reliability, except for C1. This shows a very good model of the design.

Compressive strength reliability = $0.989 - 0.000095A + 0.00420B$ (3)

3.4 Selection of fabrication conditions for better multiple mechanical reliability

From Weibull distribution analysis for hardness and compressive strength, the results showed difference in fabrication conditions to have the best multiple mechanical reliability. A need of HAp scaffold exhibiting both hardness and compressive strength reliability in orthopedics may arise. Therefore, it is important to optimize fabrication conditions for the best mechanical (hardness and compressive strength) reliability. This section employed the correlation coefficients for both the hardness and compressive strength, and then average them. The average correlation coefficient was employed to determine a common fabrication settings for higher hardness and compressive strength (mechanical) reliability.

The effect of sintering temperature and compaction load on mechanical reliability (correlation coefficient) of HAp is shown in Fig. 12. The graphs showed that sintering temperature impacted inconsistency in the trend of mechanical reliability of HAp, but increase in compaction load resulted to an increase in the mechanical reliability of HAp. The results displayed 1100 °C as the sintering temperature that led to the best mechanical reliability, while 15 KN compaction load gave the best reliability. It can be concluded that HAp fabricated under 15 KN compaction load and sintered at 1100 °C sintering temperature would give the best overall mechanical (hardness and compressive strength) reliability. This reflected sample C9 as the best mechanically reliable material for load bearing orthopedic application.

Figure 13 (a) presents the interaction between sintering temperature and compaction load over the mechanical reliability. Equation 4 presents the mathematical model of the mechanical reliability. The experimental and modeled mechanical reliability are also presented in Figure 13 (b). The graph shows that all the experimental mechanical reliability aligned with the modeled reliability, which authenticate the design.

Equation presented the mathematical model of the mechanical reliability.

Mechanical reliability = $0.865 + 0.000045A + 0.00305B$ (4)

4.0 Conclusion And Recommendations

This study for the first time reported the mechanical reliability assisted by graphical and mathematical modeling of HAp. The HAp powdery sample employed was characterized with SEM/EDS analysis. The results reflected the characteristics of HAp and also revealed the synthesis route used through its irregular morphology. The Two-parameter Weibull distribution analysis was conducted on HAp hardness and compressive strength. The characteristic hardness and compressive strength, coupled with their corresponding bounds, failure rates and correlation coefficients have been presented in in this article. The Weibull analysis when assisted with modeling revealed HAp fabricated under 10 KN and sintered at 1100 °C as the most reliable sample under hardness condition, while HAp fabricated under 15 KN compaction load and sintered at 1000 °C gave the most reliable characteristic under compression. Essentially, the study conducted on the overall mechanical reliability showed that HAp fabricated under 15 KN compaction and sintered at 1100 °C gave the best multiple mechanical reliability.

Future study has employed the mechanical reliability under hardness and compressive strength. Future study is recommended to be conducted on other essential mechanical properties for biomedical applications. Biological reliability is also important to be investigated.

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Figures

Figure 1

Figure 2

Captured HAp sample area and resultant EDS

Figure 3

SEM images at different magnifications; A: 100 μm , B: 20 μm , and C: 10 μm

Figure 4

Two-parameter Weibull plot and characteristic hardness value of HAp for different experimental runs

Figure 5

Effect of fabrication conditions on hardness reliability (correlation coefficient): (a) Sintering temperature; (b) Compaction load

Figure 6

Fabrication parameters' interaction on hardness reliability

Figure 7

Comparison of experimental and modeled hardness reliability

Figure 8

Two-parameter Weibull plot and characteristic compressive strength of HAp for different experimental runs

Figure 9

Effect of fabrication conditions on compressive strength reliability (correlation coefficient): (a) Sintering temperature; (b) Compaction load

Figure 10

Fabrication parameters' interaction on compressive strength reliability

Figure 11

Comparison of experimental and modeled compressive strength reliability

Figure 12

Effect of fabrication conditions on mechanical reliability (average correlation coefficient): (a) Sintering temperature; (b) Compaction load

Figure 13

The mechanical reliability model; (a) Fabrication parameters' interaction on mechanical reliability (b) Comparison of experimental and modeled mechanical reliability