Effect of forming condition on compressive strength of hydroxyapatite-bioactive glass compact rod

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Abstract. With rising concerns regarding alloy implants, alternative biomaterials are currently studied, to avoid the adverse effects of metal on the human body such as irritation and inflammation. Hydroxyapatite (HA) and Bioactive glass (BG) are two bio-ceramics, which have been implemented in medical applications such as bone implants and fixation parts due to their biocompatibility and close resemblance to the mineralized phase of human bone. Furthermore, these materials can be synthesized from natural sources. In this study, M8 screws rod which are commonly implemented for bone fixation was selected as a case study to investigate an effect of forming conditions on the mechanical property of the composite structure. HA and BG were synthesized and formed into a composite specimen using a hydraulic pressing machine. Full factorial experimental design was employed to solve for an optimal forming condition. 3 factors consisting of mixing ratio, pressure and holding time for pressing were investigated for their impacts on the specimen's compressive strength. The result revealed that the BG ratio and pressure have a significant effect on the structural strength. The maximum compressive strength of 32.20 MPa was obtained from the specimen with 7.5 wt% of BG, 120 kg/cm^2 of pressure and 30 second of holding time.

1 Introduction

Nowadays, fixation devices or artificial human organ implants often contain metal parts, due to its superior mechanical properties. However, much literature has illustrated the adverse effects of metal implants on the human body such as irritation and inflammation [1-2]. Thus, investigation on biomaterials for medical part fabrication is a relevant area of research [3-4]. HA and BG are biomaterials that have been widely investigated for their potential use in medical implant and fixation parts since their composition and biocompatibility are close to the mineralized phase of human bones [5-7]. HA is currently used in many medical and dental applications such as orthopedic implants and implant coatings because HA has high biocompatibility and osteoconductivitiy [8-11]. Nonetheless, the disadvantage of HA is its low elasticity, which becomes a problem in load-bearing applications [12]. Therefore, reinforced biomaterials are being investigated; HA-composites may be developed for medical application to improve its characteristics in terms of mechanical strength, as well as other relevant physical and biological properties. BG is one of alternative reinforced material which improves on the mechanical performance of HA [13-15]. In this study, the investigation is focused on the development of a BG-HA composite bone fixation screw because this screw is frequently used as a basis for qualitative comparison [16-19]. Thus, the purpose of this research is to investigate the effect of forming conditions on the compressive property of BG-HA compact rod using factorial design as a technique for statistical evaluation and optimization of these parameters.

2 Methodology

2.1. Preparation of materials

For this study, 2 types of biomaterials were synthesis in the research center. HA was synthesized from cattle bone, while BG was partially synthesized from mollusc shell. For preparation of HA powder, a cattle bone was cut and cleaned by hydrogen peroxide (H_2O_2) solution to eliminate organic substances and collagen. Then, its moisture was reduced by drying in an oven at 120 °C for 7 days. Finally, it was calcined at 800 °C for 3 hr, and ground into powder with an average size of 37 µm. In the meantime, BG with the composition of 45 wt% SiO_2, 24.5 wt% CaO, 24.5 wt% Na_2O, and 6 wt% P_2O_5 [20], which CaO was prepared using mollusc shell as the raw material. This CaO was thoroughly mixed with other chemical reagents and poured into a crucible. Then, it was heated at 1,100 °C for 2.5 hr to generate glass phase [21]. Finally, the glass was ground into powder with an average size of 17.58 µm.

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2.2. Fabrication of HA/BG compact specimen

To investigate the effect of mixing ratio, various HA and BG mixtures were prepared, and then uniaxially pressed on a hydraulic pressing machine, as shown in Figure 1(a). The specimen dimension was conformed to ASTM F1044-05 (2011). Then, a specimen with 8-mm diameter and 50-mm long compact rods (Figure 1(b)) were sintered in an electrical furnace at 1,200 °C for 3 hr.

(a) **(b)**
Fig. 1. (a) Hydraulic pressing machine (b) compact specimen

2.3 Experimental Setup

The effects of mixing ratio and pressing condition were investigated using full factorial design with center point. Table 1 describes the experimental parameters and boundary conditions, consisting of BG mixing ratio, pressure and time, for a total of 12 experimental conditions (2 replications). The specimen for each condition will be further characterized to investigate the relationships among these parameters based on statistical evaluation.

Table 1. Experimental parameters

Factors	**Level**		**Unit**
	Low (-)	**High (+)**	
1. BG mixing ratio	5.0	7.5	wt%
2. Pressure	100	120	kg/cm^2
3. Time	30	60	Second

2.4 Evaluation of Mechanical Property

The compressive strength of specimen was tested on a Instron 5566 universal testing machine with a setup of crosshead speed at 0.5 mm/min and 10 kN load cell. The test was evaluated based on the load to failure of the cylindrical specimens, with dimensions of 8 mm. and 5 mm. in height, as shown in Figure 2.

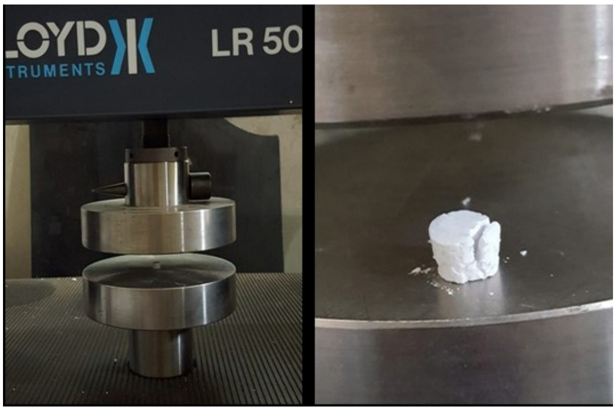

Fig. 2. Compression test on universal testing machine

3 Results and Discussion

The compressive strength results of the HA-BG specimen based on the experimental design is illustrated in Table 2. The full factorial design with center point was generated by MINITAB 16 software to investigate the effect of 3 forming conditions on the mechanical property of the specimen. Statistical evaluation in terms of the significant effect of each parameter and its interaction was performed using Analysis of Variance (ANOVA) at 95% confidence interval, as shown in Table 3. In addition, R-squared and adjusted R-squared statistics were analysed to specify the extent to which fitted models could explain the variability of the results.

Table 2. Experimental results

No.	Factors			Compressive Strength (MPa)
	BG (wt%)	Pressure (Kg/cm^2)	Time (Sec)	
1	5	100	30	14.02
2	7.5	100	30	25.99
3	5	120	30	12.43
4	7.5	120	30	29.81
5	5	100	60	14.97
6	7.5	100	60	27.18
7	5	120	60	19.21
8	7.5	120	60	31.08
9	5	100	30	14.50
10	7.5	100	30	27.09
11	5	120	30	19.51
12	7.5	120	30	28.93
13	5	100	60	16.82
14	7.5	100	60	26.21
15	5	120	60	18.02
16	7.5	120	60	27.82
17	6.25	110	45	22.65
18	6.25	110	45	25.00

Table 3. Regression coefficients and ANOVA

Term	Coefficients	T	P
Constant	22.1000	43.53	0.000
BG	5.9150	11.65	0.000
PRESS	1.2516	2.47	0.036
TIME	0.5655	1.11	0.294
BG*PRESS	0.1454	0.29	0.781
BG*TIME	-0.5059	-1.00	0.345
PRESS*TIME	0.1156	0.23	0.825
BG*PRESS*TIME	-0.1355	-0.27	0.796
R-Sq = 94.18%	R-Sq (adj.) = 89.00%		

According to the ANOVA, the parameters that influence the compressive strength of the HA-BG specimen were the BG ratio and forming pressure and. Hence, the regression model of rod specimen compressive strength (Y) based on significant factors can be described as equation 1.

$$Y = 22.1+5.915(BG)+1.2516(PRESS) \qquad (1)$$

The validation of experimental results in terms of compressive strength's residual plot is shown in Figure 3. This normal probability plot was evaluated to evaluate the fitted distribution, estimate percentiles, and correlate different sample distributions. Since the fitted distribution, the relation of the theoretical and sample percentiles, form a straight line, it can be concluded that our experimental results were normally distributed. In the meantime, the surface and contour plot, as illustrated in Figure 4 and 5 show the effect of both forming parameters on the compressive strength of the HA-BG specimen. After analyzing with the response optimizer function, the optimal conditions within the experimental boundary was found at 120 kg/cm^2 of pressure, 7.5 wt% of BG ratio which yields the highest compressive strength of 29.95 MPa (as shown in Figure 6). It is worth mention that pressing time and parameter interaction have no significant effect on the compressive strength. Consequently, the obtained optimal condition was validated by performing another set of experimental setup (with 10 replications). The average compressive strength from confirmation tests was 32.20 MPa, which found to be not significantly different from the previous evaluation (p-value = 0.108).

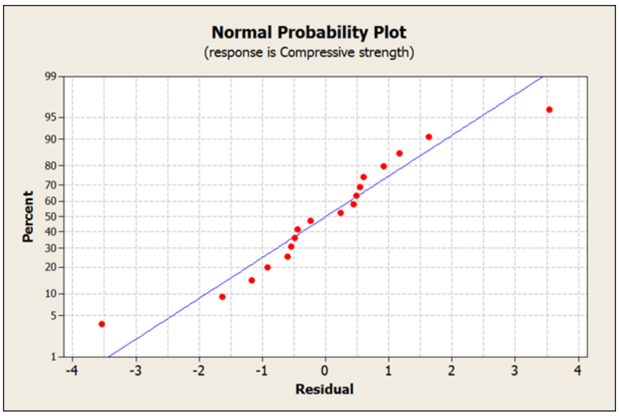

Fig. 3. Normal probability plot of compressive strength

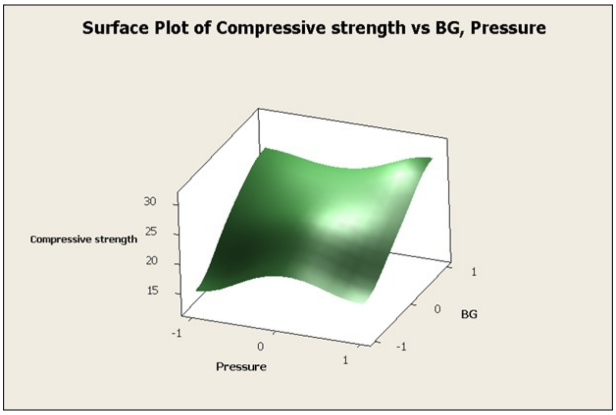

Fig. 4. Response surface of compressive strength

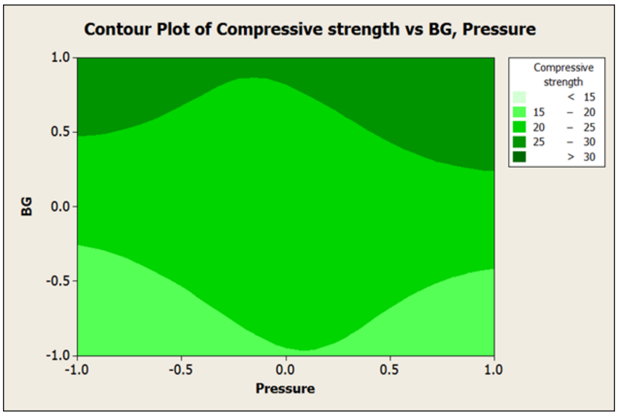

Fig. 5. Contour plot of compressive strength

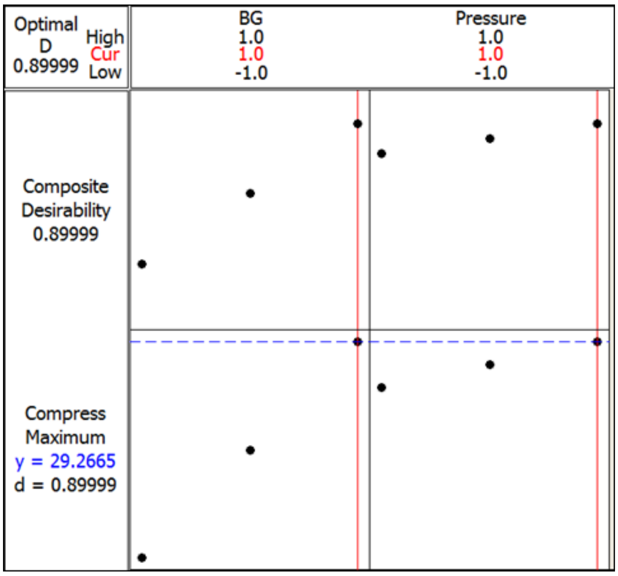

Fig. 6. Optimal condition of compressive strength for experimental boundary

According to the experimental results, the compressive strength of this rod-type specimen was greater at the upper boundary conditions, which could relate to the formation of crystalline phase [22]. Furthermore, this study revealed that the compressive strength of the rod-type structure is significantly lower than bar-like specimen at the similar experimental setup of the former investigation [15]. Because during fabrication of the rod-type specimen, the friction between the side of model and the mold was proportional to the pressure applied during forming process. Too much forming pressure could lead to difficulty in specimen removal. Nonetheless, this present study shows that rod-type structure fabricated by compression molding technique still has superior mechanical properties than 3D printed techniques [11].

4 Conclusion

The formation condition of rod-type HA-BG compact was investigated. The effect of forming conditions on compressive strength was analysed and optimized using full factorial experimental design. The optimal condition of 32.20 MPA compressive strength was found at 7.5 wt% BG ration, with a pressure of 120 kg/cm^2, while holding time has no significant effect on the strength. Further study will focus on other mechanical and biological properties both *in vitro* and *in vivo* prior to the development of bio-based artificial screw for medical application.

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References

1. D. C. Hansen., Elec. Soc. Int. **17**, 2 (2008)
2. L. Chenglong, Y. Dazhi, L. Guoqiang, Q. Min, Mat. Let. **59**, (2005)
3. S. Bauer, P. Schmuki, K. von der Mark, J. Park., Prog- in Mat- Sc-, **58**, 3, '2013(
4. S. R. Paital, N. B. Dahotre, Mat- Sc- & Eng- R66, '2009(
5. M. N. Rahaman, D. E. Day, B. S. Bal, Q. Fu, S.B. Jung, L.F. Bonewald, A. P. Tomsia, Acta bio, **7**+6, '2011(.
6. P. D. Silva, M. H., Lemos, A. F. Ferreira, J. M. F. and J. D. Santos, Mat- Res-+6, 3+'2003(
7. O. Bretcanu, X. Chatzistavrou, K. Paraskevopoulos, R. Conradt, I. Thompson, A. R. Boccaccini, J- Euro-Cer-Soc-, **29**, (2009)
8. S. Pal, *Design of Artificial Human Joint & Organs*, (Springer, 2013)
9. Y. W. Gu, N. H. Loh, K. A. Khor, S. B. Tor and P. Cheang, Biomat., **23**, (2002)
10. W. Wattanutchariya and W. Changkowchai, Lec. Notes Eng. & Com. Sc., 2210, (2014)
11. L. H. Hernández-Gómez, A. I. Rangel-Elizalde, J. A. Beltrán-Fernández, A. González-Rebatú, N. Corro-Valdez, G. Urriolagoitia-Calderón, R. Rodríguez-Martínez. App. of Com. Tool. in Biosc. and Med. Eng., 43-59, (2015)
12. H. B. Guo, X. Miao, Y. Chen, P. Cheang and K. A. Khor, Mat. Let., **58**, (2004)
13. R. Ravarian, F. Moztarzadeh, M. S. Hashjin, S. M. Rabiee, P. Khoshakhlagh and M. Tahriri, Cer. Int., **36**, (2010)
14. W. Wattanutchariya and P. Yenbut, Adv. Mat. Res., 931-932, (2014)
15. W. Wattanutchariya, J. Ruennareenard, P. Suttakul, Eng. J., **20**, (2016)
16. A. Gupta, C. Lattermann, M. Busam, A. Riff, Amer. J. of Sp. Med., **37**, (2009)
17. R. M. Felfel, I. Ahmed, A.J. Parsons, C.D. Rudd, J. Me. Beh. of Bio. Mat., **18**, (2013)
18. L. Handolin, T. Pohjonen, E.K. Partio, I. Arnala., P. Törmäläb, P. Rokkanena, Biomat., **23**, (2002)
19. J. A. Rano, R.T. Savoy-Moore, L.M. Fallat, J. Foot & Ank. Sur., **41**, (2002)
20. L. L. Hench and J. Wilson, *An Introduction to bioceramics*, (World Scientific, 1993)
21. X. Chatzistavrou, T. Zorba, E. Kontonasaki, K. Chrissafis and P. Koidis, Phy. St. Sol.(a), **201**, (2004)
22. Q. Z. Chen, J. L. Xu, L. G. Yu, X. Y. Fang and K. A. Khor, Mat. Sc. & Eng., **C32**, (2012)