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Taguchi Grey Relational Optimization of the Multi-mechanical Characteristics of Kaolin Reinforced Hydroxyapatite: Effect of Fabrication Parameters

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Abstract: Comparative study of kaolin reinforced hydroxyapatite (KHAp) and pure HAp using different production parameters has been done through traditional experimentation. However, the quantitative effect, optimization of kaolin reinforcement and fabrication parameters have not been investigated. Hence, this study examines the effect of kaolin reinforcement, compaction pressure and sintering temperature on the experimental mechanical properties of HAp. Taguchi design assisted by grey relational analysis was employed with L36 (2**2 3**1) orthogonal array. The Minitab 16 software was used to analyze the Taguchi design. The result showed a disparity in kaolin reinforcement as the optimum condition for individual mechanical properties, but the grey relational analysis showed better mechanical properties with kaolin reinforcement, 500 Pa compaction pressure and 1100 ^{o}C sintering temperature. The obtained result further revealed kaolin reinforcement as a strong and promising reinforcing material for high strength clinical application, having a contribution of 93.16% on compressive strength of HAp. Therefore, future studies can be conducted in the use of different wt% of kaolin on the multi-response mechanical characteristics of HAp.

Keywords: Grey relational analysis; optimization; hydroxyapatite; kaolin; production parameters; mechanical properties

1. Introduction

Hydroxyapatite (HAp) with the chemical formula of Ca10(PO4)6(OH)2 is a calcium phosphate based bio-ceramic because it forms a mechanically strong bond to bone and typically no fibrous particles are found inherent on the implants' surface. These attractive properties make HAp a good material in clinical applications such as bio-medical implants and substitutes for the repair of damaged bones (Caliman *et al.*, 2017; Adeogun *et al.*, 2018). The constant use of HAp is due to its unique chemical composition, in addition to its biological and crystallographic similarity with the mineral portion of hard tissues, for instance, bones and teeth. HAp has received attention as a good candidate for biomedical application, due to its excellent biocompatibility and high rate of cell proliferation. Because of its poor mechanical strength, extensive research has been geared towards the improvement of HAp mechanical properties. The use of foreign material as a reinforcing agent has been reported in the literature (Santos *et al.*, 1994; Lahiri *et al.*, 2012; Zhao *et al.*, 2018; Singh *et al.*, 2020; Singh *et al.*, 2021). Reinforcement is an action or process of reinforcing or strengthening a weak material.

Taguchi design of the experiment is a method of mitigating laboratory robustness and also a way of optimizing design parameters or process parameters of singular response of a product or system. The design was named after the Japanese quality guru Genichi Taguchi who invented it (Taguchi & Phadke, 1989; Taguchi, 1993; Taguchi *et al.*, 2005). While Grey Relational Analysis (GRA) is employed when there is a need to optimize design parameters for multiple response characteristics. It is also used when a process or design is uncertain or complicated (Julong, 1989; Javed, 2019). This study employed Taguchi design assisted by the GRA to mitigate the indecision on the best combination of fabrication parameters for better mechanical integrity of HAp. Taguchi-grey relational analysis has been usedare as follows: Sylajakumari *et al.* (2018) optimized production parameters on the multi-wear responses of a co-continuous composite with the help of Taguchi-grey relational analysis. Bademlioglu *et al.* (2020) employed Taguchi grey relational analysis to investigate and to optimize the working parameters affecting the multiple performance characteristics of an organic Rankine cycles. Almetwally (2020) carried out multi-objective optimization of woven fabric parameters using Taguchi-grey relational analysis.

In light of the synthesis and mechanical improvement of hydroxyapatite, Abifarin *et al.* (2019) synthesized and characterized pure HAp for biomedical application. The mechanical properties of the synthesized HAp were further worked upon by Obada *et al.* (2020) and Obada *et al.* (2021) using 15 wt% kaolin and different sintering parameters. Recently, Abifarin (2021) employed Taguchi grey relational analysis to determine and to optimize quantitatively the effect of sintering parameters on pure HAp. Kaolin was employed as a reinforcement in the bulk HAp because it is a silica based materials with excellent biocompatibility (Obada *et al.*, 2021). The reinforcement of HAp with kaolin in this study is referred to as kaolin reinforced HAp. Having reported traditional experimentation of 15 wt% kaolin reinforcement and the effect of compaction pressure and sintering temperature on the mechanical properties of HAp, it is expedient to examine the quantitative effect and the optimization of fabrication parameters of kaolin reinforced HAp. Hence, this study employed Taguchi grey relational analysis as the statistical tool to investigate the optimum production parameters and its quantitative effect on the mechanical properties of HAp. Table 1 describes the employed based materials and its fabrication parameters.

Table 1. Definitions of base materials and fabrication parameters

Variable	Definition	Reference
Hydroxyapatite	Hydroxyapatite is a calcium phosphate based ceramic, having chemical formula of $Ca_{10}(PO_4)_6(OH)_2$	Posner *et al.* (1958); Orlovskii *et al.* (2002)
Kaolin	Kaolin is a silica based material with a chemical formula of Al_2O_3 $2SiO_2$	Murray (1980); Chen *et al.* (1997); Schroeder and Erickson (2014)
Reinforcement	Reinforcement is an action or process of reinforcing or strengthening a weaker material.	Seward (1956); Byrne and Clore (1970)
Kaolin reinforced hydroxyapatite	Kaolin reinforced hydroxyapatite is a composite showing hydroxyapatite as the main matrix, strengthened by kaolin	The present work
Sintering temperature	It is the annealing temperature at which material mechanical and microstructural properties are improved	Kuang *et al.* (1997); Abifarin (2021)
Compaction pressure	It is the pressure at which a material scaffold is formed or made	German (2010); Abifarin (2021)

2. Materials and Method

HAp/KHAp synthesis, characterization and mechanical properties evaluation have been reported in the previous study (Obada *et al.*, 2021). This study employed statistical Taguchi grey relational analysis to examine the quantitative effect of kaolin reinforcement and production parameters (compaction pressure and sintering temperature) on the reported experimental hardness and compressive strength. Figure 1 shows the overall experimental procedure on how the mechanical properties were obtained in the study of Obada *et al.* (2021).

2.1 Taguchi experimental design

The factors and their respective levels were employed based on the design consideration based on the previous study (Obada *et al.*, 2021), and was formulated using Taguchi design strategy as shown in Table 2. The suitable orthogonal array employed according to Minitab 16 software was L36 (2**2 3**1), and it is displayed in Table 3. The corresponding experimental hardness, compressive strength and resultant grey relational grade were analyzed using Taguchi on Minitab. The steps for generating the resultant grey relational grade for the experimental mechanical properties are shown in section 2.2, and are similar to the work of Abifarin (2021) and Awodi *et al.* (2021). All the plotted graphs were obtained using Minitab. Figure 2 shows the overview of Taguchi-grey relational optimization analysis.

2.2 Grey relational analysis

Integration of GRA into the Taguchi method can improve the performance of Taguchi method for optimization (Chang *et al.*, 2000). As it is impossible to directly average experimental hardness and compressive strength, grey relational analysis was employed to address the impossibility (Julong, 1989; Javed *et al.*, 2019). First, hardness and compressive strength values were converted to grey relational generation (normalizing the sequence) using the larger-the-better consideration as shown in Equation 1. The larger-the-better was employed because high hardness and compressive strength is desired. After sequence normalization, deviation sequence of the reference sequence was computed using Equation 2. Next, grey relational coefficient was generated using Equation 3, and thereafter the resultant hardness and compressive strength grey relational coefficients were average to have grey relational grade (GRG) using Equation 4.

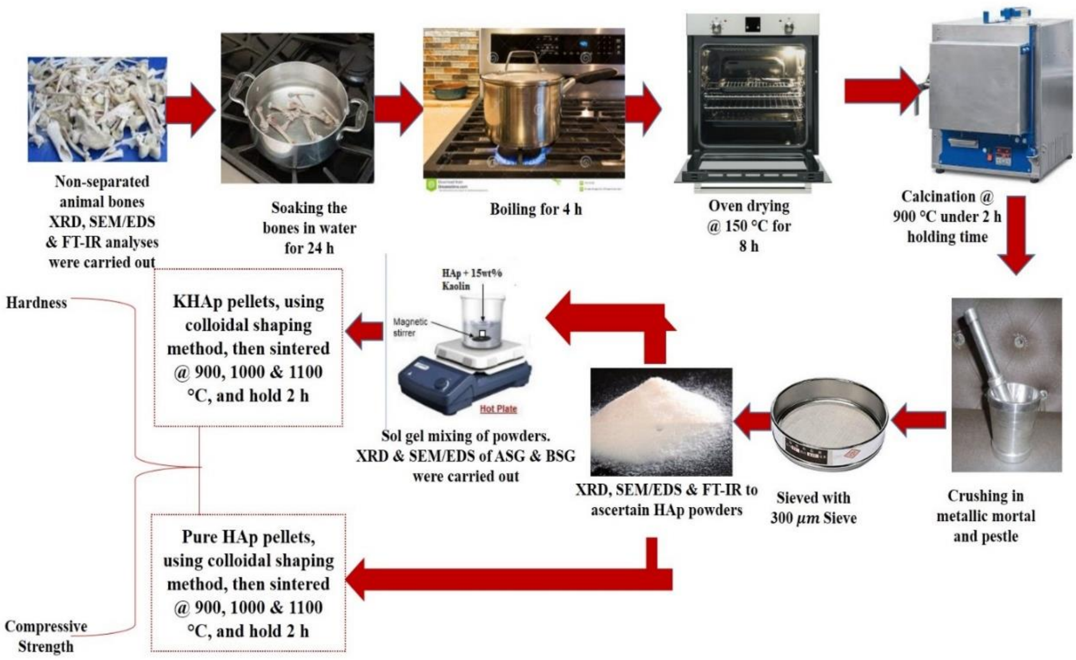

Figure 1. Mechanical synthesis of pure and kaolin reinforced hydroxyapatite

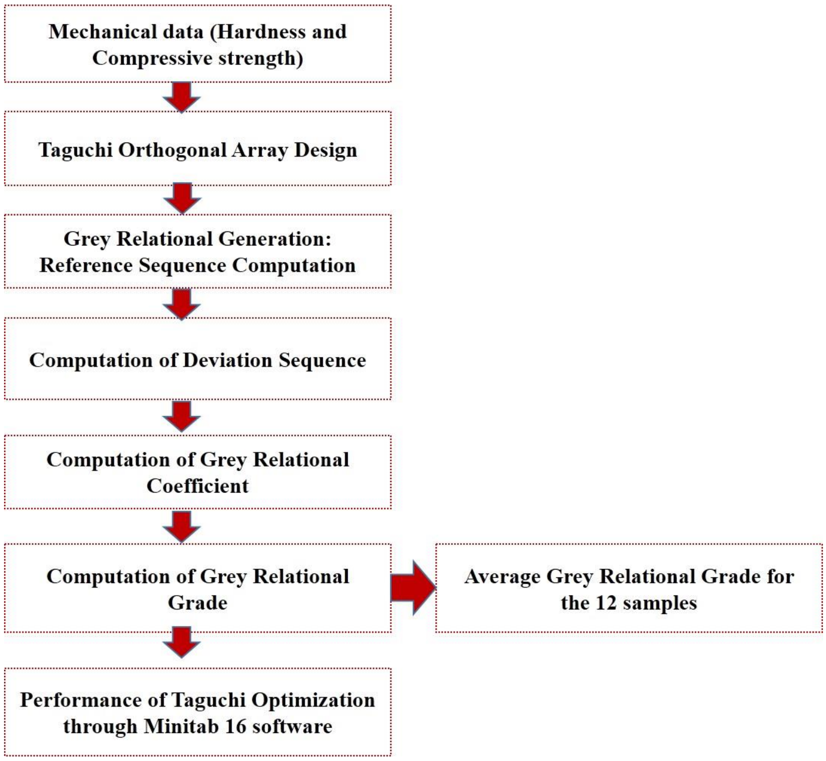

Figure 2. Taguchi-grey relational optimization analysis

$$x_i(k) = \frac{y_i(k) - \min y_i(k)}{max\ y_i(k) - \min y_i(k)} \quad (1)$$

Note that $x_i(k)$ is the normalized data for the i^{th} experiment, and $y_i(k)$ denotes the initial sequence of the mean of the responses

$$\Delta_{0i}(k) = |x_0(k) - x_i(k)| \quad (2)$$

Here, $\Delta_{0i}(k)$, $x_0(k)$, and $x_i(k)$ are the deviation, reference and comparability sequences respectively.

$$\xi_i(k) = \frac{\Delta_{min} + \zeta\Delta_{max}}{\Delta_{0i}(k) + \zeta\Delta_{max}}, \zeta \in (0,1) \quad (3)$$

where $\xi_i(k)$ symbolizes GRC of individual response variables calculated as a function of Δ_{min} and Δ_{max}, the minimum and maximum deviations of each response variable. ζ is the distinguishing coefficient (Mahmoudi *et al.*, 2020) whose value was considered 0.5 in the current study.

$$\gamma_i = \frac{1}{n}\sum_{i=1}^{n} \xi_i(k) \quad (4)$$

where γ_i is the GRG determined for the i^{th} experiment, *n* is the aggregate count of the performance characteristics. Since the Taguchi orthogonal array design displayed three replications for the experimental run, the average grey relational grade (AGRG) for each treatment (sample) was computed afterwards.

Table 2. Factors and their levels

Fabrication parameter	Wt% kaolin	Compaction pressure(Pa)	Sintering temperature (°C)
Level 1	0	0	900
Level 2	15	500	1000
Level 3	-	-	1100

Table 3. Taguchi experimental design strategy

Experimental no.	Wt% kaolin	Compaction pressure (Pa)	Sintering temperature (°C)
1	0	0	900
2	0	0	1000
3	0	0	1100
4	0	0	900
5	0	0	1000
6	0	0	1100
7	0	0	900
8	0	0	1000
9	0	0	1100
10	0	500	900
11	0	500	1000
12	0	500	1100
13	0	500	900
14	0	500	1000
15	0	500	1100
16	0	500	900
17	0	500	1000
18	0	500	1100
19	15	0	900
20	15	0	1000
21	15	0	1100
22	15	0	900
23	15	0	1000
24	15	0	1100
25	15	0	900
26	15	0	1000
27	15	0	1100
28	15	500	900
29	15	500	1000
30	15	500	1100
31	15	500	900
32	15	500	1000
33	15	500	1100
34	15	500	900
35	15	500	1000
36	15	500	1100

3. Results and discussion

3.1 Effect of kaolin and production parameters on hardness

Figure 3 shows effect of kaolin, compaction pressure and sintering temperature on HAp hardness value. The result revealed that 15 wt% of kaolin dropped HAp hardness value, but 500 Pa compaction pressure had a little increasing effect on the hardness value. However, increase in sintering temperature had a significant increasing effect on HAp hardness value. Hence, the optimal factors levels for better hardness are HAp without kaolin reinforcement, 500 Pa compaction pressure and 1100 °C sintering temperature.

3.2 Quantitative effect of reinforcement and production parameters on HAp hardness

Statistical ANOVA data of the Taguchi result is highlighted in Table 4. As effect of the considered factors has been discussed in section 3.1, it is important to have their quantitative effect which is displayed in Table 3. The result revealed that when kaolin reinforcement was not employed, there was a significant contribution of 46.04%. The sintering temperature factor shows the most significant contribution of 50.76% on HAp hardness value. Even though, 500 Pa

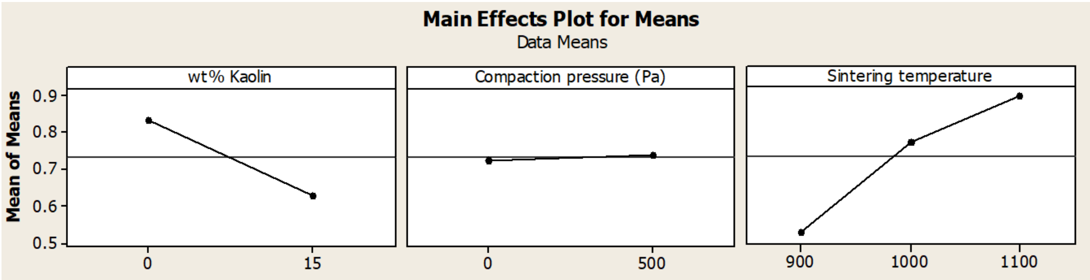

Figure 3. Effect of factors on hardness

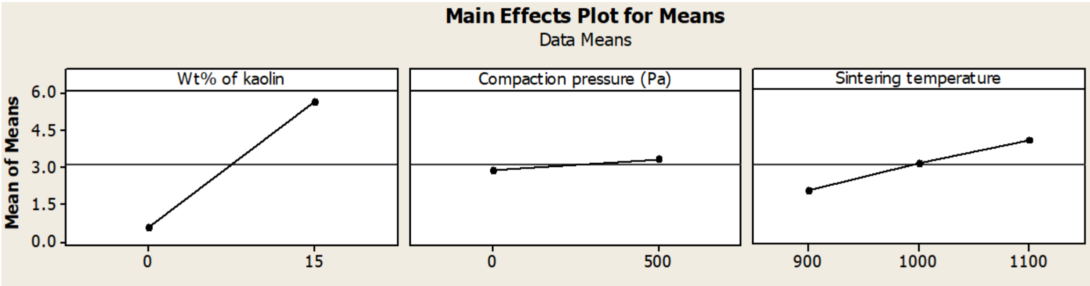

Figure 4. Effect of factors on compressive strength

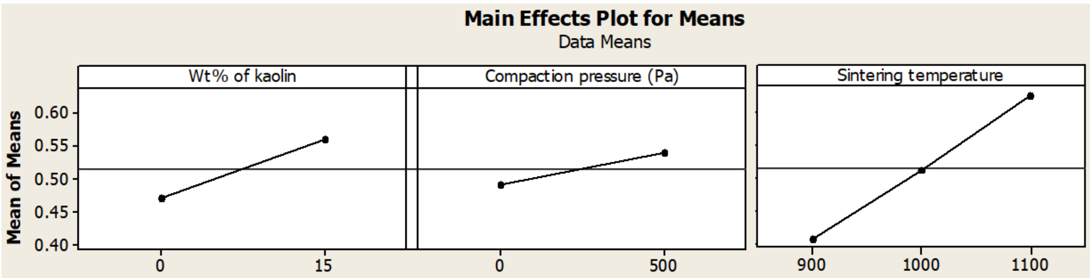

Figure 5. Effect of factors on grey relational grade

compaction pressure had a little increasing effect, its contribution (0.24%) and the contribution of residual error (2.96%) on HAp hardness are insignificant.

3.3 Effect of kaolin and production parameters on compressive strength

It is important to note from the compressive strength result revealed in Figure 4 was significantly increased with inclusion of kaolin. The reason for the increment has been discussed in the previous study (Obada *et al.*, 2021). Equally, this result showed that the two production parameters considered in this study had an increasing effect on the compressive strength of HAp. Meaning, the optimal factors are 15 wt% of kaolin reinforcement, 500 Pa compaction pressure and 1100 ^{o}C sintering temperature.

Table 4. ANOVA for HAp hardness value

Fabrication parameter	DOF	Adj SS	Adj MS	F	Contribution %	Remark
Wt% of kaolin	1	0.128133	0.128133	15.54	46.04	Significant
Compaction pressure	1	0.000655	0.000655	0.08	0.24	Insignificant
Sintering temperature	2	0.282524	0.141262	17.13	50.76	Significant
Residual error	7	0.057720	0.008246		2.96	Insignificant
Total	11		0.278296	S = 0.09081	R^2 = 87.7%	R^2_{Adj} = 80.7%

3.4 Quantitative effect of reinforcement and production parameters on HAp compressive strength

The quantitative effect of addition of kaolin and production parameters are shown in Table 5. Interestingly, other factors did not have much significance on HAp compressive strength except kaolin reinforcement having contribution of 93.16%. Meaning, without the consideration of the production parameters, kaolin reinforcement has a robust increasing effect on the compressive strength of HAp.

3.5 Grey relational evaluation

It is essential to optimize input processing parameters of HAp for its multiple mechanical properties. Grey relational analysis gives conclusive input parameters for high strength HAp. Table 6 and Table 7 highlight the analysis of grey relational grade, while Figure 5 and Table 5 show the effect of kaolin reinforcement, compaction pressure and sintering temperature on the multi-response grey relational grade. It is important to note that, for the multi-response mechanical properties, kaolin reinforcement had an increasing effect on the mechanical properties. Also, compaction pressure, 500 Pa shows a higher increasing effect compared with the individual hardness and compressive strength properties. As the sintering temperature increased, the multi-response mechanical properties increased as it is on individual hardness and compressive strength. Hence, it can be conclusively said that, kaolin reinforcement had a positive impact on the overall mechanical properties of HAp, and the optimal conditions for high strength of HAp are 15 wt% kaolin reinforcement, 500 Pa compaction pressure and 1100 ^{o}C sintering temperature.

Table 7 and Figure 6 show the grey relational grade (GRG) result. The graph in Figure 6 displays experimental number 30, 33, and 36 as the highest GRG, which is also depicted in Table 8. Since the experimental numbers displaying the highest GRG, it was then average to have the mean value of GRG, which gave 0.7445 as the experimental optimum GRG value.

Table 5. ANOVA for HAp Compressive strength

Fabrication parameters	DOF	Adj SS	Adj MS	F	Contribution %	Remark
Wt% of kaolin	1	77.335	77.335	85.92	93.16	Significant
Compaction pressure	1	0.6832	0.6832	0.76	0.82	Insignificant
Sintering temperature	2	8.1961	4.0980	4.55	4.94	Insignificant
Residual error	7	6.3007	0.9001		1.08	Insignificant
Total	11		83.016	S = 0.949	R^2 = 93.2%	R^2_{Adj} = 89.3%

Table 6. Response mean for GRG

Level	Wt% of kaolin	Compaction pressure	Sintering temperature
1	0.4704	0.4911	0.4075
2	0.5585	0.5378	0.5111
3	-	-	0.6248
Delta	0.0882	0.0467	0.2173
Rank	2	3	1
Note: Total mean of GRG: 0.515			

3.6 Quantitative effect of reinforcement and production parameters on HAp GRG

As the qualitative effect has been revealed above, Table 9 reveals the quantitative effect of the controlling factors on GRG values. The result revealed sintering temperature as the most significant production factor having a contribution of 60.21%, followed by kaolin reinforcement with a contribution of 29.72%, and compaction pressure with a contribution of 8.32%. Importantly, the result revealed that the residual error was insignificant on the HAp GRG.

3.7 Confirmation analysis

3.7.1 Confidence interval analysis: After determining the optimal fabrication parameters, its predicted grey relational grade was computed as 0.6911 using Table 5 and Equation 5 (Ross, 1996; Abifarin, 2021; Abifarin *et al.*, 2021).

$$\gamma_{predicted} = \gamma_m + \sum_{i=1}^{q} \gamma_0 - \gamma_m \tag{5}$$

γ_0 highlights the highest GRG response under each fabrication parameter, while γ_m is the total average GRG value. q is the number of fabrication parameters.

To investigate the authenticity of the predicted response and experimental response, confidence interval (CI) was calculated using Equation 6 (Taguchi & Phadke, 1989; Abifarin, 2021; Awodi *et al.*, 2021):

Table 7. Reference and deviation sequence after pre-processing of data

Experiment run	Reference Sequence x_i^*		Deviation Sequence Δ_{0i}	
	HV	CS	HV	CS
1	0.269663	0.00515	0.73034	0.99485
2	0.603371	0.03995	0.39663	0.96005
3	0.730337	0.06443	0.26966	0.93557
4	0.247191	0.01418	0.75281	0.98582
5	0.775281	0.02706	0.22472	0.97294
6	0.689888	0.06959	0.31011	0.93041
7	0.325843	0.01933	0.67416	0.98067
8	0.469663	0.0451	0.53034	0.9549
9	0.77191	0.07861	0.22809	0.92139
10	0.424719	0	0.57528	1
11	0.755056	0.02062	0.24494	0.97938
12	1	0.05284	0	0.94716
13	0.451685	0.01418	0.54831	0.98582
14	0.746067	0.03995	0.25393	0.96005
15	0.88764	0.0451	0.11236	0.9549
16	0.358427	0.00515	0.64157	0.99485
17	0.651685	0.02062	0.34831	0.97938
18	0.853933	0.0567	0.14607	0.9433
19	0.258427	0.37887	0.74157	0.62113
20	0.541573	0.5683	0.45843	0.4317
21	0.396629	0.82603	0.60337	0.17397
22	0.348315	0.38144	0.65169	0.61856
23	0.477528	0.67139	0.52247	0.32861
24	0.5	0.9317	0.5	0.0683
25	0.255056	0.34536	0.74494	0.65464
26	0.460674	0.65851	0.53933	0.34149
27	0.651685	0.82861	0.34831	0.17139
28	0.105618	0.54768	0.89438	0.45232
29	0.364045	0.86856	0.63596	0.13144
30	0.606742	0.9317	0.39326	0.0683
31	0	0.47552	1	0.52448
32	0.320225	0.71005	0.67978	0.28995
33	0.503371	0.9884	0.49663	0.0116
34	0.149438	0.55799	0.85056	0.44201
35	0.304494	0.72552	0.69551	0.27448
36	0.588764	1	0.41124	0

$$CI = \sqrt{F_\alpha(1, f_e)V_e\left[\frac{1}{\eta_{eff}} + \frac{1}{R}\right]} \quad (6)$$

$F_\alpha(1, f_e)$ is the required F ratio for risk, α; f_e is the degree of freedom (DOF) of error; V_e is variance of error; η_{eff} is effective number of replications. If *R* represents the number of experimental repetitions when the investigation is done for affirmation and *N* represents all the experiments carried out then η_{eff} is given by:

$$\eta_{eff} = \frac{N}{1 + (total\ DOF\ of\ control\ factors)} \quad (7)$$

Therefore, if V_e= 0.001372; f_e= 7; DOF of all the control factors is 4; *R* is 1; *N* is 36; α= 0.5 under 95% confidence interval (CI), then $F_{0.5}(1,7) = 5.59$ based on the F-statistical table. Consequently, $\eta_{eff} = \frac{36}{1+4} = 7.2$ and $CI = \sqrt{5.59 \times 0.001372\left[\frac{1}{7.2} + \frac{1}{1}\right]} = \pm 0.0935$. 95% of confidence interval for the optimal grey relational grade predicted is given in Equation 8 (Abifarin, 2021),

Table 8. Rank of grey relational coefficient (GRC) and grey relational grade (GRG)

Experiment run	**Grey relational coefficient, $\varepsilon_i(k)$**		**GRG γ_i**
	HV	**CS**	
1	0.40639	0.33448	0.37044
2	0.55764	0.34245	0.45005
3	0.64964	0.34829	0.49896
4	0.3991	0.33651	0.36781
5	0.68992	0.33946	0.51469
6	0.6172	0.34955	0.48337
7	0.42584	0.33768	0.38176
8	0.48528	0.34367	0.41447
9	0.68673	0.35177	0.51925
10	0.46499	0.33333	0.39916
11	0.67119	0.33798	0.50459
12	1	0.3455	0.67275
13	0.47696	0.33651	0.40673
14	0.66319	0.34245	0.50282
15	0.81651	0.34367	0.58009
16	0.43799	0.33448	0.38624
17	0.5894	0.33798	0.46369
18	0.77391	0.34643	0.56017
19	0.40271	0.44598	0.42435
20	0.52169	0.53665	0.52917
21	0.45316	0.74187	0.59752
22	0.43415	0.447	0.44058
23	0.48901	0.60342	0.54622
24	0.5	0.87982	0.68991
25	0.40162	0.43304	0.41733
26	0.48108	0.59418	0.53763
27	0.5894	0.74472	0.66706
28	0.35858	0.52503	0.44181
29	0.44016	0.79184	0.616
30	0.55975	0.87982	0.71978
31	0.33333	0.48805	0.41069
32	0.42381	0.63295	0.52838
33	0.50169	0.97733	0.73951
34	0.37022	0.53078	0.4505
35	0.41823	0.64559	0.53191
36	0.54871	1	0.77435

Table 9. Grey relational grade (GRG), and average grey relational grade (AGRG)

Sample	Experiment run	GRG	AGRG	Sample	Experiment run	GRG	AGRG
Sample 1	1	0.370	0.373	Sample 7	19	0.424	0.427
	4	0.368			22	0.441	
	7	0.382			25	0.417	
Sample 2	2	0.450	0.460	Sample 8	20	0.529	0.538
	5	0.515			23	0.546	
	8	0.414			26	0.538	
Sample 3	3	0.499	0.501	Sample 9	21	0.598	0.652
	6	0.483			24	0.690	
	9	0.519			27	0.667	
Sample 4	10	0.399	0.397	Sample 10	28	0.442	0.434
	13	0.407			31	0.411	
	16	0.386			34	0.451	
Sample 5	11	0.505	0.490	Sample 11	29	0.616	0.559
	14	0.503			32	0.528	
	17	0.464			35	0.532	
Sample 6	12	0.673	0.604	Sample 12	30	0.720	0.745
	15	0.580			33	0.740	
	18	0.560			36	0.774	

Table 10. ANOVA for HAp GRG

Fabrication parameter	DOF	Adj SS	Adj MS	F	Contribution %	Remark
Wt% of kaolin	1	0.023324	0.023324	17.00	29.72	Significant
Compaction pressure	1	0.006530	0.006530	4.76	8.32	Significant
Sintering temperature	2	0.094503	0.047252	34.43	60.21	Significant
Residual error	7	0.009607	0.001372		1.75	Insignificant
Total	11		0.078478	S = 0.03705	R^2 = 92.8%	R^2_{Adj} = 88.7%

$$\gamma_{predicted} - CI < \gamma_{experimental} < \gamma_{predicted} + CI \quad (8)$$

$$0.5976 < \gamma_{experimental} < 0.7846 \quad (9)$$

The CI findings showed that the experimental GRG value of 0.6911 correlates with the predicted optimal GRG value. This affirms the efficacy of the optimal fabrication parameters on the multi-mechanical characteristics of kaolin reinforced hydroxyapatite.

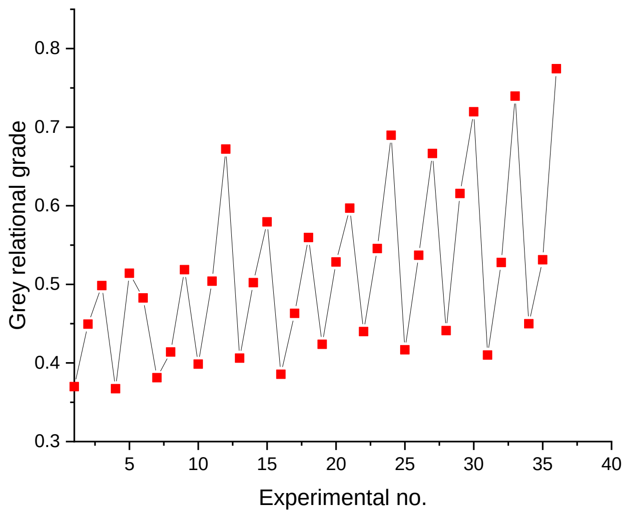

Figure 6. Grey relational grades

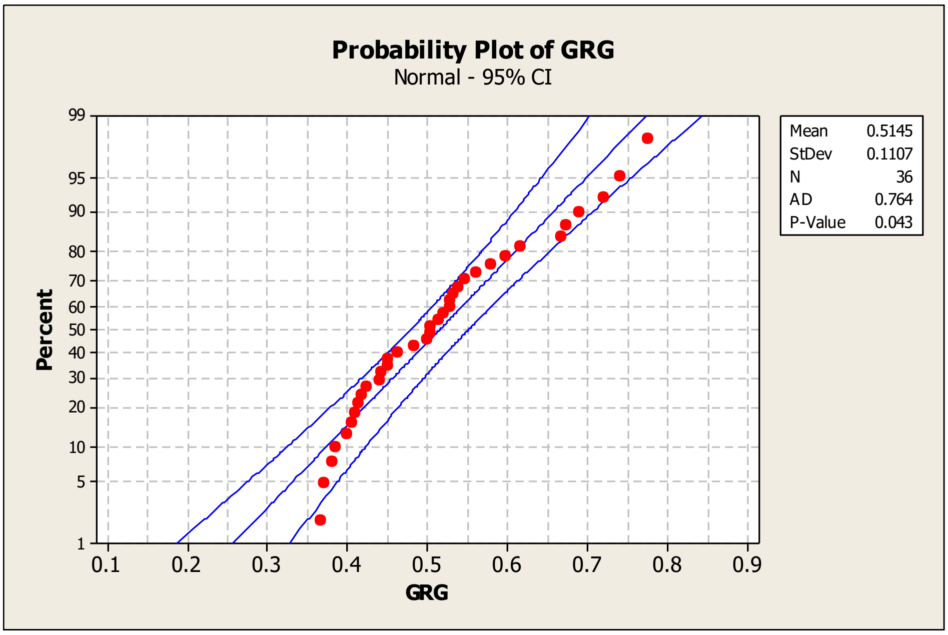

Figure 7. Probability plot of Grey Relational Grades

3.7.2 Probability distribution analysis: Figure 7 shows the probability plot and the statistical information of the multi-mechanical response of kaolin reinforced hydroxyapatite. The plot shows that all the GRG values except one are within the 95% confidence interval, which is supported by the confirmation analysis.

4. Conclusion

The quantitative effect of kaolin reinforcement, compaction pressure and sintering temperature has been examined with the help of statistical analysis technique assisted by grey relational analysis. It was noted that there was disparity in kaolin reinforcement as the optimum condition for individual mechanical properties, showing better hardness but poorer compressive strength when HAp was not reinforced. However, the grey relational analysis showed better mechanical properties with kaolin reinforcement. 500 Pa compaction pressure and 1100 ^{o}C sintering temperature are the optimum fabrication parameters for better mechanical properties, and was the same for individual mechanical properties. It is interesting to note that kaolin reinforcement significantly increased the compressive strength of HAp with a contribution of 93.16%. This influenced GRG values, resulting to 15 wt% kaolin as the optimum with 500 Pa and 1100 ^{o}C sintering temperature as the optimal fabrication parameters for high strength of HAp. The confirmation analysis also revealed that the experimental multi-mechanical response is within the 95% confidence interval.

These findings are useful in orthopedics industry in order to produce a mechanically fitted HAp for load bearing clinical application. These findings recommend fabrication parameters at which mechanically improved clinical hydroxyapatite would be achieved. Further studies are also recommended conducted investigate the use of different wt% of kaolin on the multiple mechanical characteristics of HAp.

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