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TENSILE STRENGTH OPTIMIZATION OF ZIRCONIA BASED DENTAL COMPOSITE USING TAGUCHI METHOD

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ABSTRACT

The dental fillings in the cavity of a diseased tooth are improving by leaps and bound. Nano composite with improved mechanical properties are been invented. Recent composites display improved properties in specific area. There is a need to have a holistic approach to have an improved dental composite with balanced mechanical properties. An attempt has been made to fabricate a dental composite using Zirconia as a filler and Bis-Gma and TEGMA as matrix. Optimization of Tensile strength in the experimental dental composite has been a major challenge, while balancing other mechanical properties. Taguchi's optimization technique keeps the experimentation within limit giving valid results in the domain of tensile strength optimization.

Keywords: tensile strength, taguchi's optimization method, zirconia nanofiller.

1. LITERATURE REVIEW

The mechanical properties of the dental composites are a function of the matrix and filler composition. The volume and weight of the filler is an important parameter that determines the characteristics of the dental composite. Variation in the size, shape and types of filler particles have been attempted to vary and modify the dental composite. Fillers like borosilicate glass, ceramic, silica, prepolymerized resin have been used in sizes varying from (20-150μm^4) to micro fillers of 0.1 μm^4 .This particular study was conducted to analyse the effect of a specific filler zirconia nanocluster along with glass and silica on the mechanical properties of composite in general and tensile strength in particular. The Tensile strength has been optimized for the same experimental dental composite [1].

The dynamic forces involved in mystification present challenges for variation in the parameters to obtain desired results.

In the last decade most studies on dental nanocomposites have shown improvement in the mechanical properties but most of it has been focused on fracture toughness. Fever studies show the influence of particular nanofiller on Tensile strength of dental composites [3].Recently nanohybrid fillers have been introduced. This resulted in improved loading, as the same volume of fillers in the matrix accommodated more nanofiller particles and the particles were more in surface contact with the matrix [4].Consequent there was improvement in the mechanical properties and aesthetic properties without compromising its workability. Contemporary dental composite use silica, glass, quartz either individually or in combination. We have used fillers like silica, glass and zirconia as conglomerate.

2. MATERIAL AND METHODS

The monomer system can be viewed as the backbone of the composite resin system. BisGma (Bisphenol A Glycidyl Methacrylate) and TEGDMA (Triethylene glycol dimehtacrylate) continues to be the most-used monomer for manufacturing present-day composites [3]. Recent studies reveal introduction of zirconia as filler for improvement in mechanical properties. So in addition to conventional fillers namely silica and glass we have added Zirconia nanoparticles as a new filler. Matrix used is combination of the following components:

a) BisGma (Bisphenol A Glycidyl Methacrylate)

b) TEGDMA (Triethylene glycol dimehtacrylate)

We have varied the volume fraction of the three fillers namely: 1) Silica 2) Glass 3) Zirconia.

The fillers used are hybrid with two sets to improve the loading of the resin. The average size of set 1 is 10-100 nm and that of set 2 is 2 -15 micro meters. Hybrid composites contain a broad range of particle sizes. A wide range of particle sizes can lead to high filler loading with resultant high strength. While they may contain a small fraction of filler particles in the nanomer particle size range, they also contain a range of substantially larger filler particles which influences the optical properties of these composites and detracts from polish retention. The dispersion of the fillers in the matrix is uniform, and this is improved when the composite is cured with uv radiation at the time of cavity filling. Fully stabilised zirconia is used with doping levels of 8mol% yttrium, 16mol% magnesia or 16mol% calcium. It has a cubic solid solution and has no phase transformation from room temperature up to 2500 degrees C. Silica and Glass fillers are used in a wide range of dental applications including tooth fillings and tooth building because of its biocompatibility, wear resistance and aesthetics. Important properties of zirconia are exceptional mechanical properties, excellent durability and stability, high biocompatibility and are very resistant to corrosion and scratches. Zirconium Oxide is ivory in colour and matches perfectly with natural tooth, also its light reflection is

better. It is tough, durable and has superior strength and aesthetics [2].

2.1 Preparation of Experimental Composite

The fillers used were zirconia, barium aluminium silicate glass and silica in the volumetric percentages as shown in the Table-1. Nine experimental composites were prepared using the Taguchi's L9 orthogonal array for deciding the filler volume percentage as shown in table 2. The matrix of experimental photo cure composite resins were composed of 2, 2-bis [4-(2-hydroxy-3-methacryloxypropoxy) phenyl] propane (bis-GMA) and triethylene glycol dimethacrylate (TEGDMA) with 2:1 in weight. One percent of 2-(dimethylamino) ethyl methacrylate and 200ppm of 4-methoxyphenol used as a catalyst and an inhibitor respectively were added to the mixture of monomers. A magnetic stirrer was used to agitate the mixture for 3 days, resulting in the homogeneously mixed viscous liquid. 1.0g of the monomer mixture and 0.01g of camphorquinone were mixed mechanically for 1-2min in an automatic mortar. The fillers was gradually added to the monomer mixture and stirred for 15min. After mixing, the mortar was removed from an automatic apparatus, and then the mortar with the experimental composite resin was evacuated for 1min in a vacuum desiccator. Silane treatment was given to the fillers .

2.2 Preparation of Test Specimen for Tensile Strength

A cylindrical test specimen was made using a glass tube of 4mm inner diameter (6mm outer diameter), and 8mm long as mould. Polyester film of 0.1mm thick was used to position each mould. The experimental dental composite was slightly overfilled in the mould and all air bubbles were excluded. The mould was slightly overfilled with the test composite and all air bubbles were excluded. A second piece of polyester film was placed onto the material in the mold and covered with a glass plate, and then pressure was applied gently, thus exuding excess material from the mold. The specimen was irradiated for 15 minutes. The light-cured specimen was removed from its mold and stored in distilled water at 37 degree centigrade. The ASTM Standard adopted was D638 - 14 [1]. The dimensions of specimens were checked using a digital calliper (Digimatic caliper, Mitutoyo Corp., Tokyo,Japan). Test specimen was placed between the surfaces of the compression tool, taking care to align the center line of its long axis with the center line of the plunger and to ensure that the ends of the specimen are parallel with the surface of the compression tool. The loads and corresponding compressive strain at appropriate intervals of strain were recorded. After the yield point was reached, the speed was increased from 5 to 6 mm/min (0.20 to 0.25 in./min) and allowed the machine to run at this speed until the specimen breaks. c for 24hr prior to testing. The cylindrical specimen was tested in a universal testing machine (Make-Autograph) & utilizing a crosshead speed of 1mm/min. Three specimens of each experimental group were tested [5]. The mean value of the three specimens was accepted as an observed Tensile strength of the tested composite resin.

2.3 Taguchi Experiment: Design and Analysis

Traditional experimental design procedures are too complicated and not easy to use. When the number of process parameters increases, a large number of experimental works have to be carried out. Dr. Genichi Taguchi has designed a method which reduces the number of experiments by the use of orthogonal array. The information from the behaviour of the process is obtained from a plan of experiments with the objective of acquiring data in a controlled way. This design of high-quality system called Taguchi's design method reduces efforts of conducting a number of experiments saving time, cost and discovering significant factors quickly. But the main advantage of Taguchi's method is by conducting less number of experiments we get a reliable result and also the higher the value of sum of square of an independent variable, the more it has influence on the performance parameter. One can also calculate the ratio of individual sum of square of a particular independent variable to the total sum of squares of all the variables. This ratio gives the percent contribution of the independent variable on the performance parameter. In addition to above, one could also find the optimal solution to the problem. A statistical analysis of variance (ANOVA) and signal to noise ratio (SN Ratio) can be employed to study the influence of different filler volume percentage on Tensile strength values. Following are the steps followed in Taguchi's optimisation study [7].

a) Select noise and control factors.

b) Select Taguchi orthogonal array.

c) Conduct Experiments and measure the Tensile strength for each of the experimental composites.

d) Analyze results (Signal-to-noise ratio).

e) Predict optimum performance.

f) Confirmation experiment.

2.4 Experimental Procedure

Plan of Experiments:

Taguchi methods is used which include the experiment design theory and the quality loss function concept. The degrees of freedom for three parameters in each of three levels were calculated as follows: Degree of Freedom (DOF) = number of levels -1. For each factor, DOF equal to: For Glass; DOF = 3 – 1 = 2 For Zirconia; DOF = 3 – 1 = 2; For Silica; DOF = 3 - 1 = 2 .Taguchi L9 orthogonal array was used, which has three columns at three levels and nine rows corresponding to the number of tests [7].

Nine experiments were conducted at different parameters in this study. For this L9 orthogonal array has eight DOF, in which 6 were assigned to three factors (each one 2 DOF) and 2 DOF was assigned to the error.

Table-1. Percentage of filler volume at each level.

Process Parameters	Levels		
	Level 1	Level 2	Level 3
Glass %	23.7	25.7	31
Zirconia %	21	27.5	29
Silica %	13.5	16	20

The Tensile strength was tested for each experimental composite with filler volume percentage corresponding to each level. The Tensile strength values corresponding to each experimental composite are shown in Table-2.

Table-2. Taguchi L9 orthogonal array for Tensile strength.

Experimental Composites	Filler Volume levels			Compressive strength (MPa)	Signal to noise Ratio (SN ratio)
	Glass	Zirconia	Silica		
1	1	1	1	249.33	47.94
2	1	2	2	579.83	55.27
3	1	3	3	630.08	55.99
4	2	1	2	231.93	47.31
5	2	2	3	657.14	56.35
6	2	3	1	316.97	50.020
7	3	1	3	230	47.23
8	3	2	1	419.41	52.41
9	3	3	2	589.49	55.41

3. RESULT AND DISCUSSIONS

3.1 Regression Analysis

A mathematical model was developed with the volumetric percentage of the three fillers in the composite namely glass (Barium Aluminium Fluoride), zirconia and silica and its effect on the Tensile strength. The correlation between factors {glass (barium aluminium fluoride), zirconia, Silica}and Tensile strength were obtained by multiple linear regressions [8]. To derive the models, the standard commercial statistical software package MINITAB was used:

$$TS=5268-774G+176Z+264S+8.38G^2-8.48Z^2$$

Analysis of the signal to noise ratio: For the minimization of quality characteristic variation due to uncontrollable parameter the response variation is studied using signal to noise ratio (S/N Ratio) as enumerated in Taguchi's method. In the dental composites larger the Tensile strength will be better so the Tensile strength was considered as the quality characteristic and the concept of "the larger-the-better" [7] was used. The Signal to noise ratio used for this type response is taken as "larger-the-better" and given by the relation: S/N = -10 Log10 $(1/n\Sigma 1/y^2)$

Where y is the measured value in a run/row and n is the number of measurements in a trial/row (n=1 in this case).The equation no 1 was used to calculate the Signal to noise (SN) ratio. The Signal to noise (SN) ratios and the corresponding values of Tensile strength are shown in Table-2. (Equation-1) For the volumetric percentage of Glass, zirconia and silica concentration, the Tensile strength response table and the ranks are shown in Table-3. A greater S/N ratio value will give better performance irrespective of the performance characteristic. The greater S/N ratio value will give the optimum level filler volume percentage [6]. The optimal Tensile strength was obtained at Glass %=23.7(level1), Zirconia %=27.5(level2),

Silica %=20 (level3), based on the analysis of the S/N ratio [8].

The effect of the process parameters on the Tensile strength is shown in Figure-1, which is evaluated using statistical software package MINITAB.

Table-3. S/N ratio values for compressive strength by factor level.

Sr	Input parameter	Level 1	level 2	level 3	Delta (Max-Min)	RANK
1	Glass %	53.06	51.22	51.69	1.83	3
2	Zirconia %	47.49	54.69	53.80	7.19	1
3	Silica %	50.13	52.66	53.19	3.05	2

3.2 Analysis of Variance (ANOVA):

For finding out the differences in the average performance of groups of items tested, a statistical tool named ANOVA which is an objective decision making tool is used. At a specific confidence level the mean square is compared with an estimate of experimental error in ANOVA. The interaction and significance of all the main factors is tested. The following equation is used to find out the total sum of squared deviations SST from the total mean S/N ratio nm $SST=\sum(ni-nm)^2$
i=1

Where, ηi is the mean S/N ratio for the ith experiment and where n is the number of experiments in the orthogonal array. For calculation of P i.e percentage contribution:

$P = SSd/SST$

where SSd is the sum of the squared deviation

Table-4 shows the results of ANOVA. To find out the significance of a factor, a ratio of the mean square error to the residual error, is taken and called as F-ratio named after Fisher. Fisher test is used to study the significant effect on quality characteristic by the design parameters [9]. To study the effect of the filler volume percentage on the Tensile strength, we use the percentage (%) shown in Table-4. From the Table-4, it can be seen that nanofiller glass has 13.46 %, zirconia has 66.83% and silica has 19.71 % influences on the Tensile strength respectively. is the sum of the squared deviations.

Table-4. Analysis of Variance (ANNOVA) results for compressive strength for dental composite.

Source of Variation	Degree of freedom (DOE)	Sum of Squares (S)	Mean sum of the squares (MSS)	F ratio	Percentage (%)
Glass %	2	12636.93	6318.46	←POOL	
Zirconia %	2	176526.04	88263.02	13.97	66.83
Silica %	2	52070.26	26035.13	4.12	19.71
Residual	2	22912.49	11456.24		13.46
Total	8	264145.72			100

3.3 Prediction of the Optimum Results:

For the experimental dental composite we know that for the Tensile strength, higher is the better [7], We have obtained the optimum values of filler volume and from this, the optimum value of Tensile strength is found out by the following relation:

Maximum Optimum Tensile strength=A1+B2+C3 - 2T

A1=Average value of Tensile strength at the first level of glass volume percentage. B2=Average value of Tensile strength at the second level of zirconia volume percentage.

C3=Average value of Tensile strength at the third level of silica volume percentage.

T= Average values of Tensile strength for all the 9 trials.

Maximum Optimum Tensile strength is calculated as 575.68 MPa as shown in Table-5.

Uncertainly analysis has been done with a percent uncertainty of 0.00214137

Table-5. Calculation of optimum maximum tensile strength (MPa).

Averages	Maximum average values of Tensile strength			Average Tensile strength	$A_1+B_2+C_3$	2T	Maximum tensile Strength (MPa)
A1	209.32	559.83	610.08	487.13	1476.74	901.06	575.68
B2	511.83	601.14	401.41	504.79			
C3	599.23	601.12	254.12	484.82			

3.4 Confirmation Test

Taguchi has strongly recommended a confirmation test for verifying the results [7]. The experiments are conducted with the optimum conditions and the Tensile strength is found out. This Tensile strength is compared with that calculated value using the relation

enumerated above. We had obtained optimum filler volume as A1B2C3 and the Tensile strength found out experimentally was 580 MPa.

4. RESULTS AND DISCUSSIONS

a) In our study we have prepared a experimental dental composite. Contemporary dental composites are using glass and silica either individually or in combination as fillers. Zirconia was introduced as nanofiller. We have used three nano fillers namely Zirconia, Glass (Ba-Al-F) and silica in combination (conglomerate) [9].

b) It is observed that the influence of zirconia as a filler material on experimental dental composite is very significant and highest in our study, effect of zirconia on Tensile strength being 66.83 %. Statistical results (at a 95% confidence level) show that the glass, zirconia and silica affect the Tensile strength by 13.46%, 66.83 % and 19.71 % of the experimental dental composites, respectively.

c) Statistically designed experiments based on Taguchi methods were performed using L9 orthogonal arrays to analyze the Tensile strength as response variable. Conceptual S/N ratio and ANOVA approaches for data analysis drew similar conclusions [10].

d) The maximum optimum Tensile strength is calculated as 575.68 MPa by Taguchi's optimization method.

e) In this study, the analysis of the confirmation experiment for Tensile strength has shown that Taguchi parameter design can successfully verify the optimum filler volume percentages (A3B3C3), which are Glass=23.7% (A1), Zirconia = 27.5 % (B2) and Silica =20% (C3).

f) The confirmatory experimental tests results are matching with the optimal value of Tensile strength calculated [11].

g) The compressive properties of the dental composites mainly depend upon the type and size of the filler properties. Many studies have been conducted to investigate the various mechanical properties. The challenge still continues for a improving the mechanical properties of dental composite with a predicted clinical life [12].

5. CONCLUSIONS

It is observed that the influence of zirconia as a filler material on experimental dental composite is very significant and highest in our study. We have used nano particles of fillers in conglomerate; this has increased the loading and improved surface finish of the composite. We have optimized the volume fraction of filler content and the consequent Tensile strength. This will be useful for the composite for supporting compressive and flexural mastication forces more effectively than other restorative material tested.

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