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OPTIMISATION OF COMPRESSIVE STRENGTH IN ZIRCONIA NANOCLUSTERS OF THE BIS-GMA AND TEGDMA BASED DENTAL COMPOSITES

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ABSTRACT

The aim of this work is to present an analysis of the composites and furnish dentists with a basis that can provide criteria for choosing one or another to suit their therapeutic requirements. Mastication is a dynamic process and tooth is an important component of it. Restoration of lost tooth structure is a challenge to the dentist as well as scientists testing the restorative materials. One of the most commonly used restorative materials is composite resin. The mechanical properties of composite resins are mainly due to its filler content. Dental composites are using silica, quartz and glass as filler for long time. Zirconia is the latest addition as filler. Zirconia is super hard because of the peculiar arrangements of atoms. Mastication is a dynamic situation in which many forces are acting in all possible direction, the most important being the compressive force. We wish to study the contribution of Zirconia as filler in the mechanical properties of the dental composite. Our aim is to optimize the compressive strength in Zirconia nanoclusters of the Bis-GMA and TEGDMA based dental composites. The aim of this paper is to evaluate the important role of Zirconia as filler in the compressive strength of dental composite. Contemporary dental composites are using either one or two fillers individually or in combination. Attempt is made to use Zirconia, glass and silica conglomerate. The optimum composition of hybrid fillers and the optimum compressive strength is obtained.

Keywords: zirconia, dental composites, nanoclusters, compressive strength, optimium hybrid fillers, regression.

INTRODUCTION

Most composites used in dentistry are hybrid materials; so-called because they are composed of polymer groups reinforced by an inorganic phase of glass fillers with different compositions, particle sizes and filler percentages. [1,2], The clinical choice of a composite must consider whether priority should be given to mechanical or aesthetic requirements: if mechanical considerations are paramount the material with the greatest volume of filler will be chosen; if aesthetic considerations predominate, particle size will be the most important factor.

The introduction of composite-based resin technology to restorative dentistry was one of the most significant contributions to dentistry in the last century. The advantages of composites include conservation of sound tooth structure, reduction of micro leakage, prevention of postoperative sensitivity, marginal staining and recurrent caries, transmission and distribution of functional stress across the bonding interface to the tooth. Composites also offer the potential for tooth reinforcement, cosmetic restoration and recon touring of teeth[3,4],

It has been about thirty years since composite resins were introduced into dentistry. Fillers varied from relatively large particles (20-150μm^4) in the early stage, to micro fillers of 0.1 μm^4 or less mean diameter, to recent hybrid fillers [5]. More recently [6], the composites comprising high content hybrid fillers were widely used in dentistry because of their superior mechanical properties. The development of dental composite was so strongly aimed at clinical use that many basic problems have been left unsolved [7]. The dental composites consisted of those of inorganic phase and organic phase, and the surface characteristics of the filler were one of the dominant factors [7] over the properties of such materials. Laboratories and manufacturers have treated the filler-matrix interface according to their own original methods, and general consensus regarding silanation of fillers has not yet been obtained. Many reports deal with method of filler surface treatments [8] and obtained significant information about silane treatment and mixing method of fillers, and also contents of the micro fillers [9]. If we apply this information to the filler treatment of the hybrid filler, another problem arises regarding the method of hybridization of fillers. Particularly, what particle sizes would be preferable as far as the hybrid filled composite resin was concerned? The classification and evaluation of commercial composites according to the filler shape and size are reported and indicate various filler systems. Although most studies on composites with hybrid fillers were mainly concerned with the admixing effect of the micro filler, the influence of the size of the filler particles has rarely been studied. The properties of composite resin, i.e., water sorption, polymerization process [10], wear and shrinkage stress, were studied in relation with the particle size of fillers, which consisted of either micro filler or traditional macro filler, but did not include the hybrid system. Moreover, the hybrid system constructed by the mixture of different sizes of macro fillers, or constructed by a mixture of micro fillers and macro fillers, whose sizes were changed systematically, has not yet been studied. Therefore, the purpose of the present study was to investigate the effect of different fillers on the mechanical

properties particularly compressive strength of the composite containing hybrid fillers of the binary system, and to obtain information about a optimum composition of hybrid fillers [11].

The disperse phase of composite resins is made up of an inorganic filler material which, in essence, determines the physical and mechanical properties of the composite. The nature of the filler, how it is obtained and how much is added largely decide the mechanical properties of the restoration material. The filler particles are added to the organic phase to improve the physical and mechanical properties of the organic matrix, so incorporating as high a percentage as possible of filler is a fundamental aim. The filler reduces the thermal expansion coefficient and overall curing shrinkage, provides radio-opacity, improves handling and improves the aesthetic results [11]. The filler particles used vary widely in their chemical composition, morphology and dimensions. The main filler silica, glass and quartz are commonly employed. The search is currently on for materials, which are less hard than glass ones and therefore cause less wear on the opposing tooth [12]. Nanotechnology has led to the development of a new composite resin characterised by containing nanoparticles measuring approximately 25 nm and nanoaggregates of approximately 75 nm, which are made up of zirconium particles. The aggregates are treated with silane so that they bind to the resin. The distribution of the filler (aggregates and nanoparticles) gives a high load, up to 79.5% [13]. As the particle size is smaller, resins made with this type of particle give the restoration a better finish, which is observed in its surface texture, and the likelihood of the material's biodegrading over time is reduced. This technology has also achieved sufficiently competent mechanical properties for the resin to be indicated for use in the anterior and posterior sectors. It should also be mentioned that the lower size of the particles leads to less curing shrinkage, creates less cusp wall deflection and reduces the presence of microfissures in the enamel edges, which are responsible for marginal leakage, colour changes, bacterial penetration and possible post-operative sensitivity [14]. The drawback is that since the particles are so small they do not reflect light, so they are combined with larger-sized particles, with an average diameter within visible light wavelengths (i.e., around or below (1μm), to improve their optical performance and act as a substrate.

MATERIAL AND METHODS

The monomer system can be viewed as the backbone of the composite resin system. Bis-GMA continues to be the most-used monomer for manufacturing present-day composites; whether alone or in conjunction with urethane dimethacrylate, it constitutes around 20% (v/v) of standard composite resin compositions. The resin matrix was a visible-light-cured system composed of BisGma (Bisphenol A Glycidyl Methacrylate) and TEGDMA (Triethylene glycol dimehtacrylate) as matrix and nano filler amounts of percentage volume shown in Table-1. No colouring or fluorescent agents were added. The resin specimens were polymerized by means of the ICI light*. The light output was monitored before and after use by means of a light meter designed to measure the output in the critical wave length range. Minimal acceptable output was established at 1000 W/m^2.The nano hybrid fillers were: silica, glass and Zirconia with varying volume percentage as mentioned in Table-1. All fillers were surface-treated with a coupling agent and incorporated directly into the matrix, in the amounts shown in Table-1, to produce nine filler levels [15].

One more filler which was responsible for improvement in mechanical properties was Zirconia. Presently few commercially available dental composite is using Zirconia nanoclusters. So in addition to silica and glass we have added Zirconia nanoparticles as 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.

Preparation of test specimen for compressive strength

The test specimens for compressive strength and diametral tensile strength of experimental composites were made by the following procedure. A glass tube of 4mm inner diameter (6mm outer diameter), 8mm long was used as a mold for the preparation of a cylindrical test specimen for compressive strength. Each mold was positioned upon polyester film of 0.1mm thick. The mold 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, then pressure was applied gently, thus exuding excess material from the mold. Each of the 3 molds filled with the composite were prepared for compressive strength, and placed in a visible light irradiation apparatus and, irradiated for 15min, then turned upside down, and irradiated for another 15min. The light-cured specimen was removed from its mold and stored in distilled water at 37^{o}C for 24hr prior to testing. The cylindrical specimen was tested in a universal testing machine (Make-Autograph) and utilizing a crosshead speed of 1mm/min. Three specimens of each experimental group were tested. The mean value of the three was accepted as an observed strength of the tested composite resin [14].

The purpose of the study was to study the contribution of different filler particles on the performance of dental composite in general and compressive strength in particular. Experimentation was done and testing of all the leading commercially available brands of Dental composites in the market to investigate their mechanical properties and their filler contents [16].

Procedure adopted for testing compressive strength

The ASTM Standard adopted was D695-08. We measured the width and thickness of the specimen to the nearest 0.01 mm (0.001 in.) at several points along its

length. Calculate and record the minimum value of the cross-sectional area was calculated. We measured the length of the specimen and recorded the value. 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 [17]. The crosshead of the testing machine until it just contacts the top of the compression tool plunger was adjusted. We recorded loads and corresponding compressive strain at appropriate intervals of strain or, if the test machine is equipped with an automatic recording device, record the complete load-deformation curve. After the yield point has been reached, it may be desirable to increase the speed from 5 to 6 mm/min (0.20 to 0.25 in. /min) and allow the machine to run at this speed until the specimen breaks [18].

We have taken three levels of filler volume percentages, low, medium and high. These three levels are shown in Table-1. We have varied the three levels of the volume percentage of silica, glass and Zirconia. By L9 (Taguchi's orthogonal array) we have nine experimental composites. For each trial three readings are taken and the average compressive strength is calculated. The graphs for each filler and compressive strength is plotted keeping the other volume percentage constant, to study the contribution of each filler in the compressive strength. Then we have calculated Regression equation for each mechanical property and Analysis of variance (Table-2) and Sensitivity analysis (Table-3) for each mechanical property, Error calculations and uncertainty analysis [19].

Table-1. Experimental evaluation of compressive strength.

Experimental composites	Filler volume %			Compressive Strength
	ZIRCONIA	GLASS	SILICA	MPa
1	23.7	21	13.5	50
2	23.7	27.5	16	120
3	23.7	29	20	160
4	25.7	21	16	60
5	25.7	27.5	20	320
6	25.7	29	13.5	660
7	31	21	20	299
8	31	27.5	13.5	295
9	31	29	16	297

Table-2. Analysis of variance (ANNOVA).

FACTOR	df	SS	MSS	Fratio		Rho
Zirconia %	2	93446.88889	46723.44444	1.9973733	0.33288106	33.28810597
Glass %	2	83718.22222	41859.11111	1.7894287	0.298225129	29.82251294
Silica%	2	46784.88889	23392.44444	←POOL		
Residual	2	56771.55556	28385.77778			36.88938109
Total	8	280721.5556				100

Table-3. Sensitivity analysis.

Experimental composite	ZIRCONIA %			GLASS %			SILICA %			Comprssive strength (experimental)
	23.7	25.7	31	21	27.5	29	13.5	16	20	
1	50			50			50			50
2	120				120			120		120
3	160					160			160	160
4		60		60				60		60
5		320			320				320	320
6		660				660	660			660
7			299	299					299	299
8			295		295		295			295
9			297			297		297		297
Total	330	1040	891	409	735	1117	1005	477	779	2261
No. of values	3	3	3	3	3	3	3	3	3	9
Mean	110	346.6666667	297	136.3333333	245	372.3333333	335	159	259.6667	251.2222
	XS1	XS2	XS3	XG1	XG2	XG3	XZ1	XZ2	XZ3	

Grand Mean = 251.222

The regression equation for compressive strength is as follows:

$CS = -1018.49 + 18.45M_1 + 24.34\ M_2 + 8.88\ M_3$

Uncertainty analysis

Experimental measurement in the process of finding heat transfer coefficients required many parameters which were measured by various instruments. These instruments were of various degree of accuracy. The measurement in error of these parameters affects the accuracy of the heat transfer coefficients. It is, therefore, of considerable importance to examine the combined effect of uncertainty in each variable. The experimental uncertainty is the absolute value of the maximum expected deviation from the reported experimental result [20].

Schultz and Cole (1979) suggested the following method of analyzing the effect of uncertainty in each variable of the uncertainty of the result:

$$E_R = \left[\sum_{i=1}^{n}\left(\frac{\partial_R}{\partial_{Y_i}}\mathrm{E_{Yi}}\right)^2\right]^{1/2} \quad (1)$$

Where, E_R is the estimate of the uncertainty in the calculated value of the desired variable R, due to the independent uncertainty E_{yi} in the primary measurements of n number of variables, Y_i, affecting the results. Using Equation (1) the experimental uncertainty of heat transfer coefficient is given by:

$$E\sigma = \left[\sum_{i=1}^{n}\left(\frac{\partial\delta}{\partial Pi}\mathrm{EPi}\right)^2\right]^{1/2}$$

Load (Δ P) = ± 0.25 x 5000/100 = ±12.5 N

Area (A) = ± 25 x 2.5 cm^2 = ± 62.5 x 10^{-4} m^2

Stress (σ) = Load (P) / Area (A)

$\pm \Delta\sigma = \pm [(\Delta P\ d\sigma/dP)^2 + (\Delta A\ d\sigma/dA)^2]^{1/2}$
We Know that:
$d\sigma/dp = 1/A$; $d\sigma/dA = P/A^2$

$\Delta P = 50 \times 10^6 \times 62.5 \times 10^{-4} = 312.5 \times 10^3$ N

$\Delta\sigma = [(12.5/62.5 \times 10^4)^2 + (62.5\times10^{-4}\times12.5/62.5\times10^{-4})^2]^{1/2}$
$= 2.83 \times 10^3$

Percentage Uncertainty = σ/ (σ min) x100
$= 2.83 \times 10^3 / (50 \times 10^6) \times 100$
= 0.00566

RESULT AND DISCUSSIONS

The R Square value is well 98.7 % which shows that the regression equation is acceptable (Table-4). If we find out other mechanical properties for the experimental composites namely diametrical tensile strength, flexural strength,elastic modulous, flexural odulous, fracture toughness, surface hardness, micro hardness,compressive strength [21], compressive modulous, polymerisation shrinkage and plot a graph for each composite it is shown in Figure-1.

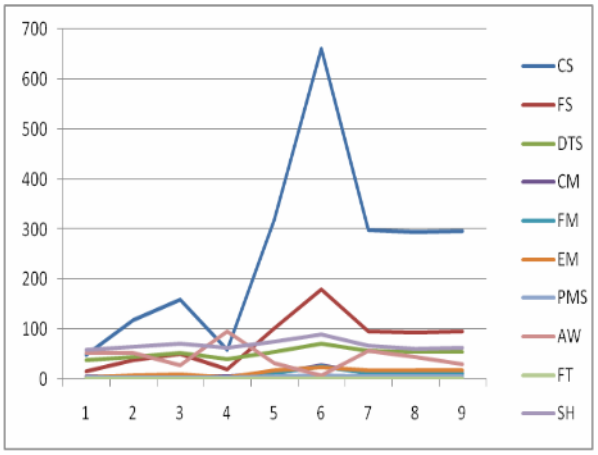

Figure-1. Mechanical properties of experimental composites.

Table-4. Summary output of statistical analysis.

Regression statistics	
Multiple R	0.993487621
R Square	0.987017652
Adjusted R Square	0.974035304
Standard Error	0.059445133
Observations	9

We observe that for experimental composite no.6 all the mechanical properties are acceptable. We can say that the experimental composite number 6 with its filler voulme percentage shown in Table-1 will give better results that the remaining eight experimental composites. Simillarly for experimental composite no. 4 all the mechanical properties are not acceptable. We can say that the experimental composite number 4 with its filler voulme percentage shown in table will give fewer results than that of the remaining eight experimental composites. Error in the difference between predicted value of a parameters calculated by our regression equation and its actually value obtained by conducting experimental comes to 0.38%, which shows the high accuracy of our regression equation. The contribution of Zirconia volume percentage in the compressive strength is 33.29 volume percentages, whereas for silica it is 36.89 volume percentage and glass it is 29.82 volume percentages. This shows that Zirconia volume percentage is a significant factor in the contribution of compressive strength (Figure-2).

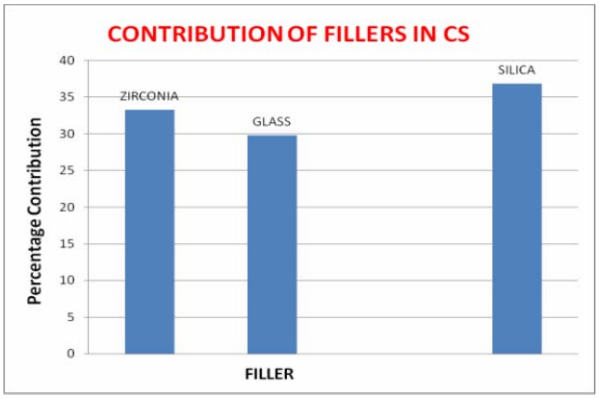

Figure-2. Contribution of filler percentage in compressve strength.

So there is variation in the compressive strength with the increase in the Zirconia volume percentage. This shows that Zirconia is a major contributing factor in the compressive strength of dental composite and with increase in the volume percentage of Zirconia the compressive strength definitely increases [22]. Further, we can find out the exact volume percentage of Zirconia, silica and glass which will give maximum compressive strength. This can be done by using optimisation procedures. Although these data are interesting, the underlying reasons for the variation in the compressive strength with variation in filler is probably quite complex, the result of an interaction of multiple factors associated with the mechanical properties of the resin matrix and the filler particles, including particle size and distribution. The investigators feel it would be unwise to extrapolate from these results to predict the behaviour of other systems. It is obvious that further work is warranted in order to determine the effects of the degree of loading with Zirconia filler, as well as the examination of the role of the Zirconia on other mechanical properties which may be effect as well [23].

Optimal Solution obtained by simplex linear programming model:

Zirconia = 31%
Glass = 29%
Silica =20%
Compressive strength = 1455.41 MPa

A confirmatory experimental test was conducted and the average of three readings came to 1450 MPa. This is matching the optimal value calculated or compressive strength by simplex linear programming model.

CONCLUSIONS

a) Confirmatory test results are matching with the optimal value of compressive strength.
b) Uncertainty analysis of various measuring systems is within the acceptable limits.
c) Uncertainty in the compressive strength measurement is 0.00566%.

d) We have used three fillers namely Zirconia, Glass (Ba-Al-F) in combination (conglomerate). Cotemporary dental composites are using fillers either individually or a combination of two fillers.
e) We have used nano particles of fillers in conglomerate; this has increased the loading and surface finish of the composite.
f) We have optimized the volume fraction of filler content and the consequent compressive strength by simplex linear programming model.

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