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INFLUENCE OF ZIRCONIA NANOCLUSTERS ON THE COMPRESSIVE STRENGTH OF BIS-GMA and TEGDMA BASED DENTAL COMPOSITES

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ABSTRACT

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. 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. Our aim is to prepare an experimental composite with Zirconia as hybrid nanofiller and to study its contribution in the compressive strength of the experimental composite.

Keywords: zirconia, nanoclusters, compressive strength, filler.

INTRODUCTION

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 [1]. More recently [2], the composites containing high content hybrid fillers were widely used in dentistry because of their superior mechanical properties. There are many problems which are unsolved in dental composites [2-3]. Moreover, the hybrid system constructed by the mixture of different macrofillers, or constructed by a mixture of microfillers and macrofillers, 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 suitable composition of hybrid fillers.

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, but addition of maximum filler content can adversely affect some of the important mechanical property. We wish to increase the desirable mechanical property and minimize the undesirable mechanical property. The filler reduces the thermal expansion coefficient and overall curing shrinkage, provides radio-opacity, improves handling and improves the aesthetic

results [4]. 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 filler materials, which are less hard than glass ones and therefore cause less wear on the opposing tooth [5]. 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% [6]. As the particle size is smaller, and resins made with this type of particle give the restoration a better finish, which is observed in its surface texture. 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 [7]. 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.

The effects of filler concentration and resinous components on the Compressive strength of highly filled composites were determined for prediction of the durability of the restorative resins. Several reports have

dealt with the relationship between filler levels in composite resins and their properties (Abdell *et al*., 1981; Draughn, 1981; Harrison and Draughn, 1976; Harrison and Draughn, 1978; Hirasawa, 1963; Hirasawa, 1965; Raadal, 1978; Raptis *et al*., 1979; Sbderholm, 1982; Swartz *et al*., 1982). For the most part, these investigations have been concerned with large particle fillers and or commercial products. In addition to filler level, the latter materials may well differ in other respects, including the size and type of filler and the nature of the resin matrix. Thus, it is difficult to extrapolate from these studies the role of filler level, per se, on properties. Therefore, the purpose of this research was to examine the role of filler level on the compressive strength of microfilled resins. Selected properties were determined for two microfilled resin series, each containing a different silica, glass and zirconia filler particle incorporated in graduated amounts into a light-cured BisGma (Bisphenol A Glycidyl Methacrylate) and TEGDMA (Triethylene glycol dimehtacrylate) matrix. The most important property investigated was Compressive Strength. Many attempts have been made at the correlation of the mechanical properties of composites with different filler concentrations, No corelation was found between filler concentration and hardness in highly filled posterior composites. Vougiouklakis and Smith, 1980; Raptis *et al*., 1979; Wilson *et al*., 1980; Boyer *et al*. 1982, studied ten commercial composites, including microfilled resin composites, and reported that tensile strength and filler concentration had a negative correlation coefficient. The tensile and compressive strengths were reported to be superior for a lower (45 volume percentage) rather than a higher (55 volume percentage) filler-volume fraction (Draughn, 1981a, b). The negative effect is assumed to be related to inherent flaws, especially air porosity included in the composite material (Smith, 1985). In addition, a critical filler concentration is needed for reinforcement and significant improvement in the properties of composites. Atsuta and Turner (1982) reported that with increasing loading of the silicate filler, an abrupt decrease in strength was found for composites with greater than 83 wt volume percentage silanated LiAlSiO4 particles. Although there are differences in the molecular structure and composition in the resinous matrix of the composites tested, positive relationships were determined between volume fraction of filler and diametral tensile strength, and Knoop hardness number. No correlation was observed between the degree of conversion and the mechanical properties tested in any of the resins. It thus seems plausible that it be assumed that the filler concentration and the nature of bonding between filler particle and resinous matrix play a prominent role in determining the properties of contemporary dental composites (Wuand McKinney, 1982; Sbderholm, 1985; Calais and S6derholm, 1988. Concerns raised regarding the applicability of the diametral tensile test to composite wear thus appear to have some validity (Goldberg *et al*., 1983). However, no correlation was observed in this study between compressive strength and mean roughness value.

Material: 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.

One more filler which was responsible for improvement in mechanical properties was Zirconia. Presently one 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.

Why Zirconia as a filler?

Silica and Glass fillers are used in a wide range of dental applications, such as inlays, onlays, implants, crowns and fixed partial dentures, because of its biocompatibility, wear resistance and aesthetics. They are, however, subject to brittle failure. The Zirconia powder is composed of highly agglomerated particles of nano sized crystals. The average particle size is 1.5-2 μm and the crystal size is ~6 nm. Zirconias have exceptional mechanical properties, excellent durability and stability, high biocompatibility and are very resistant to corrosion and scratches [9]. As for the colour, Zirconium oxide is ivory which makes it easy to match the colour of your natural teeth, which is important when you want to replace your lost or damaged tooth, especially in front. In addition, its ability to reflect light makes Zirconia dental implants more suitable when the colour needs to match perfectly [10]. Zirconia is a durable and tough dental material, which has esthetical advantages over metals. Compared to other ceramics Zirconia has superior strength and rising fracture resistance (R-curve behaviour) [11, 12, 13]. The mechanical properties of Zirconia are mainly due to transformation toughening when loaded above the fracture resistance. As Zirconia has a higher refractive index than the polymer matrix, a large particle size will result in a very opaque material. To improve the aesthetics of the composite a low particle size of the filler is necessary in

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order to prevent the particles from scattering light [14, 15]. For this reason a particle size below 40-60 nm is desirable.

MATERIAL AND METHODS

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 [16]. 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, and 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 37^0C 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 [17]. 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.

The purpose of the present work 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. The most effective fillers were segregated after analyzing their comparative mechanical properties [18].

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. 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 [19]. The crosshead of the testing machine until it just contacts the top of the compression tool plunger was adjusted. NOTE 1-The compression tool may not be necessary for testing of lower modulus for example, 700 MPa to 3500 MPa (100, 000 psi to 500, 000 psi)) material if the loading surfaces are maintained smooth, flat, and parallel to the extent that buckling is not incurred. Place thin specimens in the jig (Figure-1) so that they are flush with the base and centered [20].
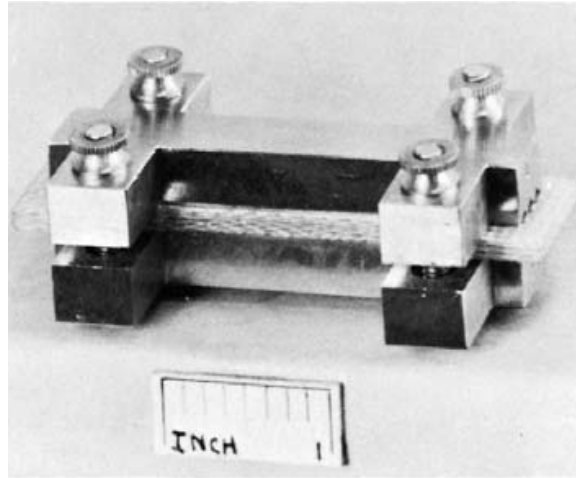
Figure-1. Support jig for thin specimen.

The nuts or screws on the jig should be finger tight. The assembly in the compression tool as described in note 1 was placed, A round-robin test, designed to assess the influence of specimen positioning in the supporting jig (that is, flush versus centred mounting), showed no significant effect on compressive strength due to this variable. However, flush mounting of the specimen with the base of the jig is specified for convenience and ease of mounting. NOTE 2 -A round-robin test on the effect of lateral pressure at the supporting jig has established that reproducible data can be obtained with the tightness of the jig controlled as indicated [21]. As only compressive strength or compressive yield strength, or both, were desired, following procedure was followed: Set the speed control at 1.3 mm/min (0.050 in. /min) and start the machine. We recorded the maximum load carried by the specimen during the test (usually this will be the load at the moment of rupture). For stress-strain data desired, we proceeded as follows: Attaching compressometer Set the speed control at 1.3 mm/min (0.050 in/min) and start the machine. We recorded loads and corresponding compressive strain at appropriate intervals of strain or, the test machine was equipped with an automatic recording device, so the complete load-deformation curve was recorded. After the yield point was reached, it was 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.

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 Taguchi's orthogonal array we have nine experimental composites. For each trail 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.

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Table-1.

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

ZIRCONIA = 23.7 %

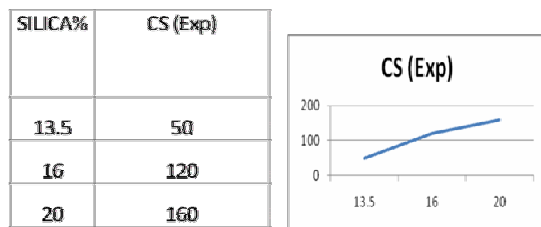| GLASS %	CS (Exp)
21	50
27.5	120
29	160
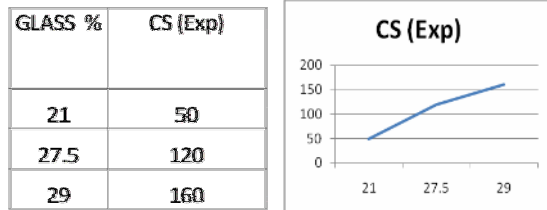
Figure-2. Variation in the compressive strength with zirconia volume fraction (percentage) kept constant at 23.7% and increasing the percentage of glass volume fraction (percentage).

GLASS = 21 %

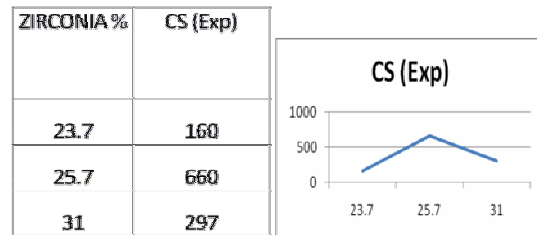| SILICA%	CS (Exp)
13.5	50
16	60
20	299
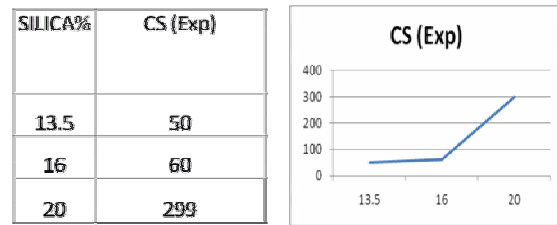
Figure-4. Variation in the compressive strength with zirconia volume fraction (percentage) kept constant at 23.7% and increasing the percentage of glass volume fraction (percentage).

ZIRCONIA = 23.7 %

SILICA%	CS (Exp)
13.5	50
16	120
20	160

Figure-3. Variation in the compressive strength with zirconia volume fraction (percentage) kept constant at 23.7% and increasing the percentage of silica volume fraction (percentage).

GLASS = 29 %

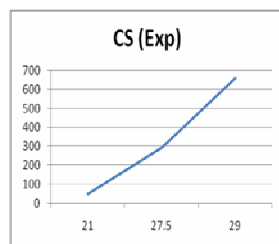| ZIRCONIA %	CS (Exp)
23.7	160
25.7	660
31	297

Figure-5. Variation in the compressive strength with glass volume fraction (percentage) kept constant at 23.7% and increasing the percentage of zirconia volume fraction (percentage).

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SILICA =13.5 %

Glass %	CS (Exp)
21	50
27.5	295
29	660

Figure-6. Variation in the compressive strength with silica volume fraction (percentage) kept constant at 23.7% and increasing the percentage of glass volume fraction (percentage).
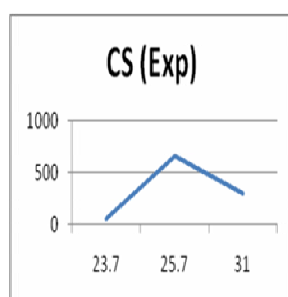
SILICA = 13.5 %

ZIRCONIA %	CS (Exp)
23.7	50
25.7	660
31	295

Figure-7. Variation in the compressive strength with silica volume fraction (percentage) kept constant at 23.7% and increasing the percentage of zirconia volume fraction (percentage).

RESULTS AND DISCUSSIONS

a) If Zirconia volume percentage is kept constant and glass volume percentage is increased the compressive strength increases.

b) If Zirconia volume percentage is kept constant and silica volume percentage is increased the compressive strength increases.

c) If glass volume percentage is kept constant and silica volume percentage is increased the compressive strength remains constant and then it increases.

d) If glass volume percentage is kept constant and Zirconia volume percentage is increased the compressive strength increases and then again decreases.

e) If silica volume percentage is kept constant and glass volume percentage is increased the compressive strength increases.

f) If silica is kept constant and Zirconia volume percentage is increased the compressive strength increases and then again decreases.

CONCLUSIONS

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 (Graph-7). So there is variation in the compressive strength with the increase in the Zirconia volume percentage. This shows that Zirconia is a significant contributing factor in the compressive strength of dental composite and with increase in the volume percentage of Zirconia the compressive strength definitely increases. Further, we can find out the exact volume percentage of Zirconia, silica and glass which will give maximum compressive strength and its effect on other mechanical properties. This can be done by using optimization 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.
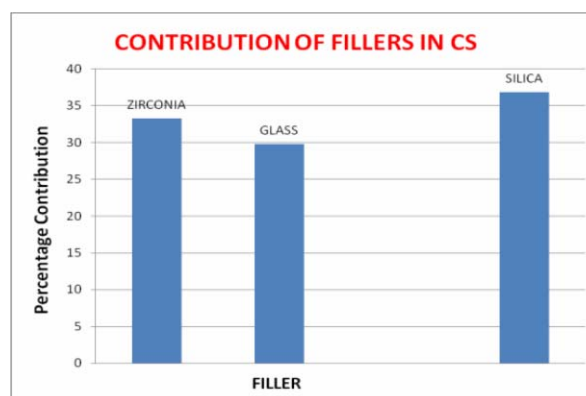
Figure-8. Contribution of filler percentage in compressve strength.

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