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EXPERIMENTAL EVALUATION OF DIFFERENT FILLERS IN DENTAL COMPOSITES IN TERMS OF MECHANICAL PROPERTIES

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ABSTRACT

The aim of this study was to evaluate in vitro the influence of nano-sized filler particles and agglomerates of nano particles (nano cluster) in resin based dental composite material. Four commercially available resins based dental composites-Filtek Z250, Filtek Supreme, Filtek Z100 (3M ESPE, St Paul, MN, USA), Heliomolor (Ivoclar Vivadent, Schaon, Liechtenstein) containing different filler particle type and morphologies were investigated. The compressive strength, compressive modulus, flexural modulus and flexural strength of these composites were evaluated. The nano clusters provided a distinct reinforcing mechanism compared with the microhybrid, microfill or nanohybrid resin based composites resulting in significant improvement to the strength and reliability.

Keywords: resin based dental composites, filler, compressive strength, flexural strength.

1. INTRODUCTION

The demand by the dental patients for tooth-coloured restoration, concerns regarding environmental impact and the adverse clinical reactions to amalgam filling materials have accelerated research into development alternate restoratives. However, despite the development of resin based dental composite (RBCs) materials the clinical longevity of dental amalgam remains superior [1]. Posterior amalgam restoratives exhibit a median survival time exceeding 11 years, whilst tooth coloured materials including, resin based composites (RBCs) possess median survival rates below 7 years [1]. A frequent cause of premature restoration failure is the occurrence of fatigue as a result of cyclic masticatory forces initiating crack propagation and manifested as fracture of resin based composites (RBCs) following several years clinical service [2]. Anterior placement restorations will typically be subjected to masticatory forces ranging from 100 to 200N [3], while posterior restorations may be loaded up to 800N [4]. Although forces generated while chewing foodstuffs are considerably lower (~10-20N) [5], It is frequently the accumulation of localized microscopic loading induced damage that influence the survival rate of restoration [6]. A recent response to the challenge of combining strength with asthetic appearance and working characteristics uses a combination of individually dispersed nano-sized filler particles and agglomerations of these particles, described as ‘nanoclusters’. The use of resin based dental composites increased enormously during the last two decade. Their increasing popularity could be attributed to the paradigm shift from G.V. Blacks “extension for prevention “[1] to minimal invasive dentistry [2] established by the development of adhesive dentistry. The adhesive resolution and popularity of resin based composites was initiated by two major breakthroughs: the introduction of the acid etch technique by Bunocore in the mid-1950s [7] and the development of Bis-GMA as an organic matrix for resin composites by Bowen in the early 1960s. Hybrid resin based composites (RBCs) consist of dispersions of individual silanated inorganic particles within an organic resin matrix. The development of resin based composites (RBCs) as an alternation to dental amalgam has resulted in optimization of the particle size distribution and filler loading, resulting in an improvement in the mechanical properties [8]. In order to achieve superior aesthetics, sub-micron fillers were introduced to the development of RBC materials. However, filler loading of early homogeneous microfill or resin based composites (RBCs) types was reduced due to high surface area to volume ratio, thereby limiting mechanical properties. The introduction of hetrogeneous microfills increased the filler loading (~50 voulme %) as pre polymers containing a high volume fraction of silanated nanofillers (~50nm) were incorporated into a resin matrix containing discreet sub micron particles. Although the approach improved the flexural strength of hetrogeneous resin based composites (RBCs) (80-160 Mpa) compared with “homogeneous” microfills (60-80Mpa) [9]. A recent response to the challenge of combining strength with aesthetic appearance and working characteristics uses a combination of individually dispersed nano sized filler particles and agglomerations of these particles, described as nanoclusters.

Filtek TM Supreme (3M ESPE, St. Paul MN,USA) contains silica and zirconia nano particles, which are partially calcinated to produce micro-sized porous clusters that are infiltrated with silane prior to incorporation into a resin matrix (Table-1).

A previous study suggested nano sized particles and “nano clusters” providing distinct mechanical properties as water uptake and subsequent strength loss was modified by size, morphology and resulting surface area of fillers [10].

This study was carried out on resin based composites with different filler type and content to evaluate the compressive and flexural properties.

2. MATERIALS AND METHODS

2.1 Material

Five commercially available resin based composites (RBCs) were investigated. Heliomolar (HM; batch GO5532: ShadeA3) (Ivoclar Vivadent, Schaan, Liechtenstein), Filtek Z100 TM MP Restorative (Z100: batch 7YG: sh A3), FiltekTM Z250 (FZ: batch 6EE: SHADE A3) and FiltekTM Supreme XT in 'body' (FSB: batch 6EE; shade A3) and 'translucent'shade (FST: batch 6CL: shade YT) (3M ESPE Dental Product, St. Paul, MN, USA).

2.2 Preparation of specimen

Four groups of each resin based composites (RBC) were produced consisting of 10 nominally identical disc shaped specimen (12mm diameter, 1mm thickness)using a black nylon ring mould covered with acetate strips (~0.1mm thick) to limit surface oxygen inhibition. The specimen were irradiated in one shot from a single surface at ambient temperature (23 ± 2^oC) for 20 seconds using an Optilux 501 light cure unit (Kery, Orange, CA, USA) with 12 mm curing tip diameter placed in contact with the acetate strip. Prior to testing the specimens were maintained in water bath at 37 ± 1^oC for 24 hours.

A 2mmx2mmX30mm rectangular bar was prepared of each group.

2.3 Mechanical Testing

2.3.1 Compressive Testing

The post cured composite specimens were tested in compression mode to measure their compressive strength, Yield stress, and compressive modulus. The compression test specimens were cut according to the D695 ASTM standard (length to diameter ratio = 2:1) using a diamond saw. Tests were performed using a servo hydraulic machine (MTS Mini-Bionox 2, Eden Prairie, MN) using a crosshead speed of 1mm/min. The compressive strength was calculated using the following equation:

$$\sigma = F/A$$

where

σ = compressive strength.

F = maximum failure load

A = cross sectional area of the specimen

The moduli of the specimen were determined from the slopes of a straight line fit to the initial linear portion of the stress strain curve. The peak slope value was used to calculate the compressive modulus.

2.3.2 Three point bending test

Flexural strength and flexural modulous of the post cured composites were determined by a three point bending test. The 2mmX2mmX30mm rectangular bars were used. The specimen was loaded to failure in a servo hydraulic testing machine (MTS Mini Bionix, eden Prairic, MN) at a cross head speed of 1mm/min. The distance between the support beams of the 3 point test jig was 25mm.

The flexural strength (in MPa) was calculated by the formula:

$$\sigma = 3LF/2BH^2$$

where

l = distance between the support in mm

F = Failure load (N)

B = width in mm.

H = height of the beam in mm.

The flexural modulous (E in Gpa) was calculated from

$$E = \frac{F\,L^3}{D\,4\,BH^3}.$$

where

L = Distance between the supports in mm

B = Width in mm

H = Height in mm

F/D = Slope in the linear region of the load-displacement curve.

3. RESULTS

The photocured composites were opaque and were without any visible surface crack. The composites also did not chip during cutting by using a diamond saw. The values of compressive modulous, yield stress and compressive strength of the composite are listed in Table-2.

Composites with nanofillers have a higher compressive modulous (p value <0.01) when compared with composite that are microfilled. As expected the yield strength increased with an increase in filler content. It is also interesting to note that the composites containing 70 weight % filler (irrespective of the filler type) had the same compressive strength (Table-2). The flexural modulous and flexural strength of nanofill and microfill composites were evaluated. Composites with nano filler particles were having significantly higher flexural modulous (p value<0.01) when compared to the composites containing micro filler particle. The flexural strength however was not statically significant.

4. DISCUSSIONS

The pre-loading of dental resin based composites (RBCs) materials to stimulate masticatory fatique reduces the strength and reliability to differing extents depending upon the filler particle load, size and morphology [11]. The pre-loading of dental resin based composites (RBCs) materials to stimulate masticatory fatigue reduces the strength and reliability to differing extents depending upon the filler particle load, size and morphology (2, 12, 13). During pre-loading to 2000 cycles in the current study

Heliomolar (HM) specimens completely failed to survive loading to 50 or 100N. This might be expected since low filler content generally provides inferior mechanical properties [4, 6, 8]. However, a resin based composites (RBCs) exhibiting a high strength does not necessarily possess a correspondingly high ability to resist clinical fatique wear [2, 12] approximately Z100 specimens survived pre-loading regimes of 100N. The high flexural modulous Z100 may inhibit the ability of the RBCs to resist deformation loading and the accumulation of surface and bulk defects resulting in pre mature failure [2, 12-14]. Futhermore, the differing reinforcement provided by varing filler particle morphologies may influence their ability to resist pre-loading regimes. FZ, FSB and FST survived pre-loading regimes to 100N. Both the micro hybrid (FZ) and "nanocluster" (FSB and FST) systems contain spheroidal fillers that have been associated with reduced stress concentration compared with the sharp edges present within irregular-shaped filler particulates [15]. The irregular filler particles present in Heliomolar (HM) may have failed to provide effective reinforcement and may not have deflected or dissipated the energy of the propogating crack-tip as effectively as speroidal filler. Therefore, irregular fillers may act as a deflect centre promoting the accumulation of stress-induced damage. Micro hybrid resin based composites (RBCs) systems usually posses a homogenous distribution whilst microfills exhibit a non-uniform size distribution of filler sizes. The microstructural inhomogenity of microfills derives from the relatively higher density of nanoparticles in the pre-polymerized regions, whilst lower filler proportions and inhomogenity of particle distribution in microfills can ultimately lead to reduced strength and a greater tendency for strength degradation under cyclic pre-loading leading to microfilled RBC material being contradicted where high contact stresses can be expected.

Since FZ, FSB and FST possess comparable resin chemistries, the differing responses to loading were attributed to the reinforcement delivered by the 'nanoclusters'. The term 'nanoclusters' may appear misleading as the cluster size extends to the micron range. However, each cluster consists of agglomerations of numerous nano-sized particles, which produces the distinctive microstructure. Previously,' nanoclusters' were known to exhibit distinct fracture mechanism compared with spheroidal and irregular filler particles, suggesting that pre-loading of FSB and FST modified the 'nanocluster' and produced a more damage tolerance system [16] The enhanced damage tolerance may be a consequence of crack bifurgation and the ability of the 'nanocluster' to absorb and disipitate crack stresses by collapsing into the pre-existing cluster porosities or loss of fragments from the main cluster structure [16]. The ability of discrete 'nanocluster' to deform and disipitate the accumulated fatigue loading stresses may enhance resistance to premature fracture within the resin composite system.

Interestingly, the 'nanocluster' particles contained within FSB and FST exhibit similarity to the traditional" agglomarated microfill complexes" (AMCs) described in the classification of resin composite systems by Lutz and Philips [17]. Agglomarated microfill complexes" (AMCs) consisted of primary particles (1-100nm diameter) produced by either hydrolysis or precipitation, subsequently heat treated to agglomerate the microfillers in the range of 1-25μm and admixed with pyrolytic silica particles (0.05 μm diameter) into the resin matrix. A number of commercial resin based composites (RBCs) containing agglomarated microfill complexes" (AMCs) particles were introduced in the 1980s including Nimetic-Dispers (ESPE, Seefeld Germany)and Answer (Johnson and Johnson, East Windsor, NJ, USA) with a filler loading of 39.8 and 39.1 volume %, respectively with an average sintered agglomerate particle size 13.8 and 20.7 μm, respectively [18]. According to the manufacturer, the 'nanocluster' within FSB and FST consist of a random interconnected network of silica and zirconia nanoparticles with an average agglomeration diameter of ~1 μm. However, in the current study, SEM images suggest 'nanoclusters' appeared to possess a diameter up to ~5 μm. Nevertheless, if silane infiltration is apparent within cluster interstices, silanated 'nanoclusters' effectively produce an interpenetrating phase composite (IPC) structure. The interpenetrating phase composite (IPC) essentially compromises of an interconnected network of silica/zirconia nanoparticles with an interpenetrating phase formed by the silane coupling agent. The IPC particles are then bonded into the resin matrix using additional silane. The 'nanocluster' possesses a high internal porosity infiltrated by a relatively weak second phase and would be expected to be inherently weaker than a comparable dense silica particle of the same size. However the interpenetrating phase "nanocluster" appears to be highly effective as reinforceing filler within the polymeric matrix. In 'dry' testing environments the interfacial silane phase will be relatively stable, however, the transmission of loading induced stresses through the silane layer may deform the nanocluster creating defects in and around clusters. The presence of water in the resin matrix may inhibit crack-tip, reduces the stress concentration and dissipates the crack [19]. Furthermore, in a 'wet' testing environment, hydrolysis and polymerization within the nanocluster silane phase could modify stress transfer both to and within the cluster particles, producing an enhanced capacity to tolerate local stresses and cluster deformation.

The ability of the nanocluster to deform and dissipate the accumulated fatigue loading stresses may enhance the resistance to premature fracture. However, such improvements in performance may be compromised over time by hydrolytic degradation of the silane [11]. Clinically the distinctive properties of the nanocluster system may have the potential to deliver improved clinical performance in the essentially aqueous oral environment. The observed resistance to strength degradation and the reduced risk of low stress failures may have the potential to increase average restoration lifetimes of dental resin based composites (RBCs).

Table-1. Summary of the constituents and quantities/ratios of components contained in the resin based composites (RBCs) investigated.

#	**Resin based composites** (RBCs)	**Classification**	**Resin**	**Filler**	**Total Filler Content**	
1	Heliomolar (HM)	Microfill	BisGMA, UDMA, DEMA	Pre-polymer (containing silica) Ytte-bium triflouride: 40-200nm (66.7%)	66.7 wt%	46.0 vol%
2	FiltekZ250 (FZ)	Microhybrid	BisGma, UDMA, $BisEMA_6$ TEGDMA	Zirconia/silica:0.01-3.5 µm (84.5% wt)	84.5 wt%	60.0 vol%
3	Filtek Z100 (Z100)	Microfill	BisGma, TEGDMA	Zirconia/silica:0.01-3.5 µm (84.5% wt)	84.5 wt%	66.0 vol%
4	Filtek Supreme Body (FSB)	Nanofill	BisGma, UDMA, $BisEMA_6$ TEGDMA	Silica: 5-20nm nano particle (8%wt): zirconia/silica: 0.6-1.4 µm nanocluster (71wt %)	79.0 wt%	59.5 vol%
5	Filtek Supreme Translucent (FST)	Nanofill	BisGma,UDMA,$BisEMA_6$ TEGDMA	Silica:75nm nanoparticle(40wt%) silica:0.6-1.4 µm nanocluster (30%wt)	70.0 wt%	57.5 vol%

BisGma: bisphenol A diglycidy ether dimethacrylate
TEGDMA: triethyleneglycol dimethacrylate
BisEMA: bisphenol A polyethylene glycol diether dimethacylate
UDMA: urethane dimethacrylate
HEDMA: hydroethyl dimethacrylate
DEMA: decandiol dimethacrylate

Table-2. Showing the compressive properties of the resin based composites (RBCs) investigated.

#	**Resin based composites** (RBCs)	**Modulous** (GPa)	**Yield strength** (MPa)	**Compressive strength** (MPa)
1	Heliomolar (HM)	4.7±0.12	164±3	214±35
2	FiltekZ250 (FZ)	5.7±0.4	191±10	227±53
3	Filtek Z100 (Z100)	5.3±0.11	189±10	230±10
4	Filtek supreme body (FSB)	8.0±0.5	184±13	210±20
5	Filtek supreme translucent (FST)	8.2±0.6	179±4	222±12

Table-3. The three point flexural modulous and strength of the resin based composites (RBCs) investigated.

#	**Resin based composites** (RBCs)	**Flexural modulous** (GPa)	**Flexural strength** (MPa)
1	Heliomolar (HM)	6.1±0.8	72±5
2	FiltekZ250 (FZ)	7.6±0.5	77±1
3	Filtek Z100 (Z100)	8.3±0.2	73±6
4	Filtek supreme body (FSB)	9.9±0.5	76±4
5	Filtek supreme translucent (FST)	9.5±0.7	74±6

5. CONCLUSIONS

Five resin based fillers were tested having different types of fillers. The 'nanocluster' system exhibited distinctive properties in response to the cyclic fatique pre-loading regimes such as, increased compressive modulous, flexural modulus, flexural strength. Consequently, it was accepted that 'nanoclusters' provided a distinct reinforcement mechanism to the resin matrix. The combination of unique reinforcement and silane infiltration of structural porosities improved the damage tolerance and may enhance the clinical longevity of 'nanocluster' resin based composites (RBC) restorations.

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