THE EFFECTS OF RESIN MATRIX COMPOSITION, FILLER VOLUME AND PARTICLE SIZE ON THE MECHANICAL PROPERTIES OF DENTAL RESIN COMPOSITES

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Abstract
The behaviour of dental resin composites (DRC's) under mechanical loading has been of concern in dentistry as it determines their clinical application. The aim of this study is therefore to check the effect of the resin matrix composition, filler volume and particle size on the mechanical properties of DRC's. In this experimental in-vitro study, six types of composite resins were used as follows: Two(2) Micro hybrid- Super Cor (SC) and Natural look(NL); Two(2) hybrid- Alpha dent (AD) and Henry Schein (HS) and two(2) Nano hybrid- i-Xcite(IX) and Fusion(FS),each with varying resin matrix composition, filler volume and particle size. Samples were prepared in aluminum molds and light cured using Optilight Max GNATUS light curing unit at constant time intervals of 20 seconds as specified in literature and designed by curing light manufacturers. The specimens were stored in distilled water at 37°C for 48 hours. Subsequently, the specimens were subjected to tensile test and three point loading using the BOSE Electro Force 3200 system at cross head speed of 1.0mm/min and varying speed of 0.5, 2.0 and 5.0mm/min. Henry Schein 20/20 (hybrid;bis-GMA;56%) exhibited flexural strength of 17MPa but tensile strength of 29.7MPa amongst the group. i-Xcite (nanohybrid; bis-GMA,TEGDMA,UDMA;76.5%) showed the flexural strength of 138MPa and relatively low tensile strength of 25.3MPa. All resin types showed increasing flexural strength with increasing strain rate. Tensile and Flexural strength are dependent on the resin matrix composition, the filler particle size and then the filler volume in that order.

Keywords: *Micro hybrid composite**,** Nano hybrid composite, Flexural strength, Filler volume, Particle size*

1. Introduction
All materials fail, corrode or undergo deformation under certain loading conditions, uni-axially or multi-axially, and or environmental impacts. The tooth is no exception as it fails either due to mastication, polymerization shrinkage or some biological degradation (In the case of decay). To keep its functionality and aesthetics, certain dental restorations are required. Many of the dental restoration materials were developed in 1840 and 1900, each having advantages and disadvantages. Dental Resin composites were developed in the late 1950s, and they have become a class of materials that are well reckoned with in dentistry.

Basically, a dental composite is a mixture of silicate glass particles within an acrylic monomer that is polymerized during application (Spiller Martin, 2012). In more detail dental composites consist of four major components (Craig and Powers, 2002). These are organic polymer matrix, inorganic filler particles, coupling agent, and the initiator-accelerator system (Craig and Powers, 2002, Van noort, 2002). The resin forms the matrix of the composite material, binding the individual filler particles together through the coupling agent (Van noort, 2002). The most commonly used monomer is Bis- GMA (bis A Glycol dimethacrylate). This monomers was invented by Bowen, and has been available for more than 30 years (Van noort, 2002, Leinfelder1997) Although, other dimethacrylates have been used, the Urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA) are known to improve the relative density of DRC's (Atai *et al*., 2004, Krämer *et al*., 2008, Roberson *et al*., 2002). It was established that addition of other dimethacrylates other than bis-GMA increases the mechanical and physical properties of DRC's (Asmussen and Peutzfeldt, 1998).

Good mechanical properties have been attributed to the degree of conversion of the monomers to polymers which is mostly depended on the curing time, shade of the resin, thickness of resin, type of filler (Lovell *et al.*, 2001, Albers, 2002, García *et al.*, 2006). The inorganic filler content that is added to the resin matrix serves as the reinforcement. The physical and mechanical properties of these composites have also been linked to the filler volume and particle size. It is discovered that as the particle size reduces, the dental resin composites strength increases. Consequently nano composites with very small particle size usually have better mechanical properties than the regular coarse hybrid (Tanimoto *et al.,* 2006). Although it has been argued that only time dependent properties like the fatigue life and wear resistance are better predictors (Belli*et al.,* 2014). The mechanical properties that are believed to show good description of how the DRC's behave clinically include the compressive strength, Elastic Modulus, Fatigue resistance, Flexural strength, Hardness, Tensile strength, wear, etc.(Ferracane, 2013). DRC's are basically classified based on their filler particle size and volume. The hybrid has the highest particle size followed by micro hybrid and then the nano hybrid. It has been discovered that as the particle size reduces, the strength increases (Spiller Martin, 2012). These materials are mostly polymeric and require light curing which sometimes causes polymerization shrinkage or too much stress on the teeth leading to spaces or cavities in the teeth. Apart from this, aesthetic purposes also have forced researchers to come up with composites that try to balance or compensate between strength, aesthetics and low polymerization shrinkage. The innovation to reduce particle size of the glass and silica fillers [Miyasaka, (1996), Brosh *et al*., (1999)], some have come up with the use of glass fibers, nano-porous fillers, branched fibers or even ceramic whiskers (Roberson *et al*., 2002, Xu *et al*.,1999). Also, in the search for the optimum constituents, strengthening the bond between organic and inorganic parts to increase strength was considered (Herrera-Franco and Valadez-Gonzalez, 2004). It is therefore of utmost importance to understand the relationship between the key predictors of mechanical behaviour- namely: resin matrix composition, filler volume, and filler particle size. These determine the resultant mechanical properties. This study makes use of six different commercially available types of DRC's with varying matrix composition, filler volume and particle size with the aim of studying their behaviour under certain loading conditions.

2. Materials and Methods

2.1 Materials

The present experimental in-vitro study was conducted on six different composite resins with the characteristics presented in Table 1.

2.2 Sample preparation

For each group of composites, five samples were made in a cylindrical aluminium mould of 6mm diameter and 2mm height for tensile test. For each group of composites, five samples were made in a cuboidal aluminium mould of 8 x 2 x 2mm for three point loading test. Composite resin was applied and packed inside the mould using incremental technique and was light cured for 20 seconds using the Optilight Max GNATUS device.

Table 1: Composition of all six brands

BRAND NAME	SHADE	RESIN (MATRIX)	RESIN TYPE	FILLER VOL %	PARTICLE SIZE **(μm)**	MANUFACTURER
Alpha-dent (AD)	A2	Bis-GMA	Hybrid	56	0.02-4	Dental Technologies, Illinious, USA
Henry Schein 20/20 (HS)	B3	Bis-GMA	Hybrid	64	0-2	HenryShein, U.K
Super Cor (SC)	A1	Bis-GMA	Micro Hybrid	59	0.6	SpofaDental, Markova, Italy
Natural look (NL)	A2	Bis-GMA	Micro Hybrid	59	0.04-2.8	DFL Industria e Cormarcio S.A. RJ, Brazil
i-Xcite (IX)	A3	Bis-GMA, UDMA, TEGDMA	Nano Hybrid	76.50	0.12-1.5	i-dental, Lithuana
Fusion (FN)	A2	Bis-GMA, bis-DEMA, TEGDMA	Nano Hybrid	77	0.02-0.07	PrevestDenpro Limited, India

BisA Glycol dimethacrylate (bis-GMA), Urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA), bis A Glycol Diethylmethacrylate (bis DEMA).

2.3 Experimental procedure

After completion of the polymerization process, the specimens were conditioned for 48 hours in distilled water at 37°C and then were subjected to tensile and flexural test using BOSE Electro Force 3200 at cross head speed of 1.0 mm/min. Afterwards a flexural test at varying cross head speed of 0.5mm/min, 2.0mm/min and 5.0mm/min were performed on each category: hybrid (Alpha Dent), Micro hybrid (Super Cor), Nano hybrid (i-Xcite).

The stresses and strains are obtained from the BOSE Electro force 3200 through the WinTest® control software.

3. Results and Discussion

Using the Win test data acquisition software that is connected to the Electro Force 3200 Machine, the data for the different tests were obtained and the computations for the different mechanical properties were carried out.

Tensile loading

The stress strain behaviour of the different brands under tensile loading are shown in Figures1-7 below. Their tensile strengths are shown in Table 2.

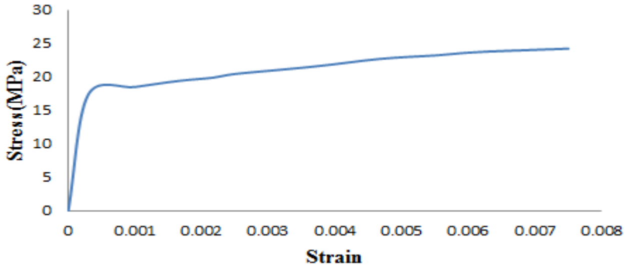

Figure 1: Alpha Dent Stress strain behaviour under tensile loading

The Stress-Strain behaviour of Alpha dent is shown above. It is easy to see that the profileis linear up to the 20MPa stress. This is the yield stress of the sample. Little straining of the material is also indicated on the plot. However from thence little increase in the strength to about 25MPa which is the ultimate stress shows significantly high strain. This means that Alpha dent is useful and will show little strain in dental restorations till 20MPa. However the small admissible strain indicates that the material is brittle.

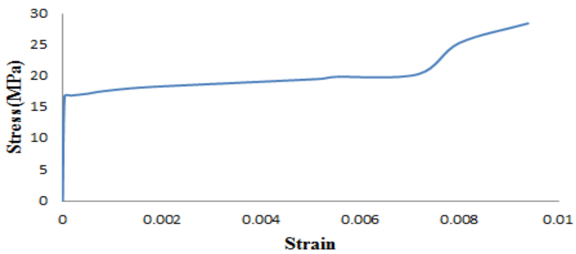

Figure 2: Henry Schein Stress strain behaviour under tensile loading

The Henry Schein 20/20 sample shows no strain till Stress of about 17MPa where it starts showing strain hardening till strain of 0.6 %(0.006). Beyond this value, it draws till a stress of about 29MPa. This shows that henry Schein 20/20 will show perfect behaviour till stress of about 17MPa and then some increase in strain with little increases in stress.

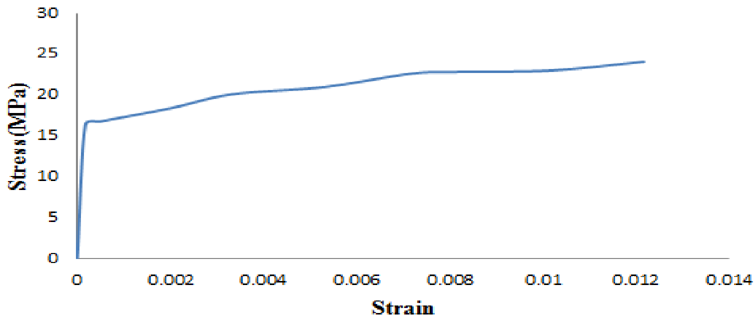

Figure 3: Super Cor Stress strain behaviour under tensile loading

The Super cor sample exhibits stress strain linear relationship up to 17MPa which is the yield stress. This is followed by non-linear elasticity until the sample fractures at about 24MPa.

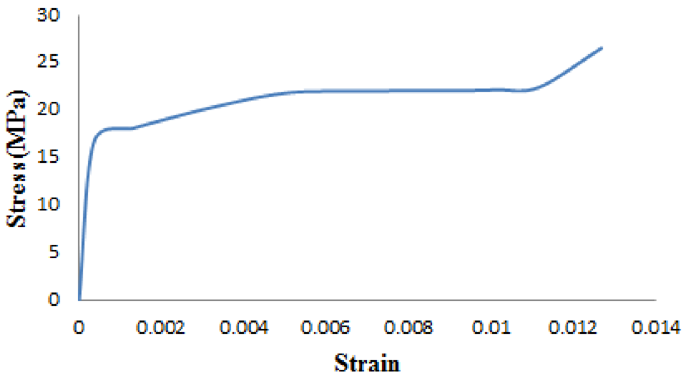

Figure 4: Natural look Stress strain behaviour under tensile loading

The Natural look specimen shows normal elasticity till it yields at 17MPa subsequently it strain harden up to 21MPa where it undergoes drawing until it fractures at about 27MPa. A maximum strain to fracture of 1.4 % was recorded because the specimen is brittle.

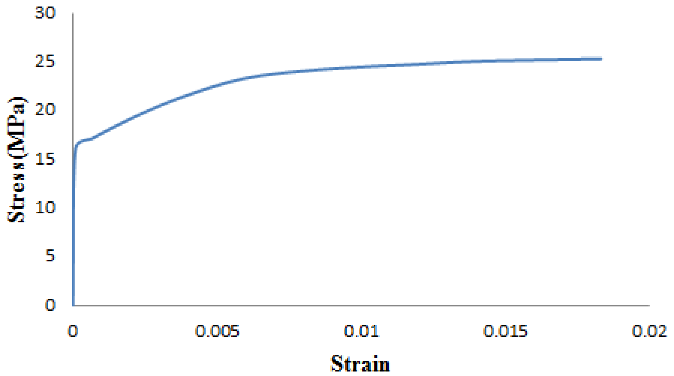

Figure 5: i-Xcite Stress strain behaviour under tensile loading

The i-Xcite sample shows a one tangent stress strain deformation behaviour. After its yield point at 17MPa it undergoes some strain hardening before failure.

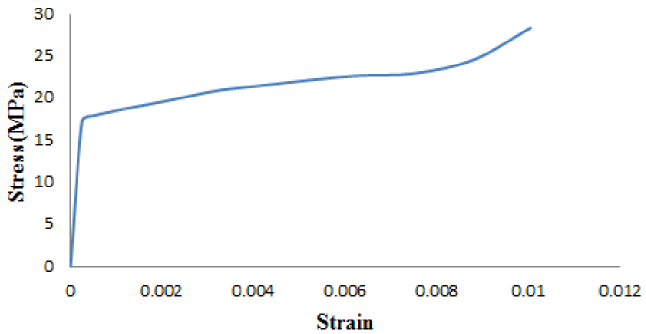

Figure 6: Fusion Stress strain behaviour under tensile loading

The Fusion brand of dental resin yields at about 18MPa. It experienced strain hardening for a stress range of 5 - 23MPa. Subsequently, it draws eventually to fracture at 29MPa.

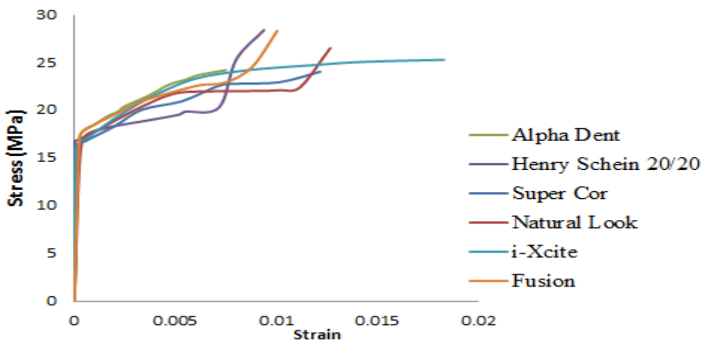

Figure 7: Stress strain behaviour of all samples under tensile loading

It can be seen that five resin types have their yield point at about 17MPa, except Fusion resin composite which has a yield point at 18MPa. They experience strain hardening due to the necking process which occurs until failure.

The i-Xcite brand will fail at a higher strain rate of 2%, than all the other types of resin composites. However, Alpha Dent will fail faster at a strain of 0.75%. Clinically,the hybrid, Henry Schein 20/20, can be used where high tensile strength is required. The Super Cor sample will fail when applied stress is greater than 24MPa. I-Xcite on the other hand can be used where high strains are expected.

Table 2: Tensile strength of all resin brands

Product type	Tensile Strength(MPa)
Alpha Dent (AD)	25.1
Henry Schein (HS)	29.7
Super –Cor (SC)	25.9
Natural Look(NL)	27.1
i-Xcite(IX)	25.3
Fusion(FS)	29.1

The tensile strength value of Henry Schein a hybrid composite is the highest followed by Fusion, a nano hybrid composite and then Natural look, a micro hybrid composite, the least being Alpha Dent, an hybrid.

Results of Flexural loading Test

The stress strain behaviour of the different samples under three point loading or flexural loading are shown in Figures 8-14 below. Their Flexural strength and moduli are shown in Table 3 below.

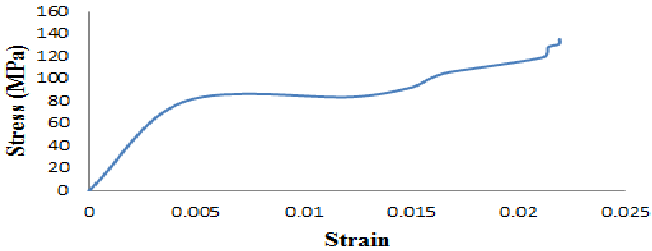

Figure 8: Alpha Dent Stress strain behaviour under three point loading

Alpha dent under flexural loading shows some linearity till 80MPa then shows strain hardening and drawing until it fails at about 134MPa. Under flexural loading Alpha Dent shows some toughness.

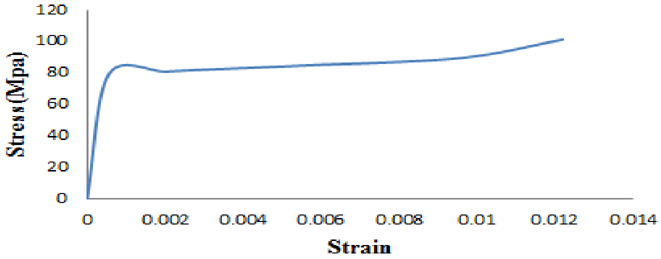

Figure 9: Henry Schein Stress strain behaviour under three point loading

The Henry Schein 20/20 brand shows normal elasticity until about 80MPa under flexural loadingand then yields before it undergoes strain hardening until stress of about 90MPa where it undergoes drawing until it fractures at stress of 111MPa. It is also observed that the maximum strain before fracture is about 1.2 % which indicates its brittleness.

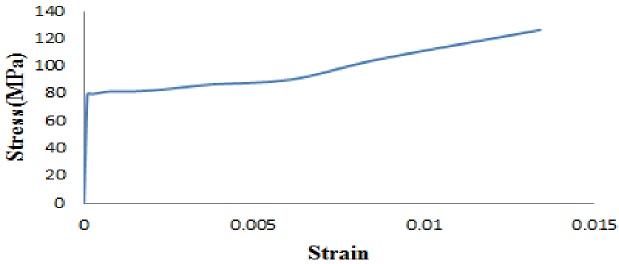

Figure 10: Natural look Stress strain behaviour under three point loading

Under the flexural loading, the Natural look sampleis some elastic up to 80MPa and then yields before it experiences a strain hardening until stress of 90MPa where it undergoes some linear elasticity until it fractures at about 125MPa. It is also observed that the maximum strain before fracture is about 1.4%.

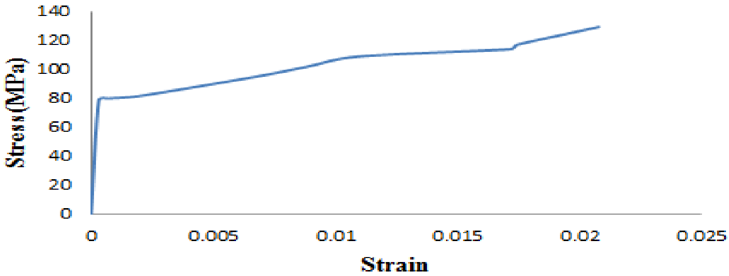

Figure 11: Super Cor Stress strain behaviour under three point loading

Under flexural loading, the Super Cor sample is elastic up to 80MPa. It yields before strain hardening until approximately 118MPa where it undergoes drawing to fracture at 121MPa. It is also observed that the maximum strain before fracture is about 2.2 % to explain its brittleness.

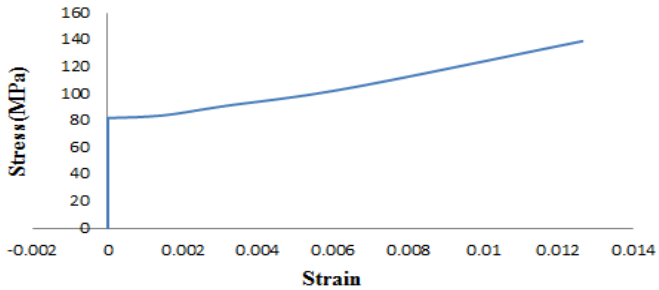

Figure 12 i-Xcite Stress Strain Behaviour under Three Point Loading

Under flexural loading, the i-Xcite sample shows no strain until about 80MPa before it experienced strain hardening up to a stress of 140MPa where it fractures. It was also observed that the maximum strain before fracture is about 1.3 %. The i-Xcite sample fractures before it yields.

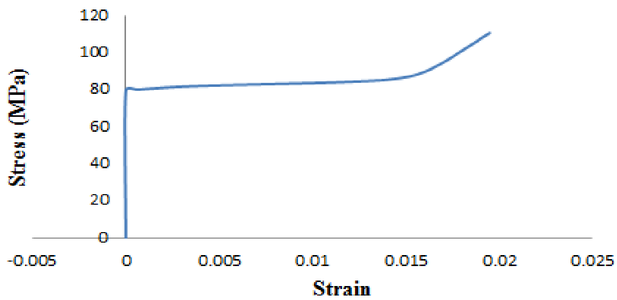

Figure 13: Stress Strain Behaviour of Fusion Sample Under Three Point Loading

The Fusion sample shows no strain under flexural loadingup to 80MPa and then yields before it experiences a strain hardening to a stress of approximately 86MPa where it undergoes drawing till it fractures at 113MPa. It is also observed that the maximum strain before fracture is about 1.9 %.

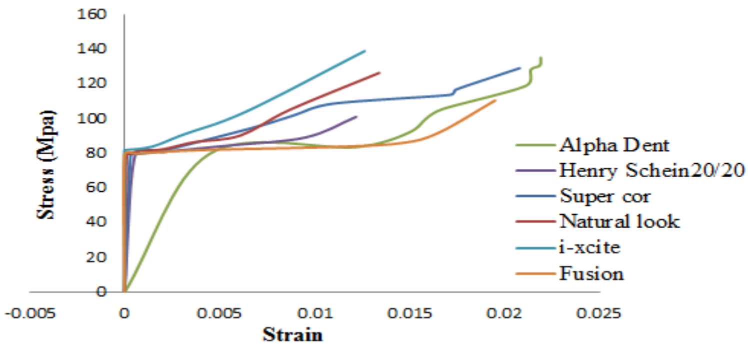

Figure 14: Stress Strain Behaviour of all the Brands under Three Point Loading

With the exception of i-Xcite, it is clearly seen that all the resin types have their yield stress at about 80MPa before they experience some form of strain hardening. This occurs till failure. It can also be observed that i-Xcite shows that it will fail before it yields and therefore fails at strain of about1.3%, on the other hand Alpha Dent shows necking and drawing before failure at 2.2% strain. Clinically speaking, i-Xcite, a nano-hybrid, can be used where high flexural strength is required. Henry Schein 20/20 will fail under flexural loading of more than 100MPa. The Alpha Dent sample on the other hand can be used where high strains under flexural are expected.

It can also be observed the elastic behavior of these materials before failure. In this case, Fusion is still hyper elastic in behavior, while i-Xcite shows almost linear elasticity behavior. The i-Xcite resin composite, a nano hybrid composite has the highest flexural strength followed by Alpha Dent, a hybrid composite and Super Cormicro hybrid composites. The least in strength is being Henry Schein.

In addition, the i-Xcitenano hybrid composite has the highest flexural modulus. This is closely followed by Natural look microhybrid composite and Alpha Dent micro hybrid composites while the least is Henry Schein resin composite.

Table 3: Flexural strength and moduli of all the samples

Product type	Flexural Strength (MPa)	Flexural Modulus (GPa)
Alpha Dent (AD)	134.4	11.6
Henry Schein (HS)	101.5	9.8
Super –Cor (SC)	129.3	10.5
Natural Look(NL)	126.6	17.3
i-Xcite(IX)	138.0	18.6
Fusion(FS)	104.8	6.3

Table 4: Flexural strength of samples under varying strain

	Product types	Strain rates			
		0.5mm/min	1.0mm/min	2.0mm/min	5.0mm/min
Flexural Strength(MPa)	Hybrid (Alpha Dent)	154.1	134.4	148.7	185.8
	Micro-hybrid (Super-Cor)	102.8	129.3	126.6	127.9
	Nano-hybrid (i-Xcite)	73.5	138.0	126.5	134.0

Flexural strength under varying strain rate

The flexural strength at varying strain rate for each of the categories- Alpha-dent (hybrid), Super-Cor (micro-hybrid), i-Xcite (Nanohybrid) - are given in Table 4. The graph that easily shows the trend is in Figure 15

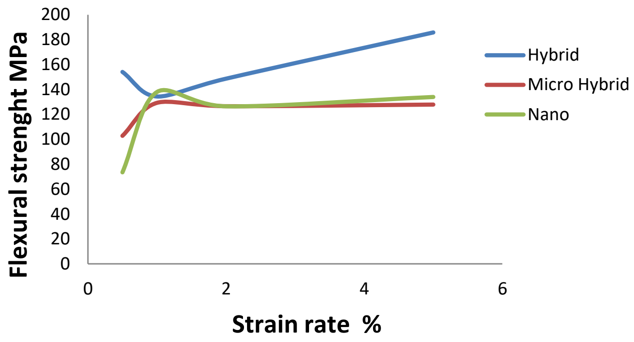

Figure 15: Flexural strength at varying strain rate

From Figure 15, it can be observed that, the strain rate increases with the increase in flexural strength of the composites. This is in agreement with Adeleye, (2014). However, for the regular grain size hybrid, there is a slight drop in flexural strength between 0.5mm/min and 1mm/min followed by a steep rise towards 5.0mm/sec. For Micro hybrid there is a rise between 0.5mm/min and 1.0mm/min and then a constant stress for other strain rates. The Nano hybrid shows an increase from 0.5 mm/min to 1.0mm/min, a slight decrease at 2.0mm/mm and then a gentle rise to 5.0mm/min, with its highest point at 1.0mm. The flexural strength shown by the Hybrid as the strain increases can be attributed to the presence of only Bis-GMA, larger particle size and consequently a tougher behaviour. In the literatures, most experimental studies are done with the three-point loading test at cross head speed of 1.0mm/min probably because this is a critical point and at least best mimics the worst possible scenario in the mouth.

It was observed that only the Henry Schein 20/20, i.e. an hybrid with filler volume of 56% and bis-GMA showed the lowest flexural strength with the best tensile strength amongst the group. Also i-Xcite, a nanohybrid with filler volume of 76.5% and bis-GMA, TEGDMA, UDMA showed the highest flexural strength and relatively low tensile strength. Micro hybrid showed relative balance in both cases. Nano hybrid showed better flexural strength in the group which agree with the works of Tanimoto *et al., (2006*) and Pontes *et al., (2013*). Worthy of note also is that the second best flexural strength was Alpha Dent which is a hybrid. This confirms the inference of Moezzyzadeh (2012) and Hamouda *et al., (*2012) that states that hybrids with normal grain size show better compressive strength than the micro hybrids.

Also that i-xcite showed better flexural strength in comparison to Alpha Dent might be due to the presence of UDMA and TEGDMA as like that in Sideridou *et al*., (2011). The poor behaviour of Fusion in the three point loading might be attributed to its hyper elastic nature which causes it to fail quickly having being subjected to so large a strain. This hyper elastic behaviour might be due to the presence of bis-DEMA.

It is observed that under tensile loading all the resin types behave the same way or at least yield at stress of 17MPa. Under three point loading, all the resin types yield at 80MPa before showing some other behaviour.

Conclusions

With increasing particle size, there is a corresponding decrease in flexural strength. The introduction of some dimethacrylates that are not Bis-GMA, better mechanical properties could be obtained. Tensile and Flexural strengths are primarily and sequentially dependent on the resin matrix composition, the filler particle size and then the filler volume. However, good flexural strength most times is associated with relatively poor tensile strength and vice versa. With respect to expected stress, if the required tensile strength is below 17MPa, and flexural strength does not exceed 80MPa, any of the resin types could be used. However, attention must be paid to the tolerable strain to avoid failure.

Recommendation

Further experimental study should be done to investigate the effects of temperature on the deformation behavior of dental resin composites.

Acknowledgement

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