THE EFFECT OF FILLER ON THE MECHANICAL PROPERTIES OF A

NOVEL RESIN-BASED CALCIUM PHOSPHATE CEMENT

by

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DEDICATION

This thesis is dedicated to my family, for all the emotional and continuous moral support

throughout my educational journey.

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INTRODUCTION

Resin-based composite is a popular dental filling material for repairing anterior and posterior teeth. Typically, dental composites consist of a methacrylate-based resin matrix and glass or ceramic fillers that are dispersed throughout the matrix. The resin matrix sets through free radical polymerization initiated by light. The most commonly used base monomer is Bis-GMA (bisphenol glycidyl methacrylate).[4] It was first introduced by Bowen more than 40 years ago. Bis-GMA is considered to be a highly viscous monomer. Its viscosity makes the resin composite more difficult to handle and negatively affects the degree of conversion. To overcome the deficiencies of Bis-GMA, a low viscosity monomer such as TEGDMA (triethylene glycol dimethacrylate) is added to thin the resin matrix. Bis-GMA/TEGDMA systems enhance the handling characteristics of the resin and improve double bond conversion.[5]

Fillers play a major role in determining the mechanical properties of composites. The addition of filler reduces the resin content of the resin composite and thereby decreases polymerization shrinkage, in addition to improving wear characteristics. The majority of research on dental composites during the last decade focused on improving the filler system. Decreasing filler particle size and modifying particle morphology have improved the mechanical properties and esthetics of dental resin.[4, 6]

However, secondary caries and restoration fracture continue to be the main challenges facing dental clinicians and researchers.[7] It has been reported that the majority of the dental clinician's time is spent replacing restorations that failed due to secondary caries.[8]

Since the caries-preventive effects of fluoride were first demonstrated, researchers have been trying to develop restorative materials that contain and release fluoride.[9, 10] Though fluoride release from resin-based composite is typically low, glass ionomers, and resin-modified glass ionomers and compomers release significant amounts of fluoride ions.[11] However, these materials have limited clinical application due to their inferior mechanical properties. Researchers have sought to improve the performance of fluoride-releasing restorative materials by adding resin to glass ionomer.[12]

Recently, a new type of resin composite was developed that releases calcium (Ca) and phosphate (PO4), the ions that form hydroxyapatite $[Ca_{10}(PO_4)_6(OH)_2]$. These new composites have the ability to increase the mineral content in caries lesions. However, $Ca-PO_4$ composites have inadequate strength to restore occlusal surfaces of posterior teeth. Several studies suggest they should only be used as lining materials.[13, 14]

HYPOTHESIS

A new composite resin with tricalcium phosphate fillers can be formulated to achieve the minimal mechanical requirements to serve as a restorative material and as a pit and fissure sealant.

REVIEW OF LITERATURE

CALCIUM-PHOSPHATE RESIN COMPOSITE

Researchers have been trying to develop a biologically active restorative material that may stimulate remineralization of tooth structure through the release of calcium and phosphate. These materials contained amorphous calcium phosphate (ACP) as a bioactive filler embedded in a polymer network.[15-17] Several studies demonstrated the capability of ACP-based materials in providing Ca and PO_4 ions needed to remineralize damaged enamel crystal structure. [18, 19] It was suggested by Skrtic and colleagues that ACP may promote the deposition of apatitic tooth mineral when used as filler in photo-polymerizable methacrylate matrices.[14] Such properties make the ACP-based restorative materials particularly useful in patients who are highly susceptible to dental caries as a result of radiation therapy or medications that cause dry mouth.[20]

However, poor crack resistance under masticatory load was defined as a problem with this type of dental composite. ACP has deficiencies when used as a filler. It is more hydrophilic and biodegradable when compared with silanized glass-reinforcing composites.[15] The inferior mechanical properties of ACP and water sorption characteristics have been reported in many studies. W.F. Regnault et al. tested the effect of adding ACP to resin on mechanical strength.[21] They compared the biaxial flexural strength (BFS) of unfilled polymer with that of ACP-filled polymers. The BFS of the unfilled polymer was found to be 68.8 ± 15.5 MPa. On the other hand, the BFS of the ACP-filled polymer was significantly less than that of the unfilled polymer having a value of 43.6 ± 2.7 MPa. He concluded that adding a mass fraction of 40 percent of ACP

into a polymer reduced their BFS by about 29 percent. He attributed the lower mechanical strength of ACP-filled polymers to the weak filler/matrix interface due to calcium and phosphate efflux that led to spacial changes. Conversion of ACP to apatite may have had a negative effect on the BFS of the ACP-filled polymers. Regnault et al. also tested the effect of adding ACP fillers to polymers on water sorption (WS) behavior. It was found that the polymer alone absorbed a maximum mass fraction of 0.85 ± 0.30 percent. ACP-filled polymers absorbed a maximum mass fraction of 1.29 ± 0.15 percent. The difference in WS values was statistically significant. The hydrophilic nature of ACP significantly affected the amount of water absorbed in ACP-filled polymers. The poor filler/matrix interface in this composite played a critical role in governing the water diffusion and hydration of the ACP particles.[21] ACP resins were found to be mechanically unstable due to the aggregation of ACP particles that compromised the interfacial interaction with the dental resins.[16]

Research suggested several methods to improve the ACP filler/polymer matrix interfacial properties. This could be achieved by controlling the particle-size distribution and ACP-filler surface properties, and by fine-tuning the resin. Mechanical milling of ACP particles has led to improvements in the flexural strength. This improvement is attributed to a more homogenous distribution of the finer ACP particles within the composite. Milled ACP composites also showed less water sorption due to the decrease in the number of voids and defects throughout the prepared samples. O'Donnell et al. studied the effect of ACP-filler particle size on the mechanical properties of these composites.[15] They investigated the behavior of milled ACP-filled composites in comparison with unmilled ACP-filled composites. The BFS of the unmilled ACP

composites had a mean value of 48 MPa, while the BFS of the milled ACP composites

had a mean value of 70 MPa. These results demonstrate the better performance of the

milled ACP composites in terms of BFS. The author attributed this to better distribution

of the filler in the matrix when milled. Improved dispersion of the filler minimized the

spaces between the particles allowing a better fit into the resin matrix. The milled ACP

composites had fewer voids, which improved the mechanical properties. O'Donnell also

compared the WS of milled ACP composites with that of unmilled ACP composites.

Composites with milled ACP fillers absorbed a maximum mass fraction of 1.55 ± 0.49-

percent water. Composites with unmilled ACP absorbed 2.28 ± 0.60-percent water,

which is almost 50-percent more WS than that of composites containing milled ACP.[15]

In another attempt to improve the mechanical properties of ACP-filled resins, Skrtic and

Antonucci investigated how several resin compositions affected the physical and

chemical properties of ACP-based composites. The average BFS values of wet composite

samples were 51 ± 8 MPa. BFS values were not affected by the change in the type of

resin used. It was found that formulations that contained hexamethylene dimethacrylate

(HmDMA) as a monomer performed better in terms of water sorption. It was also

observed that formulations with 2-hydroxyethyl methacrylate (HEMA) and ethoxylated

bisphenol A dimethacrylate (EBPADMA) released more Ca and PO_4 than other

formulations. The author attributed this to the hydrophilic nature of HEMA, which may

allow better access of the filler to water. The explanation for the improved ion release

when using EBPADMA matrices is that this polymer has lower cross-linking density,

which allows the formation of an open network enhancing the diffusion of ions. [18, 22]

Lately, researchers have been investigating the use of tricalcium phosphate (TCP) as a filler for these resin-based calcium phosphate cements. Nakagawa et al. investigated the effects of adding α – TCP and β – TCP to the basic mechanical properties of apatite cement.[23] They found that α – TCP was superior to β – TCP in terms of mechanical strength. Improved mechanical properties of α – TCP resulted from the larger apatite crystal formation. Their study concluded that apatite cement containing α – TCP as a filler showed 30-percent higher mechanical strength than α – TCP-free apatite cement.[23] Pure beta tri-calcium phosphate (B-TCP) has been used for replacing bone in oral surgery with success.[24] It was found that this material can be detected in the transitional phase of hydroxyapatite conversion. B-TCP was found to be capable of promoting periodontal regeneration in human intraosseous periodontal defects.[25] Several studies investigated the effect of B-TCP on human cells, and it was found to be biocompatible with human bone.[26-28] Other studies explored the potential of TCP in reversing the early stages of non-cavitated enamel lesions. Karlinsey et al. performed an *in vitro* study to explore the potential of B-TCP modified with fumaric acid (FA) in remineralizing non-cavitated lesions. TCP-FA material performed better than native and milled B-TCP and was found to be superior in remineralizing subsurface enamel lesions.[29] Notably, this type of TCP is crystalline in nature, suggesting a stronger form of a calcium-phosphate releasing material.

PIT AND FISSURE SEALANTS

A decline in dental caries prevalence has been noticed in the past few decades.[30] Despite this decline, some individuals and populations continue to develop dental caries.

In fact, an increase in the prevalence of dental caries has been reported in some countries.[31] Researchers generally agree the most common sites for developing caries are within pits and fissures. In the US, it was found that 56 percent of caries lesions in the permanent teeth of children between the ages of 5 years and 17 years occur on occlusal surfaces.[32] [30]

Many products and systems on the market claim to prevent dental decay, but few have been shown to be effective. Pit and fissure sealants (PFS) have been successful and their use is routine in dental practice.[33] Research demonstrates that the application of PFS has reduced dental caries occurrence in occlusal surfaces by 60 percent.[34] The PFS prevent dental decay by preventing the adherence of food and bacteria in deep fissures.[35]

In 1967 Buonocore and his co-worker published their first paper on the successful use of PFS. However, the major breakthrough in PFS research was not until Bowen used the new cross-linking dimethacrylate monomer (BIS-GMA), a substance still considered one of the main constituents of most dental sealants and resin composites.[36]

Resin sealants are primarily composed of a mixture of mono- and di-functional monomers.[33] They are characterized by a low filler content when compared with resin composite used for restorative purposes.[37] Throughout the years, researchers have experimented with different types of materials to seal the pits and fissures of teeth. The most widely accepted are resin- and glass-ionomer-based sealants. Many studies have tried to improve the preventive capacity of sealants by adding new components to the resin matrix. Fluoride has been added to many sealants now available. It makes sense that adding fluoride would increase the protective effect of PFS, but data in the literature show no significant difference in caries prevention between conventional PFS and those

containing fluoride.[38] Other studies tried incorporating ACP in dental sealants and showed promising results in promoting remineralization of artificial lesions.[39]

COMPRESSIVE STRENGTH

Compressive strength is a meaningful mechanical property to evaluate brittle materials that are weak in tension, such as resin-matrix composite; compressive strength is the force required to crush a material when a compressive force is applied axially.

Clinically, dental restorations are subjected to compressive forces, especially in the region of the posterior teeth, making the compressive strength and flexural modulus properties of importance to the dental clinician and material researchers.

A typical compressive strength value for a resin composite is 225 MPa.[40] Lee et al. studied the compressive strength values of experimental polymeric calcium phosphate cement and found it to be 0.26 MPa to 117.58 MPa.

Studies showed that the behavior of the resin-matrix composite is influenced by many factors, mainly the composition and the microstructure of the material. The morphology of the filler particles, their distribution in the matrix, and the presence of pre-existing flaws will influence the mechanical properties of a material.[41, 42]

DEGREE OF CONVERSION

Degree of conversion describes the extent of transformation from double to single bonds during free-radical polymerization of a methacrylate-based resin. It is desirable to have higher degrees of conversion, because any residual unreacted monomer could leach into the oral cavity and give rise to biocompatibility concerns.[43, 44] Also, there is a strong correlation between the degree of conversion and the physical properties of a material;

the higher the degree of conversion, the stronger the material. The degree of conversion

varies according to material composition and curing conditions and is usually 80-percent

in the bulk of the material.[40]

Bis-GMA monomer is a basic component of the majority of current resin-based

composites. It achieves high double-bond conversion when photo-cured. Researchers

have been investigating other resin systems that reach degrees of conversion comparable

to Bis-GMA monomer. Skritic and his colleagues experimented with resin systems

having ethoxylated bisphenol A dimethacrylate (EBADMA) as their basic monomer and

ACP as their filler. They found that the degree of conversion was somewhat reduced and

affected by size of the ACP fillers. Resin formulations that contained milled ACP

performed better in terms of the degree of vinyl conversion. The effect can be attributed

to the more homogenous resin/matrix interface that improved the translucency and

subsequently the photo-polymerization.[45]

DEPTH OF CURE

Research on dental resin shows that many mechanical properties such as hardness,

creep, compressive strength, and flexural modulus are directly influenced by the extent of

polymerization. Also, studies have shown that resin restorations that are not sufficiently

cured can adversely affect the dental pulp and the oral mucosa due to reduced

biocompatibility.[46]

Many factors influence the depth of cure of resin composites. The material

composition, shade, filler particle size, and light related factors, such as light intensity,

spectral distribution, and exposure time are of prime importance.[47, 48] The depth of cure

for polymer-based restorative materials, determined in accordance with ISO 4049, should not be less than 1.5 mm.

HARDNESS

Hardness is commonly described as a material's resistance to deformation that is caused by a localized indentation or a scratch. Hardness tests are simple and relatively inexpensive when compared with other tests that evaluate the mechanical properties of materials. Hardness tests are used more frequently than any other mechanical test to evaluate materials, because samples can be used more than once; the test does not destroy the samples. The test is also very useful in estimating other material properties such as tensile strength and rigidity.[40, 49, 50] Many studies have demonstrated the correlation between hardness and the degree of conversion in dental resin. Consequently, hardness has been used as an indirect measure of the degree of conversion.[51-55]

There are several methods to examine the hardness of a restorative material. The Knoop hardness test is most commonly used to evaluate dental resin composites. This technique utilizes a pyramid-shaped diamond indenter pushed with a predetermined force into the specimen. The resulting indentation is then examined under a microscope and measured. This measurement is transformed into a Knoop hardness number.[40]

Typical Knoop hardness for resin composite ranges from 22 to 80 and is lower than enamel, which has a Knoop hardness of 343. The Knoop hardness of composites is affected by the type and size of filler dispersed in the resin matrix. Increasing the size of fillers tends to give higher Knoop hardness values.[40]

VISCOSITY

Viscosity can be described as a material's resistance to flow. It is another physical property that should be considered when investigating new formulas of resin composites.

Viscosity is of importance to the dental clinician, because it affects the handling characteristics of restorative materials. It is also related to the success of resin composite restorations.[56] It has been demonstrated in studies that restorations fabricated with low-viscosity resins have a lower incidence of gaps in the marginal area. Low-viscosity composites are better adapted to walls of cavity preparations when compared with resin composites of regular viscosity.[57]

RATIONALE OF THE STUDY

The objective of this project is to investigate the effect of varying the concentrations of TCP-FA fillers on the mechanical properties of an experimental light-cured resin-matrix composite, and to evaluate the potential use of this resin composite as a restorative material or as a pit and fissure sealant. The specific aims are:

1. To determine the effect of various filler levels on the compressive strength.

2. To examine the effect of various filler levels on the degree of conversion.

3. To investigate the effect of various filler levels on the depth of cure.

4. To investigate the effect of various filler levels on the hardness.

5. To determine the effect of various filler levels on the viscosity.

MATERIALS AND METHODS

EXPERIMENTAL TRICALCIUM PHOSPHATE-BASED RESIN

The monomer mixture used for this study was composed of 34.3 percent by mass of ethoxylated bisphenol A dimethacrylate (EBPADMA), which is a low-viscosity base monomer, 34.2 percent by mass hexamethylene dimethacrylate (HmDMA), a hydrophobic diluent monomer, and 30.5 percent by mass of 2-hydroxyethyl methacrylate (HEMA), a very hydrophilic component. The decision to use these resins was in accordance with the findings of Skrtic et al., who found that calcium and phosphate ion release was higher from ACP-filled composites with HEMA/EBPADMA-containing polymers.[18]

The resin was made photo-reactive by the incorporation of 0.20 percent by mass camphorquinone (CQ, Sigma-Aldrich) and 0.80 percent by mass ethyl-4-N, N-dimethalaminobenzoate (4EDMAB) as a photo-initiator and photo-co-initiator, respectively. The CQ was added in a dark room to prevent light from initiating the reaction.

Tricalcium phosphate fillers were provided by Indiana Nanotech. The fillers were surface-treated with fumaric acid (FA). The fillers had an average particle size of 4 um. The monomers were mixed using hand spatulation. Monomers were placed on magnetic stir bars on a magnetic stir plate. After several minutes of rapid mixing, the filler was added in small amounts to ensure incorporation into the monomer mixture. After all fillers were added, the monomer and filler mixtures were left on the magnetic stir plate overnight to ensure homogenization. After that, the initiator and co-initiator were added

in the same manner. Afterwards, the homogenized pastes were kept under vacuum overnight for elimination of the air entrapped during mixing. The fillers were added to the resin matrix by continuous mixing under vacuum to avoid air-bubble entrapment. Resin composites were prepared at four different filler levels by weight percent. The TCP fillers prepared with 30 percent, 40 percent, 50 percent, and 60 percent by weight were mixed with resins of 70 percent, 60 percent, 50 percent, and 40 percent by weight respectively, using hand spatulation. The four filler level groups were labeled A, B, C and D respectively. All four concentration levels of the resin composites were tested to explore the effect of different filler levels on the compressive strength and modulus, degree of conversion, depth of cure, hardness, and viscosity.

COMPRESSIVE STRENGTH AND MODULUS

All specimens were molded and cured thoroughly in cylindrical glass tubes with an internal diameter of approximately 2.4 mm and were cut into approximately 5-mm cylinders with a diamond saw. Specimens were pushed out from the glass tubes, then stored in distilled water at 37ºC for 24 hours. Prior to testing, the specimens' diameter and length were recorded using a digital micrometer. Three measurements for the diameter and length from various areas of the samples were taken, and the mean of these measurements was recorded.

Compression testing was carried out on a universal testing machine (Sintech Renew 1121, Instron Engineering Corp., and Canton, MA) with a cross-head speed of 1mm/min. The compression force was applied axially on the flat end of the cylindrical samples. Five samples from each filler level were tested.

Compressive strength was then calculated using: $\sigma = F/A_o$

Where: $\sigma =$ compressive strength;

$F =$ instantaneous load applied perpendicular to the specimen cross-section;

$A_o =$ the original cross-sectional area before any load is applied.

Strain was calculated using: $\varepsilon = h_i\text{-}h_o/h_o = \Delta h/h_o$

Where: $\varepsilon =$ strain;

$h_o =$ The original height before any load is applied;

$h_i =$ The instantaneous length;

$h_i\text{-}h_o$ is denoted as Δh and is the change in height.

Compression modulus was then calculated using: $E = \sigma/\varepsilon$;

Where $E =$ the compression modulus.

DEGREE OF CONVERSION MEASUREMENT

The degree of conversion was determined with infrared (IR) spectroscopic technique. Specimens 0.15-mm thick and 6 mm in diameter were used. Resin was placed between two Mylar strips with a glass slab beneath and on the top to avoid air entrapment, and then a stainless jig with a 7-mm opening was placed above the assembly. The curing was done through the opening for 20 seconds.

The specimens were placed in a standard FTIR (Fourier transform infrared spectroscopy) sample holder with a 5-mm diameter opening. The IR spectra were acquired in absorbance mode from wave numbers of 1500 to 1700. The area under the peak 1608 was assigned to the aromatic C=C coming from EBPADMA and was used as

the internal standard. The area under the peak 1638 was assigned to the vinyl C=C and will be used to evaluate the degree of conversion. DC was obtained directly from the decrease in the 1638 peak intensity using the following equation:

Degree of conversion:

$$= \frac{\# \text{ of converted} C=C}{\text{Total} \# \text{ of } C=C}$$

$$= \frac{\text{Total} \# \text{ of } C=C - \text{Remaining} \# \text{ of } C=C}{\text{Total} \# \text{ of } C=C}$$

$$= 1 - \frac{\text{Remaining} \# \text{ of } C=C}{\text{Total} \# \text{ of } C=C}$$

$$= 1 - \frac{\text{Cured (area under 1638/area under 1608)}}{\text{Uncured (area under 1638/area under 1608)}}$$

Five measurements were taken for each filler level.

DEPTH OF CURE

A scraping technique was used to evaluate the depth of cure according to the ISO standards for dental resin 4049.[58] Three specimens were made for each group by condensing the respective resin into a Teflon mold, and each measured 4 mm in diameter and 6 mm in depth.

Above the Teflon mold, a 1-mm metal spacer was placed in order to hold the tip of the light-curing unit 1 mm from the surface of the resin. The specimens were light cured with an LED curing unit (DEMI, Kerr) with an average power density output of 1463 mW/cm^2 for 40 seconds. A radiometer was used to check the output consistency between specimens.

Following light curing, the specimens were removed from the mold, the soft uncured resin composite was scraped using a plastic spatula. The height of each specimen

was measured using a digital micrometer in three different areas and the average of the three measurements was recorded. The value was divided by two in order to obtain the ISO 4049 depth of cure. Five measurements were made for each filler level.

KNOOP MICROHARDNESS

A brass mold 4 mm in diameter and 1-mm thick was used to fabricate five specimens from each group. The mold was placed on a Mylar strip over a glass slab. Then, the mold was filled with the material. A second Mylar strip was placed over the uncured resin. A glass slide was placed on top of the second Mylar strip to squeeze out the excess and to provide a smooth surface. The glass slide was removed before irradiation of the material. Then, each specimen was cured for 40 seconds from the top and bottom using an LED curing unit (DEMI, Kerr) with an average power density output of 1463 mW/cm^2. After the curing cycle, the specimens were removed from the mold and were stored in distilled water for 24 h at 37ºC. Knoop microhardness numbers were obtained by a microhardness tester (M-400, Leco Co., St. Joseph, MI). A Knoop diamond tip was used to make three indentations on the top surface of each specimen. The indentations were made using a 50-gf load and a dwell time of 15 seconds. The same specimens were stored again in distilled water for 7 days at 37ºC. The same procedure was repeated to measure the Knoop hardness after 7 days.

VISCOSITY

The viscosities of the experimental resin were determined using a viscometer (DV-II$^+$ Viscometer, Brookfield; Middleboro, MA). Each resin sample was held in place and subjected to shear with a small-diameter spindle (CPE-52) used for higher-viscosity

samples. Three samples from the experimental resin with 30-percent and 40-percent filler levels were tested. A shear rate of 1.2 l/s to 100 1/s was used to determine the viscosity values.

STATISTICAL METHODS

The effects of different concentration levels of the tricalcium phosphate filler (30 percent, 40 percent, 50 percent, and 60 percent by weight) on compressive stress, compressive modulus, degree of conversion, depth of cure, Knoop microhardness, and viscosity were assessed using a one-way analysis of variance (ANOVA) with the level of filler as the main variable. A 5-percent significance level was used for all tests.

RESULTS

COMPRESSIVE STRENGTH AND MODULUS

Mean compressive strength and standard deviations are shown in Table II and Figure 10. Group A showed significantly higher compressive strength values than Group C and Group D.

Mean compressive modulus values and standard deviations for all filler levels are summarized in Table III and Figure 11. Group C revealed significantly higher modulus than all other groups.

DEGREE OF CONVERSION

Conversion values obtained from FTIR are shown in Table IV and Figure 12. Group D revealed significantly higher DC than all other groups. Group A showed the lowest DC value, but it was not statistically different from Group B and Group C.

DEPTH OF CURE

Depths of cure measurements are shown in Table V and Figure 13. Group A samples had a significantly greater depth of cure than samples from all other groups. Group D had the shallowest depth of cure but was not significantly different from Group C.

HARDNESS

Mean Knoop hardness values and standard deviations after 24 hours storage in distilled water are presented in Table VI and Figure 14. Group A had the highest KHN

and it was statistically different from Group C and Group D. Moreover, Group D resulted in significantly the lowest KHN.

Mean KHN and standard deviations after 7 days storage in distilled water are summarized in Table VII and Figure 14. Group A showed the highest KHN and it was significantly different from the other groups. Group D resulted in the lowest KHN but was not statistically different from Group C. The difference between before- and after-storage hardness was statistically significant for all groups except for Group A.

VISCOSITY

Mean viscosity and standard deviations for Group A and Group B are shown in Table VIII and Table IX, respectively. Viscosity was plotted against shear rate in Figure 15. At a shear rate of 40, Group B showed viscosity values that are statistically higher than Group A. Viscosity was investigated for Group A and Group B only, because the other groups were too viscous to be tested. At a shear rate of 40, Groups A and B reached a Newtonian viscosity of 114 mPa and 413 mPa respectively.

TABLES AND FIGURES

TABLE I

Formulation of composite resin matrix

Component	Chemical Compound	Abbreviation	Percent
Monomer	Ethoxylated bisphenol A dimethacrylate	EBPADMA	34.3%
	Hexamethylene dimethacrylate	HmDMA	34.2%
	2-hydroxyethyl methacrylate	HEMA	30.5%
photo-initiator	Camphorquinone	CQ	0.2%
photo-co-initiator	Ethyl-4-N,N-dimethylaminobenzoate	4EDMAB	0.8%

TABLE II

Mean compressive strength and standard deviation (SD)

Group	TCP-FA	N	Min (MPa)	Max (MPa)	Mean (SD)
A	30%	5	120.88	172.53	143.01 (20.16)
B	40%	5	109.18	155.56	131.35 (19.35)
C	50%	6	78.85	146.11	99.61 (24.68)
D	60%	5	29.99	66.58	52.81 (13.69)

TABLE III

Mean compressive modulus and standard deviation (SD)

Group	TCP-FA	N	Min (MPa)	Max (MPa)\	Mean (SD)
A	30%	5	755.61	945.62	824.99 (80.82)
B	40%	5	763.97	974.72	823.15 (88.30)
C	50%	6	936.20	1597.35	1157.15 (227.32)
D	60%	5	524.01	817.84	681.34 (138.20)

TABLE IV

Mean degree of conversion and standard deviation (SD)

Group	TCP-FA	N	Min	Max	Mean (SD)
A	30%	9	0.417	0.485	0.452 (0.024)
B	40%	9	0.430	0.542	0.474 (0.037)
C	50%	9	0.420	0.533	0.489 (0.045)
D	60%	9	0.495	0.590	0.533 (0.030)

TABLE V

Mean depth of cure and standard deviation (SD)

Group	TCP-FA	N	Min (mm)	Max (mm)	Mean (SD)
A	30%	9	0.53	0.915	0.7 (0.1)
B	40%	9	0.495	0.6	0.6 (0.0)
C	50%	9	0.405	0.49	0.4 (0.0)
D	60%	9	0.28	0.56	0.4(0.1)

TABLE VI

Mean Knoop hardness number after
24 hours and standard deviation (SD)

Group	TCP-FA	N	Min	Max	Mean (SD)
A	30%	5	6	9.8	8.35 (1.18)
B	40%	5	6.5	9.5	8.11 (0.86)
C	50%	5	4.7	7.4	6.37 (0.80)
D	60%	5	3.6	7.7	5.25 (1.11)

TABLE VII

Mean Knoop hardness number after
7 days and standard deviation (SD)

Group	TCP-FA	N	Min	Max	Mean (SD)
A	30%	5	5.6	10.8	7.81 (1.17)
B	40%	5	5.9	8.6	6.98 (0.86)
C	50%	5	3.8	5.2	4.37 (0.34)
D	60%	5	2.9	4.4	3.65 (0.44)

TABLE VIII

Group A mean viscosity and standard deviation (SD)

N	Shear rate (1/s)	Min (mPa.s)	Max (mPa.s)	Mean (SD)
3	100	104	128	115 (10)
3	60	131	155	141 (10)
3	40	153	178	168 (11)
3	24	212	271	246 (25)
3	20	294	308	302 (6)
3	12	501	562	524 (27)
3	10	603	714	643 (51)
3	8	716	786	744 (30)
3	6	915	1048	985 (55)
3	5	1001	1213	1083 (93)
3	4	1237	1381	1293 (63)
3	3	1445	1711	1546 (118)
3	2	1627	2902	2074 (586)
3	1.2	2697	5533	3673 (1316)

TABLE IX

Group B mean viscosity and standard deviation (SD)

N	Shear rate (1/s)	Min (mPa.s)	Max (mPa.s)	Mean (SD)
3	40	405	419	414 (8)
3	24	524	536	532 (7)
3	20	591	606	598 (8)
3	12	801	820	808 (10)
3	10	897	919	906 (12)
3	8	1039	1063	1053 (13)
3	6	1252	1280	1271 (16)
3	5	1421	1455	1442 (19)
3	4	1655	1697	1683 (24)
3	3	2040	2089	2067 (25)
3	2	2706	2809	2750 (53)
3	1.2	3906	4092	4009 (95)
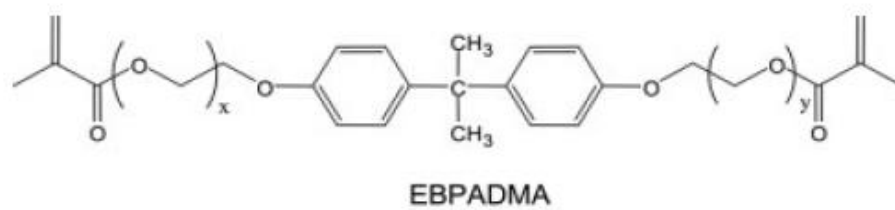
FIGURE 1. Ethoxylated bisphenol A dimethacrylate (EBPADMA).
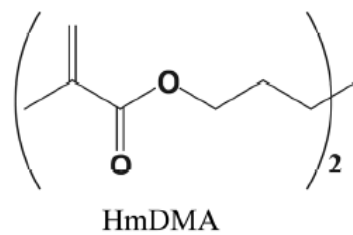
FIGURE 2. Hexamethylene dimethacrylate (HmDMA).
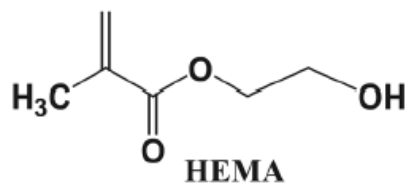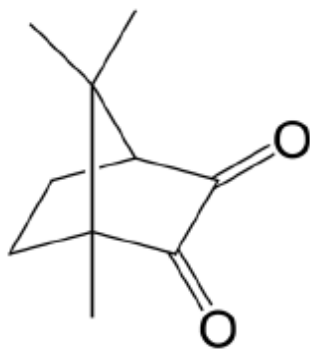
FIGURE 3. 2-hydroxyethyl methacrylate (HEMA).
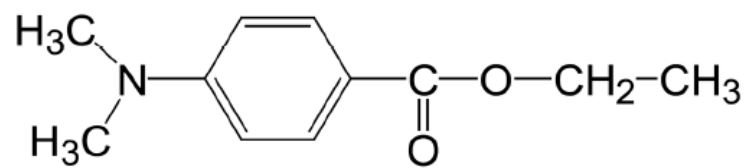

FIGURE 4. Camphorquinone (CQ).

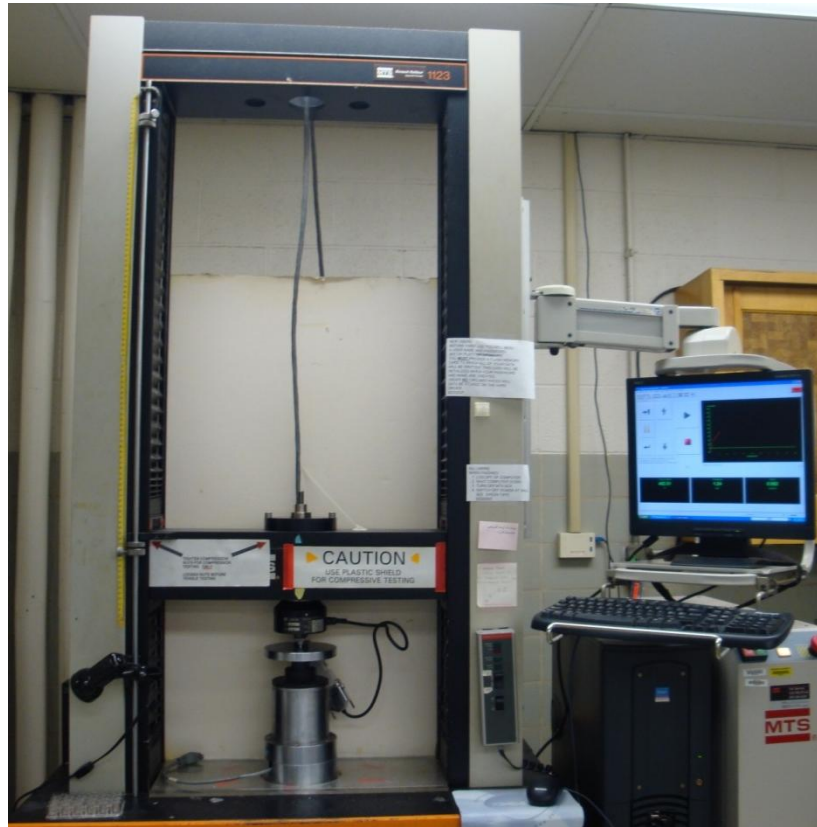FIGURE 5. Ethyl-4-N,N-dimethylaminobenzoate (4EDMAB).

FIGURE 6.   Universal testing machine (Sintech Renew 1121, Instron Engineering Corp., Canton, MA).

FIGURE 7. Fourier transform infrared spectroscopy (FTIR).

FIGURE 8. Microhardness tester (M-400, Leco Co., St. Joseph, MI).

FIGURE 9. Viscometer (DV-II $^{+}$ Viscometer, Brookfield, Middleboro, MA).
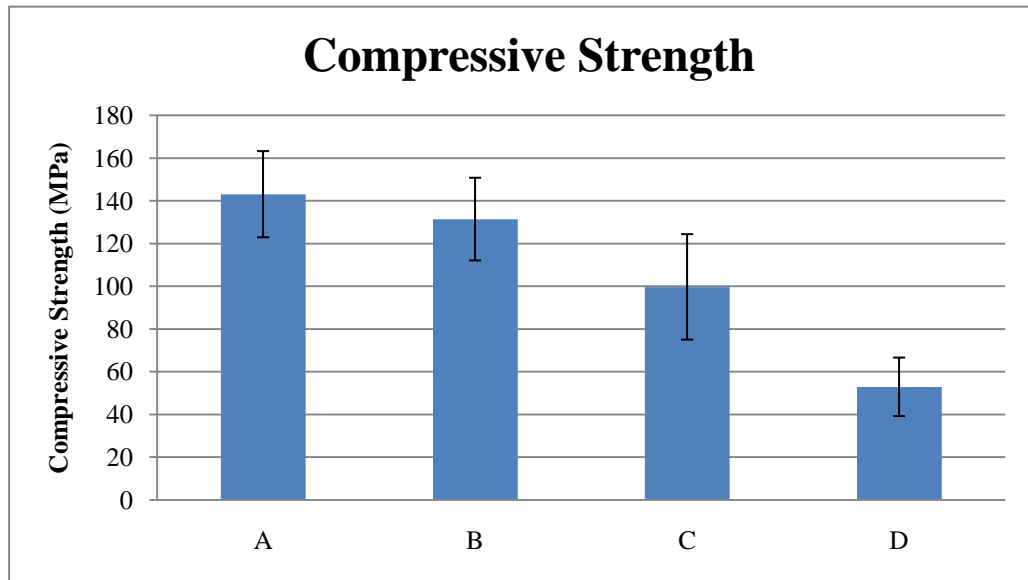
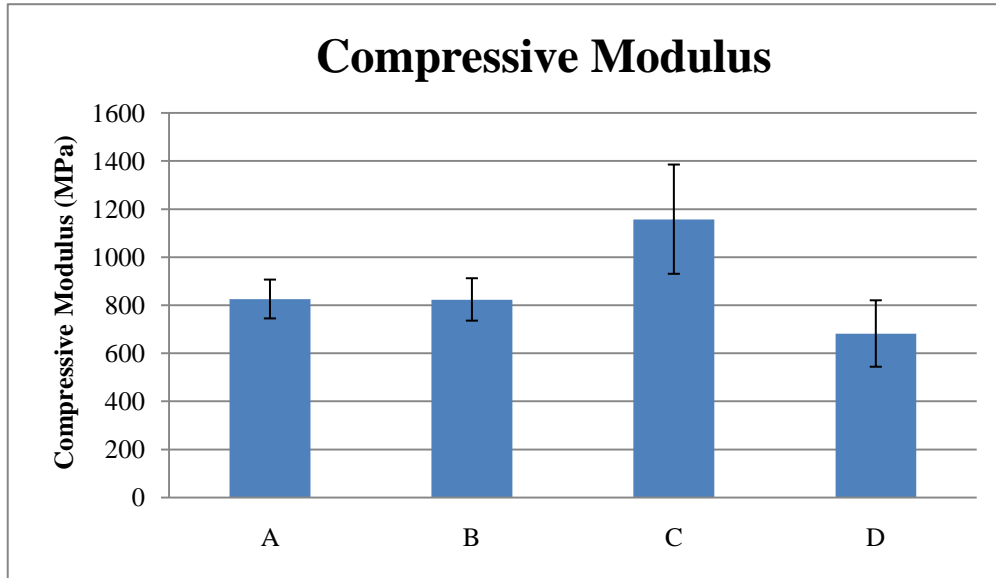FIGURE 10. Mean compressive strength and standard deviation.

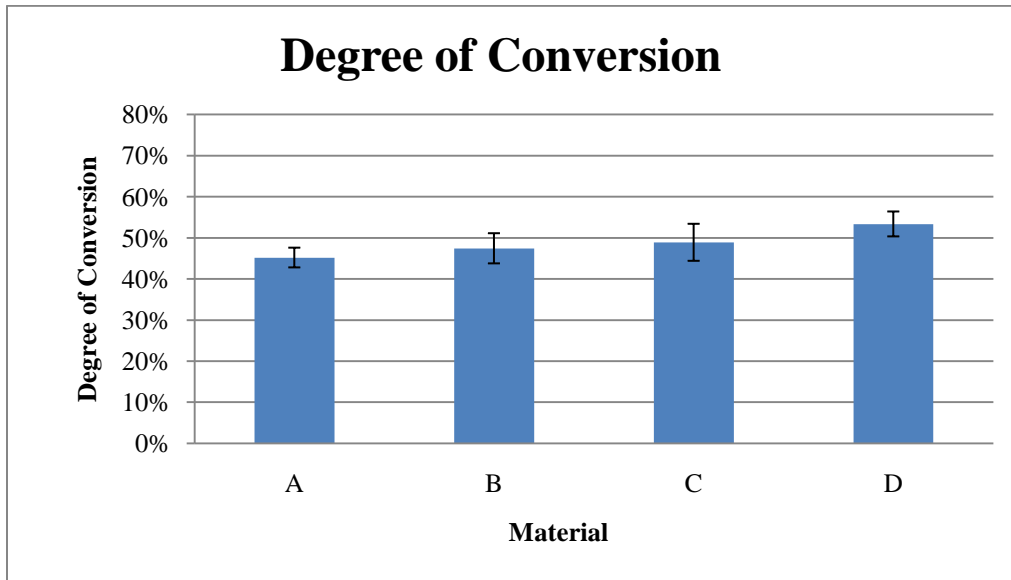FIGURE 11. Mean compressive modulus and standard deviation.

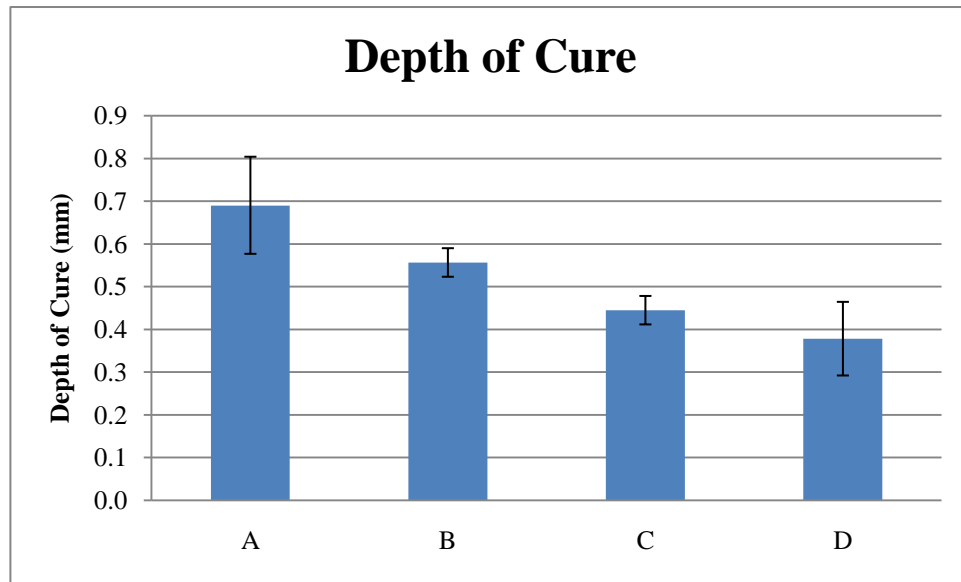FIGURE 12. Mean degree of conversion and standard deviation.

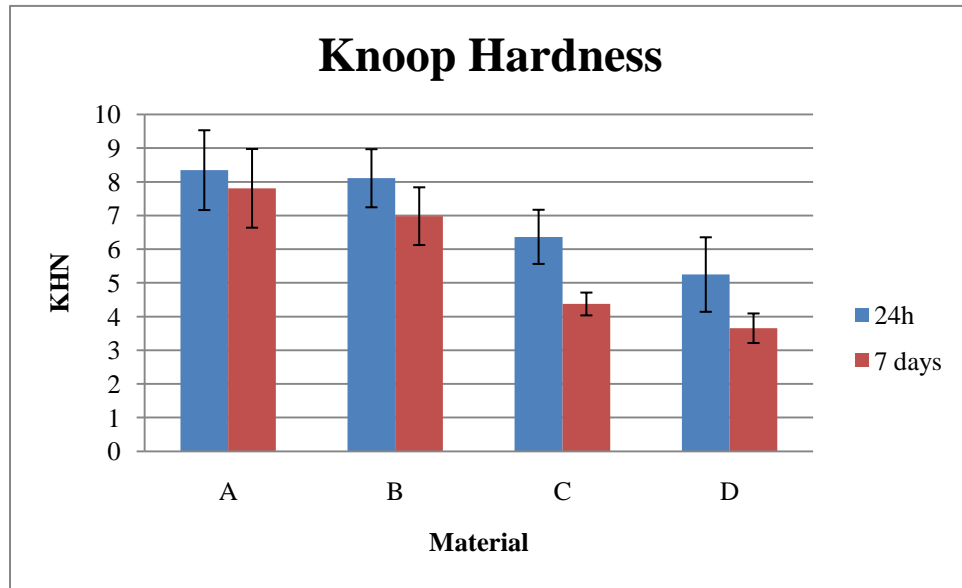FIGURE 13. Mean depth of cure and standard deviation.

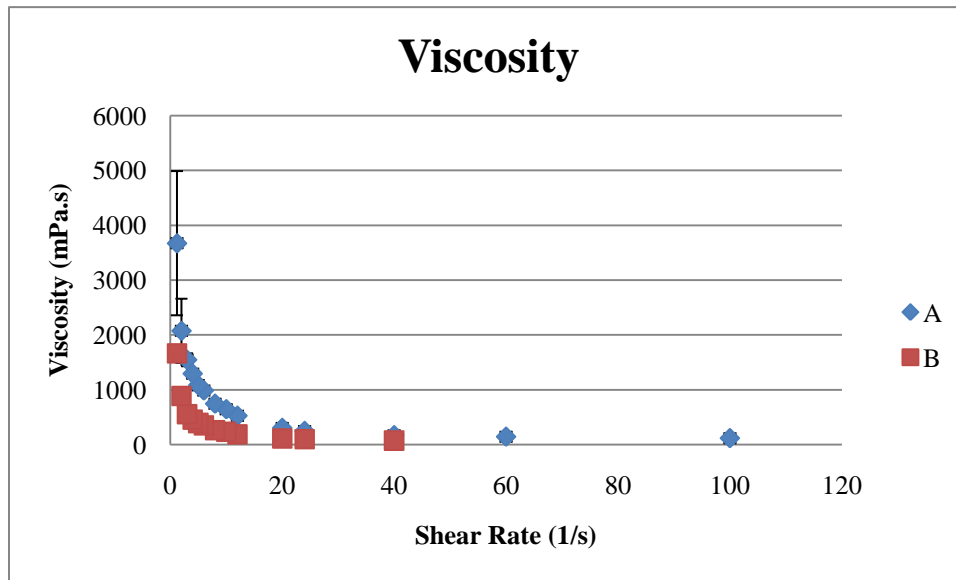FIGURE 14. Mean Knoop hardness number and standard deviation.

FIGURE 15. Mean viscosity (in millipascal seconds) and standard deviation.

DISCUSSION

The majority of light-activated dental resin composites on the market consist of a resin-matrix phase reinforced with glass. Recently presenting an alternative, researchers developed a bioactive resin composite capable of remineralizing demineralized tooth structure. This new composite's remineralizing property is derived from the amorphous calcium phosphate (ACP) dispersed into the resin matrix. Several studies have confirmed that ACP is a precursor to apatite formation. However, the substitution of ACP for glass fillers has some adverse effects on the mechanical strength. Several studies investigated various methods and technologies in an attempt to improve the strength of ACP-filled resin. Some focused on the function of matrix composition on the mechanical properties of ACP-filled dental resin.[16, 45, 59] Other investigators incorporated different calcium-releasing fillers such as tricalcium phosphate (TCP) that could produce stronger composites.

In this study, the mechanical properties of a TCP-FA resin composite were assessed. The resin-matrix system utilized had the same monomer combination as a previously investigated ACP-filled resin formulated by Skrtic and colleagues. The resin system incorporated EBPADMA as the base monomer. HmDMA was substituted for TEGDMA. Furthermore, HEMA was added as a co-monomer with a potential for adhesion.[18]

COMPRESSIVE STRENGTH AND MODULUS

Compressive strength is of major importance because many masticatory forces are compressive in nature.[60] In the current study, the compressive strength values are presented in Table II and were lower than those of commercial flowable resin composites. A typical value for a flowable resin composite ranges between 210 MPa and 300 MPa.[40] An increase in the compressive modulus was observed when the TCP-FA filler weight percent was increased. However, the compressive strength did not improve with the addition of more filler to the matrix, even though the compressive modulus continued to increase. This decrease in compressive strength with increased filler loading was contrary to expectations. With conventional resin composite systems, increased filler content is usually responsible for improved mechanical properties. The reduction in compressive strength of TCP-FA composites can be attributed to the poor bonding at the filler/matrix interface. In addition, TCP-FA particles had a tendency to agglomerate, affecting the particle size distribution into the resin matrix and consequently the strength. It is therefore necessary to investigate the particle-size distribution and find better ways to facilitate the distribution of the particles. Previous research with EBPADMA-based matrices that had ACP fillers recommended fine tuning of ACP fillers in an attempt to improve particle distribution. The monomer combination used may have also contributed to the inferior strength.

DEGREE OF CONVERSION

The degree of conversion (DC) is related to several physical and biological properties of monomer-based dental materials. In resin systems with low DC values,

monomers have the potential to leach into the oral cavity to jeopardize biocompatibility.[45] Polymerization shrinkage is also directly related to DC. Any increase in monomer conversion increases shrinkage values. Therefore, it is important to evaluate DC when polymerization contraction stresses of different resin systems are to be investigated and compared.[61]

In this study, FTIR was used to obtain DC values. This technique has many advantages. One is its ability to detect minor differences with a minimum number of samples. From Table IV, it can be seen that all test resin groups have low DC values when compared with Bis-GMA/TEGDMA resin systems that usually yield DC values of approximately 75 percent.[18] Also, the DC of Group D was significantly higher than all other test groups. The resin monomer combination used in the present study was previously tested by Skrtic et. al with the type of filler representing the only difference between the two studies. He obtained DC values that exceeded those typically obtained with Bis-GMA/TEGDMA copolymers. The average DC values in Skrtic's study were between 74 percent and 91 percent. One possible explanation for the low DC in the present study could be that the TCP-FA conversion to apatite has a negative effect on DC.

DEPTH OF CURE

In this study, depth of cure was tested according to ISO 4049. None of the experimental groups met the 1.5 mm ISO curing depth requirements for light-cured resin-based restorative materials (Table V).

When a resin composite is irradiated by a blue curing light, the light is scattered by the filler particles. As light travels through the resin matrix, its intensity is attenuated, thereby reducing the curing effectiveness. The shade of a resin composite has been proven to affect the number of photons arriving to the surface of the resin. Darker shades tend to absorb the lowest number of photons.[62] For this reason, the initial shade of the resin composite has a significant role in determining the curing behavior.[62] The TCP-FA fillers may have contributed to the darker shade of the composite. This is a possible explanation for the low depth of cure values. Another possible explanation is the agglomeration of the filler particles that could have impaired light transmission into the matrix and resulted in shallow curing depths.

MICROHARDNESS

Resin composite becomes hard after light curing and polymerization. Hardness tests have been used as an indirect way to evaluate the quality of polymerization. The hardness is primarily determined by the type and amount of filler and monomers incorporated in the resin.[62, 63]

In the present study, KHN was determined for each specimen before and after storage in distilled water. Before water storage, group A showed the highest KHN, which was significantly different from the other groups. The KHN of Group D was significantly lower than the KHN for all other groups. After water storage, Group A still showed the highest KHN, and Group D, the lowest.

Water is known to penetrate the polymer network and thereby cause dissolution of unreacted monomers and polymer chains. Polymers with low cross-linking density

should exhibit low KHN. EBPADMA-based polymers are known to have lower cross-linking compared with Bis-GMA-based polymers. As a result, EBPADMA monomers tend to form open network structures that facilitate water diffusion. Additionally, HEMA, one of the components of the resin composite used in this study, is known to be hydrophilic.[18] Its hydrophilicity allowed more water absorption and contributed to the low KHN values.

VISCOSITY

Resin composites exhibit complex rheological properties. They have the potential to behave like solids or liquids depending on the shear applied. Dental resins typically demonstrate a shear-thinning behavior (i.e. the viscosity decreases as the shear rate increases). It is very important for the dental practitioner to understand the rheological properties of resin when restoring teeth, because such knowledge helps to facilitate more appropriate handling.[56]

Several studies investigated the rheological properties of commercially available resins. These studies found that flow characteristics of dental resins of the same type can vary greatly.[56]

Bis-GMA monomer is the major constituent of many composite resins. Cured Bis-GMA has good mechanical properties. However, the main drawback is of Bis-GMA is its high viscosity. Diluting Bis-GMA with low viscosity monomers such as TEGDMA leads to major deficiencies, such as an increase in polymerization shrinkage.[64]

In the present study, increasing the filler content significantly increased the viscosity. The degree of conversion of composite resin is highly influenced by its initial

viscosity. The high-viscosity values obtained in this study could probably explain the low conversion readings. High-viscosity monomers have limited flexibility that limits conversion.

SUMMARY AND CONCLUSION

The objective of this study was to investigate the possible use of a tricalcium phosphate- (TCP)-filled composite resin as a tooth restorative material. Four groups of experimental light-cured resin composite were made by varying only the TCP filler level (30 percent, 40 percent, 50 percent, and 60 percent by weight). Compressive strength, modulus, degree of conversion, depth of cure, hardness, and viscosity were investigated. From the data collected, the results can be summarized in the following:

1. Resin composites with 30-percent TCP filler showed the highest compressive strength and hardness values. Also, this group showed the lowest degree of conversion.

2. Resin composites with 60-percent TCP filler showed the highest degree of conversion. However, this group showed the lowest compressive strength, depth of cure, and hardness.

3. Resin composites with 50-percent filler showed the highest compressive modulus.

4. Resin composites with 40-percent filler showed higher viscosity values than resin composites with 30-percent filler.

In conclusion, increasing the filler level significantly reduced the compressive strength, hardness, and depth of cure, but increased the degree of conversion. Also, resin composites with the least filler level (30 percent) had the highest compressive strength, depth of cure, and hardness. From these results, it can be concluded that the experimental TCP-filled resin used in this study cannot be used as restorative material due to its poor

mechanical properties. Further investigation is recommended to determine the effect of adding TCP to resin with different monomers. Also, including silica in the resin formulation could improve the mechanical performance. Additional mechanical milling of the filler and the addition of surfactant could result in a more homogenous composite. In turn, fewer voids could be incorporated in the composite and account for a subsequent improvement in its strength.

REFERENCES

1. Deliperi S, Bardwell DN. An alternative method to reduce polymerization shrinkage in direct posterior composite restorations. J Am Dent Assoc 2002;133(10):1387-98.

2. Roulet JF. The problems associated with substituting composite resins for amalgam: a status report on posterior composites. J Dent 1988;16(3):101-13.

3. Cassoni A, Ferla Jde O, Shibli JA, Kawano Y. Knoop microhardness and FT-Raman spectroscopic evaluation of a resin-based dental material light-cured by an argon ion laser and halogen lamp: an in vitro study. Photomed Laser Surg 2008;26(6):531-9.

4. Ilie N, Hickel R. Investigations on mechanical behaviour of dental composites. Clin Oral Investig 2009;13(4):427-38.

5. Fong H, Dickens SH, Flaim GM. Evaluation of dental restorative composites containing polyhedral oligomeric silsesquioxane methacrylate. Dent Mater 2005;21(6):520-9.

6. Ferracane JL. Current trends in dental composites. Crit Rev Oral Biol Med 1995;6(4):302-18.

7. Sarrett DC. Clinical challenges and the relevance of materials testing for posterior composite restorations. Dent Mater 2005;21(1):9-20.

8. Mjor IA, Moorhead JE, Dahl JE. Reasons for replacement of restorations in permanent teeth in general dental practice. Int Dent J 2000;50(6):361-6.

9. Hsu CY, Donly KJ, Drake DR, Wefel JS. Effects of aged fluoride-containing restorative materials on recurrent root caries. J Dent Res 1998;77(2):418-25.

10. Wiegand A, Buchalla W, Attin T. Review on fluoride-releasing restorative materials--fluoride release and uptake characteristics, antibacterial activity and influence on caries formation. Dent Mater 2007;23(3):343-62.

11. Anusavice KJ, Zhang NZ, Shen C. Effect of CaF_2 content on rate of fluoride release from filled resins. J Dent Res 2005;84(5):440-4.

12. Ellakuria J, Triana R, Minguez N, et al. Effect of one-year water storage on the surface microhardness of resin-modified versus conventional glass-ionomer cements. Dent Mater 2003;19(4):286-90.

13. Dickens SH, Flaim GM, Takagi S. Mechanical properties and biochemical activity of remineralizing resin-based Ca-PO4 cements. Dent Mater 2003;19(6):558-66.

14. Skrtic D, Hailer AW, Takagi S, Antonucci JM, Eanes ED. Quantitative assessment of the efficacy of amorphous calcium phosphate/methacrylate composites in remineralizing caries-like lesions artificially produced in bovine enamel. J Dent Res 1996;75(9):1679-86.

15. O'Donnell JN, Antonucci JM, Skrtic D. Amorphous calcium phosphate composites with improved mechanical properties. J Bioact Compat Polym 2006;21(3):169-84.

16. Skrtic D, Antonucci JM, Eanes ED, Eidelman N. Dental composites based on hybrid and surface-modified amorphous calcium phosphates. Biomaterials 2004;25(7-8):1141-50.

17. Schumacher GE, Antonucci JM, O'Donnell JN, Skrtic D. The use of amorphous calcium phosphate composites as bioactive basing materials: their effect on the strength of the composite/adhesive/dentin bond. J Am Dent Assoc 2007;138(11):1476-84.

18. Skrtic D, Antonucci JM. Dental composites based on amorphous calcium phosphate - resin composition/physicochemical properties study. J Biomater Appl 2007;21(4):375-93.

19. Skrtic D, Stansbury JW, Antonucci JM. Volumetric contraction and methacrylate conversion in photo-polymerized amorphous calcium phosphate/methacrylate composites. Biomaterials 2003;24(14):2443-9.

20. Antonucci JM, O'Donnell JN, Schumacher GE, Skrtic D. Amorphous calcium phosphate composites and their effect on composite-adhesive-dentin bonding. J Adhes Sci Technol 2009;23(7):1133-47.

21. Regnault WF, Icenogle TB, Antonucci JM, Skrtic D. Amorphous calcium phosphate/urethane methacrylate resin composites. (Pt 1). Physicochemical characterization. J Mater Sci Mater Med 2008;19(2):507-15.

22. Lee SY, Regnault WF, Antonucci JM, Skrtic D. Effect of particle size of an amorphous calcium phosphate filler on the mechanical strength and ion release of polymeric composites. J Biomed Mater Res B Appl Biomater 2007;80(1):11-7.

23. Nakagawa A, Matsuya S, Takeuchi A, Ishikawa K. Comparison of the effects of added alpha- and beta- tricalcium phosphate on the basic properties of apatite cement. Dent Mater J 2007;26(3):342-7.

24. Gera I, Dori F, Keglevich T, Anton S, Szilagyi E, Windisch P. [Experience with the clinical use of beta-tri-calcium phosphate (Cerasorb) as a bone replacement graft material in human periodontal osseous defects]. Fogorv Sz 2002;95(4):143-7.

25. Ridgway HK, Mellonig JT, Cochran DL. Human histologic and clinical evaluation of recombinant human platelet-derived growth factor and beta-tricalcium phosphate for the treatment of periodontal intraosseous defects. Int J Periodontics Restorative Dent 2008;28(2):171-9.

26. Franchi M, Ruggeri A. [beta-TCP and beta-TCMP osteoregenerators. New perspectives]. Dent Cadmos 1989;57(19):54-8, 61-4, 67.

27. Zhang F, Chang J, Lu J, Lin K, Ning C. Bioinspired structure of bioceramics for bone regeneration in load-bearing sites. Acta Biomater 2007;3(6):896-904.

28. Ghosh SK, Nandi SK, Kundu B, et al. In vivo response of porous hydroxyapatite and beta-tricalcium phosphate prepared by aqueous solution combustion method and comparison with bioglass scaffolds. J Biomed Mater Res B Appl Biomater 2008;86(1):217-27.

29. Karlinsey RL, Mackey AC, Walker ER, Frederick KE. Preparation, characterization and in vitro efficacy of an acid-modified beta-TCP material for dental hard-tissue remineralization. Acta Biomater 2009.

30. Hiiri A, Ahovuo-Saloranta A, Nordblad A, Makela M. Pit and fissure sealants versus fluoride varnishes for preventing dental decay in children and adolescents. Cochrane Database Syst Rev;3:CD003067.

31. Haugejorden O, Magne Birkeland J. Ecological time-trend analysis of caries experience at 12 years of age and caries incidence from age 12 to 18 years: Norway 1985-2004. Acta Odontol Scand 2006;64(6):368-75.

32. Oba AA, Dulgergil T, Sonmez IS, Dogan S. Comparison of caries prevention with glass ionomer and composite resin fissure sealants. J Formos Med Assoc 2009;108(11):844-8.

33. Davidenko N, Cohen ME, Diaz JM, Sastre R. Development of new photopolymerizable dental sealants. J Biomater Sci Polym Ed 1998;9(9):891-903.

34.   Griffin SO, Gray SK, Malvitz DM, Gooch BF. Caries risk in formerly sealed teeth. J Am Dent Assoc 2009;140(4):415-23.

35.   Ripa LW. Sealants revisted: an update of the effectiveness of pit-and-fissure sealants. Caries Res 1993;27 Suppl 1:77-82.

36.   Handelman SL, Shey Z. Michael Buonocore and the Eastman Dental Center: a historic perspective on sealants. J Dent Res 1996;75(1):529-34.

37.   Ikeda I, Otsuki M, Sadr A, Nomura T, Kishikawa R, Tagami J. Effect of filler content of flowable composites on resin-cavity interface. Dent Mater J 2009;28(6):679-85.

38.   Naorungroj S, Wei HH, Arnold RR, Swift EJ, Jr., Walter R. Antibacterial surface properties of fluoride-containing resin-based sealants. J Dent;38(5):387-91.

39.   Silva KG, Pedrini D, Delbem AC, Ferreira L, Cannon M. In situ evaluation of the remineralizing capacity of pit and fissure sealants containing amorphous calcium phosphate and/or fluoride. Acta Odontol Scand;68(1):11-8.

40.   Powers J, Sakaguchi R, eds. Craig's restorative dental materials. 12th ed. St. Louis: Mosby, 2006.

41.   Braga RR, Ballester RY, Ferracane JL. Factors involved in the development of polymerization shrinkage stress in resin-composites: a systematic review. Dent Mater 2005;21(10):962-70.

42.   Rodrigues Junior SA, Zanchi CH, Carvalho RV, Demarco FF. Flexural strength and modulus of elasticity of different types of resin-based composites. Braz Oral Res 2007;21(1):16-21.

43.   Dewaele M, Truffier-Boutry D, Devaux J, Leloup G. Volume contraction in photocured dental resins: the shrinkage-conversion relationship revisited. Dent Mater 2006;22(4):359-65.

44.   Lovell LG, Newman SM, Bowman CN. The effects of light intensity, temperature, and comonomer composition on the polymerization behavior of dimethacrylate dental resins. J Dent Res 1999;78(8):1469-76.

45.   Skrtic D, Antonucci JM, Liu DW. Ethoxylated bisphenol dimethacrylate-based amorphous calcium phosphate composites. Acta Biomater 2006;2(1):85-94.

46.   Eliades GC, Vougiouklakis GJ, Caputo AA. Degree of double bond conversion in light-cured composites. Dent Mater 1987;3(1):19-25.

47. Ogunyinka A, Palin WM, Shortall AC, Marquis PM. Photoinitiation chemistry affects light transmission and degree of conversion of curing experimental dental resin composites. Dent Mater 2007;23(7):807-13.

48. Vandewalle KS, Ferracane JL, Hilton TJ, Erickson RL, Sakaguchi RL. Effect of energy density on properties and marginal integrity of posterior resin composite restorations. Dent Mater 2004;20(1):96-106.

49. Watts DC, Amer O, Combe EC. Characteristics of visible-light-activated composite systems. Br Dent J 1984;156(6):209-15.

50. Chung KH. The relationship between composition and properties of posterior resin composites. J Dent Res 1990;69(3):852-6.

51. Ramp LC, Broome JC, Ramp MH. Hardness and wear resistance of two resin composites cured with equivalent radiant exposure from a low irradiance LED and QTH light-curing units. Am J Dent 2006;19(1):31-6.

52. Asmussen E. Restorative resins: hardness and strength vs. quantity of remaining double bonds. Scand J Dent Res 1982;90(6):484-9.

53. Ferracane JL. Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. Dent Mater 1985;1(1):11-4.

54. Bouschlicher MR, Rueggeberg FA, Wilson BM. Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions. Oper Dent 2004;29(6):698-704.

55. DeWald JP, Ferracane JL. A comparison of four modes of evaluating depth of cure of light-activated composites. J Dent Res 1987;66(3):727-30.

56. Beun S, Bailly C, Dabin A, Vreven J, Devaux J, Leloup G. Rheological properties of experimental Bis-GMA/TEGDMA flowable resin composites with various macrofiller/microfiller ratio. Dent Mater 2009;25(2):198-205.

57. Fruits TJ, VanBrunt CL, Khajotia SS, Duncanson MG, Jr. Effect of cyclical lateral forces on microleakage in cervical resin composite restorations. Quintessence Int 2002;33(3):205-12.

58. Moore BK, Platt JA, Borges G, Chu TM, Katsilieri I. Depth of cure of dental resin composites: ISO 4049 depth and microhardness of types of materials and shades. Oper Dent 2008;33(4):408-12.

59. Boskey AL. Amorphous calcium phosphate: the contention of bone. J Dent Res 1997;76(8):1433-6.

60.   Silva CM, Dias KR. Compressive strength of esthetic restorative materials polymerized with quartz-tungsten-halogen light and blue LED. Braz Dent J 2009;20(1):54-7.

61.   Al-Shammari M. The effect of inhibitor concentration on polymerization characteristics of light-cured resin composites. [Thesis]. Indianapolis: Indiana University School of Dentistry; 2007.

62.   Jeong TS, Kang HS, Kim SK, Kim S, Kim HI, Kwon YH. The effect of resin shades on microhardness, polymerization shrinkage, and color change of dental composite resins. Dent Mater J 2009;28(4):438-45.

63.   Cesar PF, Miranda WG, Jr., Braga RR. Influence of shade and storage time on the flexural strength, flexural modulus, and hardness of composites used for indirect restorations. J Prosthet Dent 2001;86(3):289-96.

64.   Peutzfeldt A. Resin composites in dentistry: the monomer systems. Eur J Oral Sci 1997;105(2):97-116.

ABSTRACT

THE EFFECT OF FILLER ON THE MECHANICAL PROPERTIES OF A

NOVEL RESIN-BASED CALCIUM PHOSPHATE CEMENT

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Several studies have found that resin-based amorphous calcium phosphate (ACP) composites can function well for applications that do not require high mechanical demand. Milled tricalcium phosphate (TCP), a new calcium-phosphate-releasing material, is crystalline in nature, suggesting it to be strong. In the present study, we investigated the use of a TCP-filled composite resin as a possible tooth restorative-material. An experimental TCP-based composite was prepared using monomer with a mixture of 34.3 percent by mass of EBPADMA, 34.2 percent by mass of HmDMA, and 30.5 percent by mass of HEMA. TCP fillers were added to the monomer mixture at different levels (30 percent, 40 percent, 50 percent, and 60 percent by weight). A

universal testing machine (Sintech Renew 1121; Instron Engineering Corp., Canton, MA) was used to measure the compressive strength and modulus. FTIR was used to measure the degree of conversion. The depth of cure was determined according to the ISO standards for dental resin 4049 using the scrapping technique. Knoop hardness numbers were obtained by a microhardness tester (M-400; Leco Co., St. Joseph, MI). The viscosities of the experimental resin were determined in a viscometer (DV-II+ Viscometer; Brookfield, Middleboro, MA). The data were analyzed using a one-way analysis of variance (ANOVA). A 5-percent significance level was used for all the tests.

Resin composites with 30-percent TCP filler showed the highest compressive strength and hardness values. Also, this group showed the lowest degree of conversion. Resin composites with 60-percent TCP filler showed the highest degree of conversion. However, this group showed the lowest compressive strength, depth of cure, and hardness. Resin composites with 50-percent filler showed the highest compressive modulus. Resin composites with 40-percent filler showed higher viscosity values than resin composites with 30-percent filler.

In conclusion, increasing the filler level significantly reduced the compressive strength, hardness, and depth of cure, but increased the degree of conversion. Also, resin composites with the lowest filler level (30 percent) had the highest compressive strength, depth of cure, and hardness. From these results, it can be concluded that the experimental TCP-filled resin used in this study cannot be used as restorative material.

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