

MASS AND VOLUME CHANGES OF NOVEL CHLORHEXIDINE, POLYLYSINE, AND COMPOSITES BASED ON CALCIUM PHOSPHATE

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ABSTRACT

OBJECTIVE: To determine how the mass and volume of experimental composites altered following immersion in distilled water (DW) and a simulated bodily fluid (SBF).

METHODS: The factors researched comprised the use of either adhesive monomer (4-methacryloyloxyethyl trimellitate anhydride) or Chlorhexidine level, Calcium Phosphates (CaP) level, Polylysine level, and Hydroxy Ethyl Methacrylate (2-HEM) level. Circle plates of 10 mm in diameter and 1 mm in thickness were created. Gravimetric measurements of the mass and volume changes in DW and SBF were made.

RESULTS: The average mass increase was ~2-5% and ~1-4% in DW and SBF respectively. On adding CaP, the average increase was ~6, 3.5, and 2% with 20, 10, and 0wt% of reactive CaP fillers. The mass change with high levels of Polylysine, and chlorhexidine (5 wt%) was on average ~1% higher than with low levels (0.5, and 0wt% respectively). Mass changes with the use of hydrophilic monomer 2-HEM were ~0.5-1% greater than with the adhesive monomer. In comparison to SBF, 12% was the highest volume increase with 20 wt% CaP in DW. The ultimate volume changes for 10 wt% CaP in DW & SBF at 12 weeks was 7.5% and 5% respectively. Overall volume changes in DW and SBF with 0% by weight CaP were 4% and 2% respectively.

CONCLUSION: Mass and volume changes was highest in DW as compared to SBF, while the composites showed the potential to release over period of time. This will make the composites ideal for filling the gaps created because of polymerization shrinkage.

KEY WORDS: Chlorhexidine (MeSH); Polylysine (MeSH); Calcium phosphate (Non-MeSH); Dental composites (Non-MeSH); Water sorption (Non-MeSH); Volume change (Non-MeSH); Mass change (Non-MeSH)

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The principal drawbacks of composite restorations are as follows: polymerization shrinkage, difficult bonding methods to dentine, and brittle fracture.² Composites lack antibacterial characteristics and are more likely than other restorative materials to acquire biofilm and plaque in vivo.^{3,4} If the water sorption compensates for the polymerization shrinkage during the polymerization process, it might be advantageous.⁵ The degree of expansion varies among different materials, which might have significant therapeutic implications, particularly inside closed cavities. Overexpansion may potentially create severe complications; such as tooth fracture. Hygroscopic expansion produces internal non-functional stresses that will radiate outward along the tooth structure's walls.⁶ According to studies, such overcompensation may result in internal cracks and cusp fractures in materials.⁷ The therapeutic outcome of the restoration depends on a balance between shrinking and expansion. With the addition of Calcium Phosphates (CaP), Polylysine (PLYS), and Chlorhexidine (CHEX) the added benefits of remineralizing, and antibacterial potential will be added into composite, which will increase the restoration life.

The current composite materials have high polymerization shrinkage, which leads to microleakage and as a result failure of restoration. This is the main disadvantage that is associated with

INTRODUCTION

Dental composites currently lack remineralizing and anti-bacterial properties. This makes the restorative materials vulnerable to micro leakage, and tooth decay. Which ultimately increase the burden on the health care system. Dental composites are restorative materials that are tooth-colored. A resin matrix and inorganic fillers comprise the majority of dental composites. Fillers can be glass or other materials that make the structure stronger. Most of the matrix is made up

of high-molecular-weight monomer units, like urethane dimethacrylate (UDMA) and bisphenol A-glycidyl methacrylate (Bis-GMA).¹ Fillers are used to improve mechanical property like strength, decrease polymerization contraction and heat generation.² To enhance the bond between these components and to improve filler dispersion, a silane coupling agent is employed. When extrinsic power (light) is given, an initiator and an activator are typically added to commence and then govern the polymerization process.

current restorative materials. In this study, addition of CHEX, PLYS, and CaP addition will help in the reduction of polymerization shrinkage, and microleakage. Adding these fillers will enhance the composite antibacterial, and remineralizing properties, and making it last longer in the oral environment. This will in turn benefit the patient and healthcare system.

This study was planned to determine how the mass and volume of experimental composites altered following immersion in distilled water (DW) and a simulated bodily fluid (SBF).

METHODS

Samples were created in a variety of sizes in accordance with ISO standards and test specifications. Testing was done on 48 distinct formulations. Two groups were created out of them. Storage media (distilled water or simulated body fluid), adherent monomer (4-META or Hydroxy Ethyl Methacrylate [2-HEM]), Anti-bacterial (PLYS level 5 wt percent or 0.5 wt percent, CHEX level 5 wt percent or 0 wt percent) and remineralizing (CaP level (20, 10, or 0 wt percent) comprised of the variables examined for mass and volume changes.

Each sample was a 10 mm circumference and 1 mm wide disc. On a transparent block, metal rings were fixed on an acetate film and composite pastes were used to make samples. The filled rings were immediately covered with a second acetate sheet and topped with a second glass block. These were then cured by light for 40 s from both sides, A Demi Plus Light (output power 1,000 mW/cm²) was being used. After curing, the acetate film and metal rings were removed, and specimens' margins were smoothed.

Following that, the samples were kept at 37 degrees Celsius in 15 milliliters of preservative fluid (DW & SBF) for the following durations (2, 6, 24, 48 hrs. and 1, 2, 4, 6, 12 weeks). The discs were taken out, dried off, and weighed again at each interval. Finally, using equations (1) and (2), it was possible at each point in time, to calculate the percent volume and mass change.

$$\% \text{ Vol Change} = (100[V_t - V_0])/V_0 \quad (1)$$

$$\% \text{ Mass Change} = (100[M_t - M_0])/M_0 \quad (2)$$

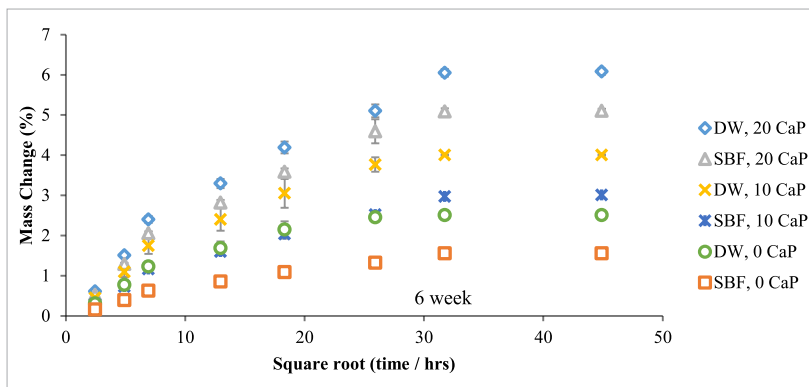


Figure 1: Plotting illustrative differences in mass versus the sum of squares time. In preceding instances, the quantities of Polylysine, CHEX, and 4-META are set by weight in the filler and monomer phases.

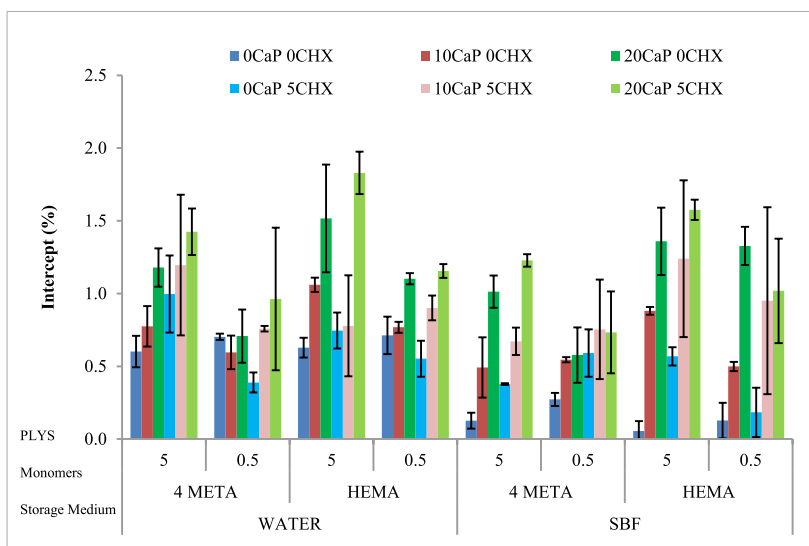


Figure 2: Initial changes in mass determined by intercept of all compositions containing 5 or 0.5 wt % PLYS, adherent amide (4-META or 2-HEM), and preservative media (Distilled water or SBF) with various concentrations of CaP (20, 10, or 0 wt %) and CHEX (5 or 0 wt percent).

Following immersion, V_t and M_t are the volume and mass respectively, whereas V_0 and M_0 are the starting volume and mass. The mean values and 95 % confidence interval values were taken, the gathered data was examined using three distinct variables and a factorial design with two intervals.

RESULTS

Variation in Mass of Experimental Composites

Figure 1 depicts illustrative differences in mass against the sum of the squares of time. Initially, there was a rapid rise in mass during the first 24 hours. Up until six weeks later, differences in mass was proportional to the sum of squares of

time. All formulations were presumed to have attained equilibrium after 12 weeks.

The CaP levels in all compositions (figures 2) increased consistently during the study, which lasted 3 months. With 20% CaP, the average difference in mass was between 4% and 7% while with 10% CaP, the average mass change was between 2.5% and 4.5% and with 0 wt % it was 1-2.5 percent after 12 weeks. The highest ultimate difference in mass observed for compositions held in Distilled Water (2.5-7 percent) resulted in greater percentage than for those stored in Simulated Body Fluid (1-5.5 percent). Like the PLYS 0.5 wt percent, ultimate difference in mass

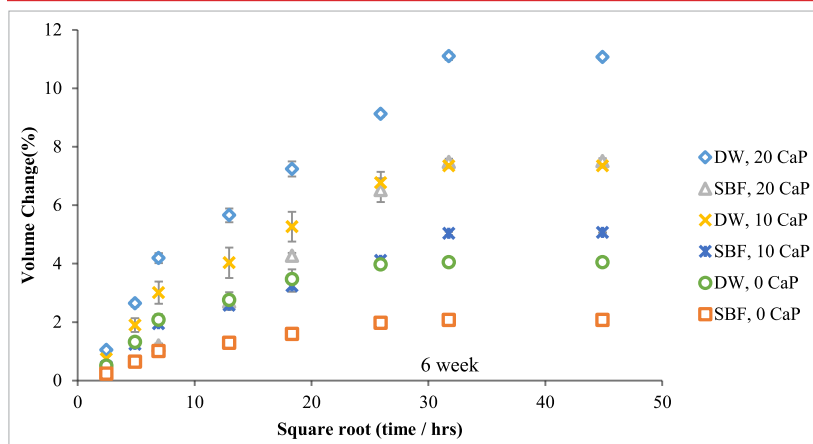


Figure 3: Illustrative difference in volume vs the sum of squares of time. In the preceding figure, the quantities of Polyllysine, CHEX, and 4-META are set by weight in the filler and monomer phases.

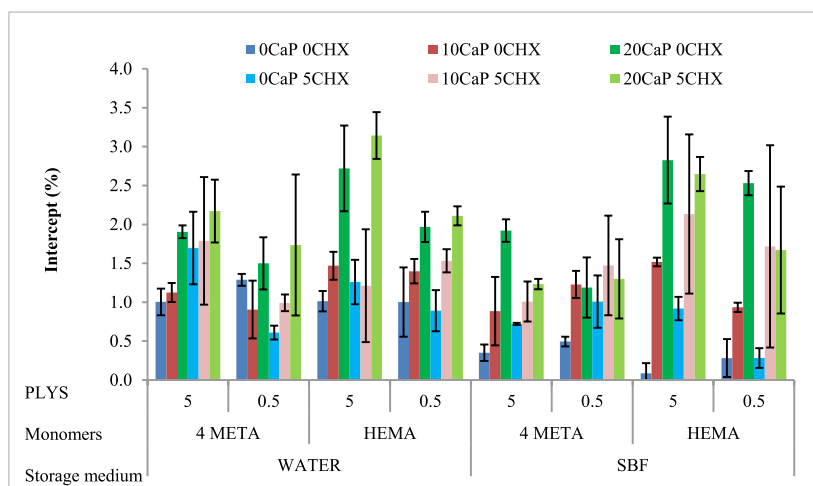


Figure 4: Initial differences in Volume determined by intercept for all compositions containing 5 or 0.5 wt % PLYS, adherend amide (4-META or 2-HEM), and storage media (Distilled water or SBF) with various concentrations of CaP (20, 10, or 0 wt %) and CHEX (5 or 0 wt percent).

with the PLYS 5 wt percent was between 2 and 7 percent (1-5 percent). With 4-META, the ultimate rise was lower than with 2-HEM. CHEX had little influence on the ultimate mass change.

Variation in Volume of Experimental Composites

Figure 3 depicts orchestrating typical differences in volume against time squared.²⁰ weight percent CaP in distilled water resulted in a 12 percent volume increase, but it was 8 percent in simulated body fluid. Similarly, the ultimate differences in volume after 3 months for 10 wt percent CaP in distilled water and simulated body fluid

was 7.5% and 5%, respectively. With 0 wt percent CaP in distilled water, the ultimate difference in volume was 4 percent, but it was 2 percent in SBF. Maximum volume change may be seen in the initial 2 days, then over the next six weeks, the volume will gradually increase. All compositions were believed to have attained equilibrium after 12 weeks.

For each composition, starting difference in volume may be determined by calculating the intercept of data for the percentage difference in volume vs the sum of squares of time. Figure 4 demonstrates CaP level has a significant influence on prevention, with average preventions of 3, 1.5, and 1

percent for CaP levels of 20, 10, and 0 wt percent, correspondingly. SBF is the additional factor having an impact. Initial difference in volume was smaller (1.5 percent) with SBF in contrast to DW (1.5 percent) (2 percent).

DISCUSSION

Immersion in an aqueous solution causes the majority of composites to absorb water to some degree.⁸ In traditional composites, the monomer phase is primarily responsible for this water absorption.⁹ Composite materials may include unreacted compounds, which might have a cytotoxic effect and encourage the growth of bacteria leading to tooth decay.^{10,11} Furthermore, excessive water absorption can result in plasticization of the polymer matrix, which degrades its mechanical characteristics.^{8,12} The trial compositions proposed for this research are intended largely for dental applications. The durability of these composites in distilled water and simulated body fluid will assist in the development of therapeutically balanced formulations.

Initial fluctuations in mass and volume may be explained by the existence of water-soluble CaP.¹³ Upon dissolving in water, mono calcium phosphate mono hydrate (MCPM) has the potential to enhance the internal osmotic pressure.¹⁴ If water replaces the MCPM, the composite's mass will decrease but its volume will stay unchanged. Similarly, if the absorbed water lengthens the molecular chains, the composite's mass and volume would increase. In addition, the inclusion of such fillers would lower the wetness of the subunits and increase the amount of air bubbles in the final combination.¹⁴ The pores of these composites will rapidly fill with water upon immersion. This would result in increasing the mass of the composite material but not its volume. These were the few alternatives that existed when CaP was added to the composites. The inclusion of CaP, specifically MCPM, designed to provide sufficient calcium and phosphorus to promote the self-healing of deteriorating tooth structure.

2-HEM has hydrophilic hydroxyl groups that bond to water molecules, hence increasing water sorption¹⁵ Poly2-HEM

is plasticized by this water sorption, which lowers its T_g and increases crosslinking density. Moreover, the density of crosslinks in the polymer matrix may be decreased by the addition of 2-HEM or 4-META. Additionally, this will cause the polymer matrix to inflate and promote water diffusion. PLYS inclusion in the research promoted water sorption into the dental composites. PLYS is highly soluble in both Distilled Water and Simulated Body Fluid.¹⁶ As the amount of PLYS in the filler phase increased, the water sorption increased. PLYS includes $-NH_3+Cl^-$ groups that disintegrate in water to create cationic polymers that attract additional water. PLYS and CalP both improve water sorption in a synergistic manner. Comparatively, raising the CHEX content in the filler phase from 0% to 5% had no outstanding influence on either the difference in mass or volume. This may be a result of its lower water solubility compared to PLYS. Tri calcium phosphate (TCP's) role in this process is to limit the breakdown of MCPM to offset its high solubility.¹⁴ The previous investigation indicated that the maximum water absorption is related to the filler phase's CalP concentration.

Water sorption can sometimes be advantageous. The absorbed water may cause material enlargement, which, for some materials, may have the beneficial effect of decreasing micro-gaps between composite-tooth repair contacts caused by polymerization shrinkage.¹⁷ Consequently extending the durability of the repair. This volumetric expansion may thus play an important role in avoiding bacterial permeability and repeated caries.

CONCLUSION

Our study concluded that the amount of reactive calcium phosphate (MCPM, TCP) and the kind of preservative media influenced mass and volume growth the most in all trial compositions, followed by further factors. The ultimate difference in volume was almost doubled the difference in mass for the majority of compositions. Likewise, the rise in distilled water was about half percent greater than the increase in simulated body fluid. Excessive volumetric growth is not desired. As this

would put unwarranted strains on the tooth organization and may result in cracks or fracture of the weakening cusps, it is not recommended. Regarding the development of a balanced composite with regulated volumetric expansion, further study is required.

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AUTHOR'S CONTRIBUTION

Following authors have made substantial contributions to the manuscript as under:

SL: Concept and study design, acquisition, analysis and interpretation of data, drafting the manuscript, critical review, approval of the final version to be published.

HJ: Analysis and interpretation of data, drafting the manuscript, approval of the final version to be published.

Authors agree to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

CONFLICT OF INTEREST

Authors declared no conflict of interest

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DATA SHARING STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request



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