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Long-term hygroscopic dimensional changes of core buildup materials in deionized water and artificial saliva

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The dimensional stability of core buildup materials plays an important role in clinical application. In this study, hygroscopic dimensional changes of four commercial core buildup materials were investigated in deionized water and artificial saliva for up to 150 days. Specimens were made within a customized cylindrical mold. The initial mass and the apparent mass in liquids were measured. All the tested materials showed hygroscopic expansion after a 150-days immersion time. Hygroscopic expansion of these four materials can partly compensate for the polymerization shrinkage. SDR showed the lowest hygroscopic expansion of the four tested materials when immersed in deionized water and artificial saliva. PC showed the highest hygroscopic expansion in deionized water, while LC showed the highest hygroscopic expansion in artificial saliva. In the case of different immersion solvents, osmotic pressure should be considered. For hygroscopic dimensional changes, the hydrophilicity of monomers and changes of intermolecular forces may be crucial factors.

Keywords: Hygroscopic dimensional change, Expansion, Core buildup materials, Solvents, Monomers

INTRODUCTION

Commercial core buildup materials have been widely used in defected teeth. For teeth with sufficient dentinal ferrule, core buildup materials are used more frequently[1,2]. Core buildup materials have been used to form the core because of their aesthetics and similar elastic modulus to dentine. A high success rate has been reported by combining fiber reinforced post and core buildup materials with high filler contents[3].

Dimensional stability plays an important role in clinical application. It may be affected by polymerization shrinkage and hygroscopic dimensional expansion. Polymerization shrinkage behavior is related to monomers, fillers, specimen dimension, temperature and curing lights, and it may create marginal gaps and influence the longevity of the materials[4-13]. Replacing diluent, changing types or sizes of fillers, as well as surface silanization are all possible methods to reduce polymerization shrinkage. However, these methods require further investigations[7,8]. Hygroscopic dimensional expansion is also related to dimensional stability. It has been suggested that hygroscopic expansion might partly compensate the volumetric shrinkage, while it still leaves contraction stress, and contraction stress would cause tooth deflection, fracture and interfacial debonding[4,14-16]. Meanwhile, long-term expansion could be a challenge for the survival of the restorations and overcompensation is not desirable[15,17,18]. When it comes to clinical application of newly developed materials, long-term follow-up investigations can not be neglected[4,19].

For core buildup materials, Bis-GMA (bisphenol A glycol dimethacrylate), TEGDMA (triethylene glycol dimethacrylate), UDMA (urethane dimethacrylate) and Bis-EMA (bisphenol A ethoxylated dimethacrylate) are frequently used matrices. TEGDMA showed the highest water absorption, followed by UDMA, Bis-EMA and Bis-GMA[20]. SDR is a new class of bulk-fill composite resins. It contains polymerization modulator to reduce polymerization shrinkage[21].

Commercial composite resins are inevitably in contact with wet conditions when applied intra-orally[20,22]. In *in vitro* studies, various liquids were used and resulted in different hygroscopic dimensional changes[23]. Deionized water is reported to be an acceptable storage medium in certain circumstances[6]. Nevertheless, hygroscopic dimensional changes in deionized water and artificial saliva are varied in other studies[23].

The aim of this study was to evaluate the hygroscopic expansion of four commercial core buildup materials immersed in deionized water and artificial saliva. The null hypothesis was: (i) there would be no differences in the hygroscopic expansion for the same core buildup materials both in deionized water and artificial saliva and (ii) there would be no differences in the hygroscopic expansion for different core buildup materials.

MATERIALS AND METHODS

Preparation of specimens

The four core buildup materials shown in Table 1 were tested in a laboratory environment: 23(±1)°C and 50(±2)% in humidity. The polymerization shrinkage of the tested materials was shown in Table 2.

Materials were placed into a customized cylindrical

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Color figures can be viewed in the online issue, which is available at J-STAGE.

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Table 1 Tested core buildup materials

Code	Product	Manufacturer	Lot No.	Resin systems	Fillers	Filler loadings (wt%)
LC	LuxaCore Z-Dual	DMG, Germany	748093	Dimethacrylate resin, Bis-GMA	barium glass, silicon dioxide	64
NF	NanoFil	Beijing AT&M biological medical materials limited liability company, China	171117	Bis-GMA, UDMA, Bis-EMA, TEGDMA	strontium glass, fluoride	70
PC	ParaCore	Coltène/Whaledent, Switzerland	H52424V	UDMA, TMPTMA, Bis-GMA, TEGDMA	amorphous silica, strontium glass, fluoride	68
SDR	Smart Dentin Replacement	DENTSPLY Caulk, USA	1701000441	SDR-UDMA, UDMA, TEGDMA	barium and strontium, alumino-fluoro-silicate glass	68

Bis-GMA, bisphenol A glycol dimethacrylate; Bis-EMA, bisphenol A ethoxylated dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; TMPTMA, trimethylolpropane trimethacrylate

Table 2 Polymerization shrinkage of core buildup materials

Code	polymerization shrinkage (%)
SDR	3.50
LC	4.60
NF	2.4–2.6
PC	3.50

The data are provided by manufacturers.

mold (15.0 mm diameter×2.0 mm thickness) and then, 40 disk-shaped specimens (n=10) were obtained according to ISO FDIS 4049: 2009 and the manufacturer's instructions. A total of 10 specimens were randomly divided into two groups for each material: five specimens for deionized water and the other five for artificial saliva. The mold with uncured material was sandwiched between two pieces of transparent polyester film with two glass slides on each side. During this process, minimizing entrapped air was a matter of great importance. Five overlapping sections on each side of the specimen were irradiated by a curing light (Ultradent, South Jordan, UT, USA) with a 480 nm output wave length and a 3,200 mW/cm^2 output irradiance. To minimize oxygen in the air, irradiation was delivered through the polyester film without glass sides and after the first irradiation. The stability of the curing light was checked periodically by a radiometer. The specimens were removed from the mold carefully and any with visual voids were discarded. To complete the preparation of the specimens and to obtain a uniform thickness, 240-, 500- and 1000-grit silicon carbide abrasive paper (Xinbei District, Changzhou, China) was used to remove excess flash.

All the specimens were stored separately in glass vials with anhydrous self-indicating silica gel in a lightproof desiccator at 37(±1)°C for 22 h, then transferred to a laboratory environment at 23(±1)°C for 2 h. This cycle was repeated until the mass change of each specimen was less than 0.1 mg in any 24 h period, and the initial constant mass m_1 in the balance (Shanghai, China) was obtained. Specimens were then placed into the holder immersed in water to get the initial apparent mass m_0. Then a total of 20 specimens were immersed into deionized water and the other 20 specimens were immersed into artificial saliva. Artificial saliva was synthesized according to the previous research: NaCl 0.4 g/L, KCl 0.4 g/L, $CaCl_2 \cdot H_2O$ 0.795 g/L, urea 1 g/L, $Na_2S \cdot 9H_2O$ 0.0105 g/L, $NaH_2PO_4 \cdot 2H_2O$ 0.78 g/L, deionized water 1,000 mL, and NaOH was used to adjust pH value to 6.8[24]. Specimens were immersed in deionized water and artificial saliva separately for up to 150 days, and their mass were measured as a function of time.

Hygroscopic dimensional changes

Archimedes' principle (buoyancy of a material in fluid) was frequently used to calculate the hygroscopic dimensional changes of the immersed resin composites

in previous studies (Fig. 1)[17,22]. The specimens were removed with tweezers and dried on the filter papers until no excess liquid was visible. The mass in the air was measured and noted as $m_{2(0)}$. Then the specimens were placed in the holder immersed in water with caution to avoid air bubbles. The apparent mass in the balance was noted as $m_{2(t)}$. ΔM is the mass upthrust. Thus, the volume of the core buildup materials was obtained as follows:

$$V=\frac{\Delta M}{\rho}=\frac{m_{2(0)}-m_{2(t)}}{\rho}$$

where ρ represents the density of the immersion fluid. The hygroscopic dimensional change was calculated as follows:

$$\Delta V\%=\frac{V_t-V_0}{V_0}\times 100$$

$$V_0=\frac{m_1-m_{1(0)}}{\rho}$$

where V_t is the volume at specific time, V_0 is the initial volume of the specimens. ΔV is the volumetric changes.

According to Archimedes' principle, the hygroscopic dimensional change can also be expressed as follows:

$$\Delta V\%=\frac{m_{2(0)}-m_{2(t)}}{m_1-m_{(0)}}-1$$

Statistical analysis

The data were analyzed using SPSS Version 22. Repeated measures ANOVA was used. Normality was tested for each group ($p<0.05$). For the same types of materials in different immersion solvents, a t test with two independent samples was used. For different kinds of materials in the same immersion solvents, one-way analysis of variance (ANOVA) was used ($p<0.05$), followed by Tukey's *post hoc* test ($p<0.05$).

RESULTS

Repeated measures ANOVA showed statistically significant differences in hygroscopic dimensional changes during the 150 days immersion time.

The maximum hygroscopic dimensional changes in different solvents are presented in Table 3. All the test materials finally demonstrated hygroscopic expansion

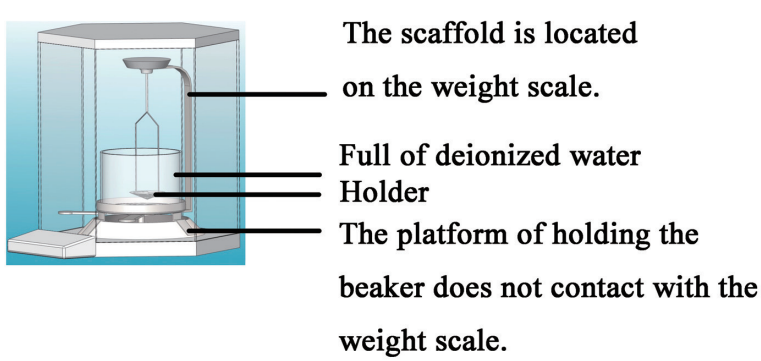

Fig. 1 Balance for testing the volumetric dimensional change.
The holder was immersed in deionized water and used to accommodate specimens. The balance, including the beaker with water, should be set to zero every time before measuring. The apparent mass was noted as the specimens were placed in the holder and the balance was stabilized. The water level should be such that the holder and the specimens are submerged.

Table 3 Percentage mass change and volumetric dimensional change (standard deviation) of test materials after 150 days immersion

	DI	AS
SDR	[a] 0.256 (0.195)	[d] 0.417 (0.080)
NF	[b] 0.875 (0.170)	[d,e] 0.882 (0.462)
LC	[b,c] 1.328 (0.169)	[d,e] 1.031 (0.283)
PC	[c] 1.363 (0.418)*	[e] 0.910 (0.127)*

For the data in row, those labelled with * were significantly different ($p<0.05$).
For the data in column, those labelled with the same alphabet were not significantly different ($p>0.05$).

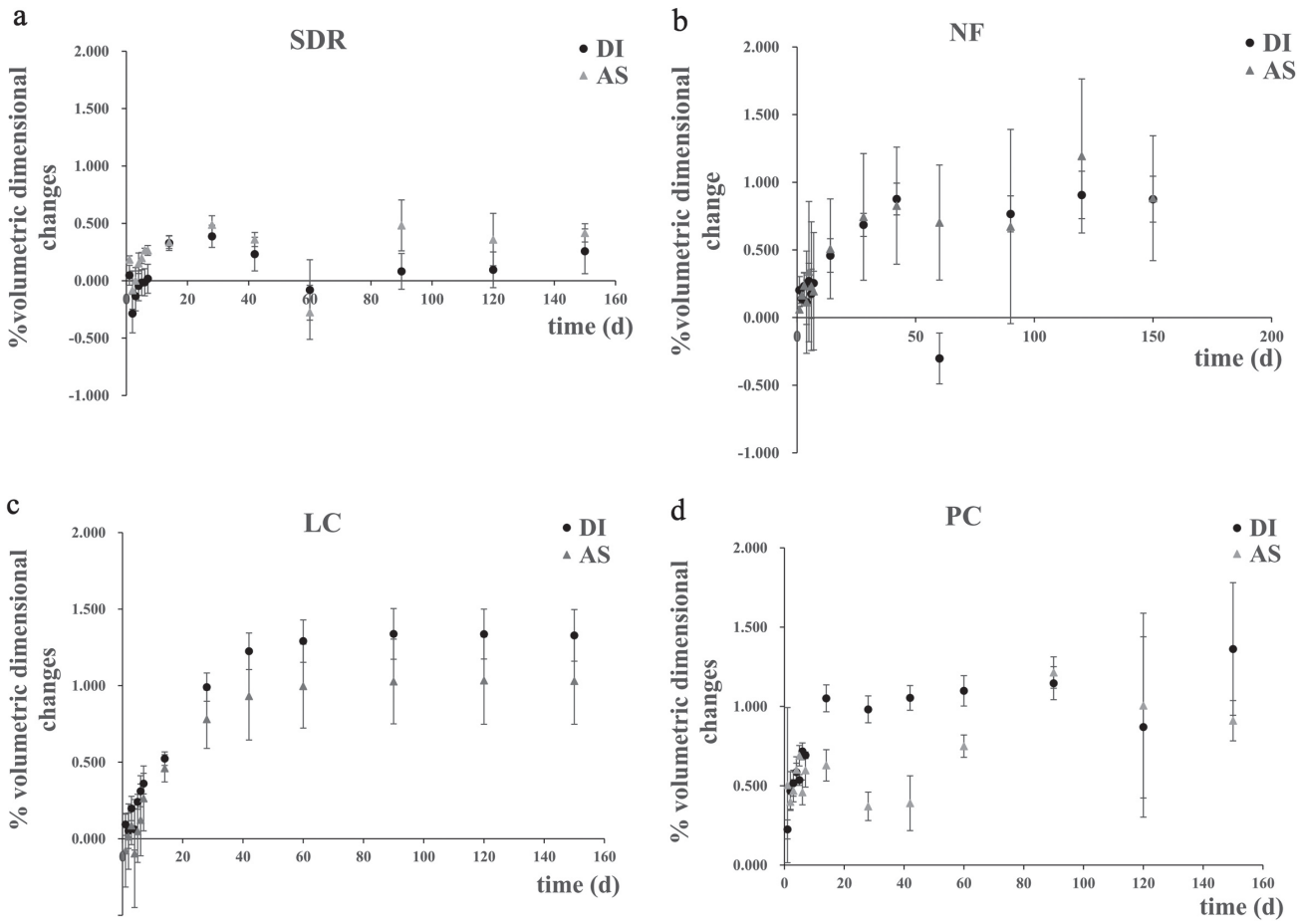

Fig. 2 Volumetric dimensional change of four tested materials in different immersion fluids (a–d) (DI: in deionized water; AS: in artificial saliva).
Measurements were taken for a total of 150 days. The two immersion fluids showed similar variation trends.

resulting from immersion in deionized water or artificial saliva (Figs. 2a–d).

For different kinds of materials in the same immersion solvent, one-way analysis of variance was performed to compare their hygroscopic dimensional changes. SDR showed the lowest volumetric expansion in two immersion solvents. In deionized water, PC showed the highest volumetric expansion, while in artificial saliva, LC showed the highest volumetric expansion (Table 3).

For the same materials in varied immersion solvents, only PC showed a significant difference in two solvents (p=0.049, Table 3).

DISCUSSION

The volumetric dimensional changes of composite resin are complex and may be of concern for clinical application. It may be affected by thermal expansion, polymerization shrinkage and immersion solvents[12,23,25,26].

In this study, the free expansion was evaluated, however, it may have been higher than that in clinical practice, as the expansion of the materials would be constrained by the cavity walls in oral conditions[26]. The same core buildup materials immersed in two different solvents did not show statistical differences in volumetric expansion except for PC, thus, hypothesis (i) was rejected.

Immersion fluids play essential roles in the hygroscopic dimensional changes of core buildup materials. They can either extract the unreacted monomers to cause shrinkage and mass loss, or conversely lead to expansion and mass increase[20,25,27,28]. Core buildup materials in different solvents exhibit various hygroscopic dimensional change[22,25,27]. There are two possible causes: solubility parameter and osmotic pressure[23,27]. Maximum liquid uptake occurs when the solubility parameters of the polymer and liquid are equal[27]. Inevitably, there is flow of ions between materials and the immersion solution under osmotic pressure. In this study, PC showed higher hygroscopic dimensional change in deionized water than that shown in artificial saliva. This may be due to the higher water absorption demonstrated in deionized water. Water flow

would increase in materials when immersed in deionized water because of the osmotic pressure gradient[23]. In artificial saliva, the liquid contains many inorganic ions, thus, the osmotic pressure may be closer to that of PC. This could also be related to the inner structure of PC, but it remains to be further verified.

Hygroscopic dimensional change also vary for different sizes, measuring conditions, chemical composition and curing conditions[4,19,23,26,28,29]. In this study, different kinds of core buildup materials behaved differently when immersed in the same fluid, thus, hypothesis (ii) was rejected. Core buildup materials vary in terms of their compositions. The relationship between chemical compositions and hygroscopic dimensional changes has been studied in detail previously. The higher volume increase may have been due to the higher percentage of water uptake[25]. NF demonstrated a smaller hygroscopic dimensional change than the other materials because of Bis-EMA. It is believed that the chemical and physical structure would affect the hydrophilicity of composite resins[22,23]. Bis-EMA processes ether groups and its structure resembles Bis-GMA. It is thought to be more hydrophilic than Bis-GMA due to the lack of -OH groups. Bis-EMA is less flexible than TEGDMA and permits less swelling of polymer chains[20,30,31]. The newly manufactured product, SDR, showed the lowest water sorption among the four tested materials. The main composition of SDR is SDR-UDMA. This is a high molecular matrix and it is likely to be more hydrophobic, as it was shown in previous research[21]. In the same immersion solvents, there are no significant differences between PC and LC. However, they do differ for different resin matrices and filler loadings. Nevertheless, they both contain Bis-GMA, a hydrophilic monomer which may form hydrogen bonds[20]. This may indicate that hydrophilic monomers tend to be more crucial for water absorption.

Moreover, filler properties, such as filler loadings, filler types, filler sizes, as well as initiating systems would all contribute to hygroscopic dimensional change[4,25,28]. The highest filler loaded material NF showed a smaller hygroscopic dimensional change than that of LC and PC. This result is in line with a previous study that found that water sorption had a negative correlation with filler loadings[21]. Simultaneously, hygroscopic dimensional changes are highly correlated with water sorption, thus, higher filler loadings indicate a lower water uptake.

Long-term hygroscopic dimensional changes are complicated. Nevertheless, it is necessary to predict the change tendency for clinical practice[17]. In this research, it can be divided into two periods as a functions of immersion time. In the initial period (1–42 days), water tended to fill in the free volume of the materials, especially near the cross-linking regions[19,25,27]. The heterogeneity of the polymer network and solubility promotes the existence of micro-voids. In the meantime, it shows the offensive zone and accommodation for water molecule[27,32]. Then, specimens absorb water without undergoing dimensional change[19,25]. After the free volume has been occupied, specimens show expansion rather than shrinkage, even though the hydrolysis of monomers and filler debonding still exist.

Little hygroscopic dimensional changes were observed during the following period (42–150 days). For SDR, NF and PC, it decreased in volume between 42 and 150 days. When the volume loss by solubility exceed the volume gains by water sorption, the decrease in volume is not surprising[22]. The leached elements are strongly related to the formation of microcracks and decrease the strength of matrix-filler interfaces. It has been reported that leaching of composite resins and debonding of the matrix-filler interface is likely to occur in long-term immersion[33]. Residual monomers and ions move outwardly and peeling stress would occur in polymer layers[34,35]. They would cause loss in volume. When immersed in water, NF showed the decrease in volume at 60 days, which was lower than the initial volume. However the mass of NF at 60 days was a little higher than that at the earlier stage (Wei, unpublished). Thus it demonstrated the increase of the density. It may owe to the intake of water. Water breaks the hydrogen bonds and restrains the polymer chain motion, then the density of the materials increases[36]. Moreover, during this period, the final volume still exceeded the initial volume. This indicated that there was a continuous water uptake. Water uptake was related to the microvoids. Microvoids could be generated followed by the disconnection of filler phase and resin matrices[25]. Unbonded water then filled the microvoids and resulted in the expansion of the materials[26,31]. The loss of interfacial bonding may occur in this stage and, subsequently, caused a higher effective expansion[18]. The process would cost time as leached elements need time to release to the immersing liquid[31] This stage continues when degradation and stretching of the molecular bonds occur[23]. The process ends up by reaching the surface of materials, which implies the equilibrium of the absorption process has been reached[31]. As for the results in this research, the volumetric dimensional change for LC and NF tended to reach equilibrium while SDR and PC would require a longer immersion period to reach to the equilibrium stage.

The test materials showed the same trend in both solvents. This indicates that for water sorption, chemical compositions rather than immersion solvents may play a crucial role.

In this research, hygroscopic dimensional expansion was expected but it was still lower than the polymerization shrinkage of certain materials (Tables 2, 3). It is beneficial for composite resins to compensate for the polymerization shrinkage *via* volumetric expansion[15]. Thus it can reduce cuspal deformation while improving the bonding quality[15]. Nevertheless, hygroscopic dimensional changes would be more complex in clinical performance than those observed in the test environment. No cavity configuration restricts its expansion outside, and *in vitro* measurements of the dimensional changes could be achieved[15,16,28]. Thus the results of *in vitro* research simply indicate the change tendency, which is beneficial as a reference but requires

further clinical verification. When the coefficient of expansion exceeds the polymerization shrinkage, cracks and fractures of tooth structure may occur, and may decrease the longevity of the materials[23].

Overall, the hypothesis (i) and (ii) could be rejected. Further studies should take the variebles of immersion liquids, sizes and temperature or other conditions into consideration to mimic the clinical situation[17,37,38]. Hygroscopic dimensional behavior is not the decisive factor for the selection of suitable materials in clinical use. However, it can provide valuable suggestions and information for material modification, research and development.

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CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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