Water Sorption and Solubility of Resin Composite Core Build-up Materials

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Abstract

The aim of the current study was to investigate the water sorption and solubility properties of various core build-up resin composites after 30 days of immersion. Three resin-based materials were tested including MultiCore®Flow, Filtek™Bulk Fill, and Filtek™Z350XT. All 30 specimens (n=10) were prepared according to ISO 4049:2009. The specimens were stored in the desiccator of 37±2 ºC for 22h and at 23±1 ºC for 2h and then weighed with a precision accuracy of ±0.1 mg to obtain the initial constant mass. After a total immersion time of 30 days in distilled water, the specimens were weighed and dried in the desiccator to obtain other constant mass. Water sorption and solubility values were calculated and any statistical differences were determined by using one-way ANOVA at a significant level of 0.05. The result found that MultiCore®Flow and Filtek™Bulk Fill showed lower water sorption value than Filtek™Z350XT significantly ($p<0.001$) with the value of 15.63±0.88 $\mu g/mm^3$, 14.46±1.72 $\mu g/mm^3$, and 18.88±1.74 $\mu g/mm^3$ respectively. Additionally, all tested materials demonstrated significantly different solubility behavior ($p<0.001$). MultiCore®Flow had the lowest solubility (2.34± 0.59$\mu g/mm^3$), followed by Filtek™Bulk Fill (4.43±0.96 $\mu g/mm^3$), and Filtek™Z350XT (6.34±1.17 $\mu g/mm^3$). In conclusion, resin-based core build-up materials tested in this study exhibited significantly different water sorption and solubility behavior. MultiCore®Flow had the lowest sorption and solubility, while Filtek™ Z350XT had the highest. According to ISO 4049 standards, all tested materials showed acceptable water sorption and solubility behavior.

Keywords: Water sorption/ Solubility/ Resin composites/ Core build-up materials

Introduction

Properties of foundation materials can affect the clinical outcome of the fixed prosthetic restorations, especially in cases with extensive loss of coronal tooth structure. Several types of resin-based materials are widely used as a core build-up material due to its favorable properties particularly tooth-like color, adequate mechanical properties, and curing time period that facilitates immediate preparation. Those types include dual-cured core build-up material, which usually comes in form auto-mixing gun type, and light-cured resin composite, which is normally used as restorative material by incremental or bulk-filled technique. However, water sorption and solubility behaviors of the resin composites have shown to have unfavorable effect in physical and mechanical properties including modulus of elasticity, surface hardness, wear resistance, and color stability. These properties are significantly concerned for the longevity of the

core build-up materials.[1-6] Since dental resin-based restorative materials are continuously exposed to the wet environment, which is the oral cavity, thus the properties of water sorption and solubility play an important role in their long term success.[4,5] Resin-based core build-up materials, which are usually filled in the extensively damaged tooth structure in conjunction with prefabricated fiber posts, are often left exposed in the oral cavity prior to the tooth preparation procedure in the next visit, which could be delayed for several weeks or months. This type of clinical situation might result in long term exposure of resin core build-up material to the moist environment inevitably.

Previous study in 2014 investigated water sorption behavior of the four different resin-based materials, including Grandio Core, Core.X Flow, Bright Flow core and Speedee. The result from this study demonstrated significantly

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different water sorption and solubility among all four tested materials.[7] Limited studies were interested in investigating water sorption and solubility of the resin-based materials commonly used as core build-up materials among most dental professions.

MultiCore® Flow, dual-cured resin composite material that comes in form of auto mix syringe which facilitates the direct injection into the prepared cavity; Filtek™ Bulk Fill, the light-activated restorative composite that could be bulk placed into the tooth cavity; and Filtek™Z350XT, the light-cured nanocomposite which has to be incrementally placed into the cavity, are those three resin composite materials most commonly used as core build-up materials in restoring severely damaged tooth. Previous study of short and long-term water sorption behavior of resin-based materials has shown that the maximum amount of water gain occurred during the first month of testing time period, therefore, one-month immersion time period would sufficiently represent accurate water sorption and solubility behavior of the materials.[7]

It would be beneficial for clinicians to have some more information on the resin composite core build-up materials being widely used in regular dental practice to assist in selection of the material with the lowest value of water sorption and solubility for long term success.

The purpose of this study was to investigate the water sorption and solubility properties of various resin composite core materials after 30 days of immersion time.

Materials and Methods

Three types of the tested materials are shown in Table 1. Ten specimens of each group (n=10) were prepared according to ISO 4049:2009. Mould for the preparation of a disc specimen with the dimensions of 15.0±0.1 millimeter (mm) in diameter and 1.0±0.1 mm in depth was used to prepare specimen discs. The materials were filled into the metal mould with a piece of transparent polyester film on the top and bottom side. Filtek™Z350X and Filtek™Bulk Fill (shade A3) were condensed into the mould using hand instrument while MultiCore®Flow was injected into the mould. After having displaced the excess material, the mould

was then covered with the glass slide while curing each side of the specimens with overlapping irradiation from the inner-center to the outer-peripheral part of the specimens using the LED light-curing unit (Elipar S10, 3M ESPE, St.Paul, MN, USA) with the light intensity greater than 1000 mW/cm.[2] All specimens were then finished using fine-grit sand paper. The thickness and diameter of each specimen disc were measured with micrometer (Series111, Mitutoyo, Kanagawa, Japan) and the volume of each specimen was calculated. The specimens were then stored in the desiccator of 37±2 ºC for 22 hours (h) and transferred to be stored in the second desiccator at 23±1 ºC for 2h. After that, the specimens were weighed to an accuracy of 0.1milligram (mg) (Precisa 262 SMA-FR, Precisa Instrument AG, Dietikon, Swizerland) and their mass were carefully recorded. The cycle was repeated until the mass loss of each specimen was not more than 0.1 mg in any 24 h, thus, the value was recorded as a constant mass, m_1. Consequently, the specimens were immersed in distilled water at 37±1 ºC for 30 days by having 3 mm space between each specimen to assure the complete immersion. After a total immersion time of 30 days, all specimens were removed from water, blotted away the surface water until they were free from visible moisture and waved in the air for 15 seconds. The specimens' mass was recorded at this point as m_2. Consequently, the specimens were reconditioned to a constant mass in the desiccators using the same cycle as described above until the mass loss of each specimen was not more than 0.1 mg in any 24 h. The constant mass m_3 was then carefully recorded. Each value at each procedure was an average of the three repeated value for each specimen. Water sorption and solubility values were calculated using the specific equation.

Water sorption (W_{sp}) of each specimen was calculated using the equation[8]:

$$W_{sp} = (m_2 - m_3) / V$$

Where

m_2 is the mass of the specimen (µg) after immersion in water

m_3 is the mass of the reconditioned specimen (µg)

V is the volume of the specimen (mm³)

Solubility (W_{sl}) of each specimen was calculated using the equation[8]:

$$W_{sl} = (m_1 - m_3) / V$$

Where

m_1 is the conditioned mass (µg) prior to immersion in water

m3   is the mass of the reconditioned specimen (µg)

V   is the volume of the specimen (mm^3)

Statistical analysis

One-way ANOVA and Tukey Method were used to determine statistical differences among the mean water sorption and solubility of each group at a level of significance of .05 using SPSS version 18.0 software (SPSS Inc., Chicago, IL, USA).

Table 1   Tested materials, their compositions, and manufacturer information

Material	Composition	Filler (wt%)	Lot number
MultiCore® Flow (Ivoclar Vivadent, Schaan, Liechtenstein)	Bis-GMA, UDMA, TEGDMA inorganic fillers (barium glass, Ba-Al-fluorosilicate glass, silicon dioxide, and ytterbium trifluoride)	54.65	T37741
Filtek™Bulk Fill (3M ESPE, St.Paul, MN, USA)	AUDMA, UDMA and 1, 12-dodecane-DMA non-agglomerated/non-aggregated 20 nm silica filler, a non-agglomerated/ non-aggregated 4 to 11 nm zirconia filler, an aggregated zirconia/silica cluster filler and a ytterbium trifluoride filler	76.5	N609985
Filtek™Z350XT (3M ESPE, St.Paul, MN, USA)	Bis-GMA, Bis-EMA, UDMA, small amounts of TEGDMA non-agglomerated/non-aggregated 20 nm silica filler, a non-agglomerated/ non-aggregated 4 to 11 nm zirconia filler, and loosely bound agglomerated zirconia/silica nanocluster	78.5	N679116

Bis-GMA (bisphenol-A-glycidyldimethacrylate), UDMA (urethane dimethacrylate), TEGDMA (triethylene glycol dimethacrylate), AUDMA (aromatic urethane dimethacrylate), Bis-EMA (bisphenol-A-diglycidylmethacrylate ethoxylated), DMA (dimethacrylate)

Results

Filtek™Z350XT showed significantly higher mean of water sorption value (18.88±1.74 µg/mm^3) compared to MultiCore®Flow and Filtek™ Bulk Fill after 30 days of immersion in water (p<0.001). Filtek™ Bulk Fill demonstrated the lowest mean of water sorption value (14.46±1.72 µg/mm^3) among other tested materials, however, the mean water sorption of Filtek™ Bulk Fill was not significantly different from that of MultiCore®Flow (15.63±0.88 µg/mm^3) as presented in Table 2.

The three tested materials were significantly different from each other regarding their solubility properties (p<0.001). MultiCore®Flow had the lowest solubility value (2.34±0.59µg/mm^3), while Filtek™ Z350XT showed the highest value (6.34±1.17µg/mm^3) among all tested groups. Solubility behavior of Filtek™Bulk Fill (4.43±0.96 µg/mm^3) was superior to Filtek™Z350XT, nevertheless, the value was still higher than that of MultiCore®Flow.

Table 2   Mean (SD) of water sorption and solubility of the tested materials

Materials	Water sorption[#] (µg/mm^3)	Solubility[*] (µg/mm^3)
MultiCore®Flow	15.63 (0.88)[A]	2.34 (0.59)[a]
Filtek™ Bulk Fill	14.46 (1.72)[A]	4.43 (0.96)[b]
Filtek™ Z350XT	18.88 (1.74)[B]	6.34 (1.17)[c]

[#]Groups with different upper case superscript were significantly different in water sorption (p<.001).

[*]Groups with different lower case superscript were significantly different in solubility (p<.001).

Discussion

The current study compared water sorption and solubility properties of three resin-based core build-up materials. The results revealed differences of those properties among all tested groups. Regarding water sorption behavior, MultiCore®Flow and Filtek™Bulk Fill showed significantly better performance than Filtek™Z350XT. Almost similar to its sorption behavior, MultiCore®Flow demonstrated the lowest solubility among all materials. According to ISO 4049 standards, the water sorption and solubility values must be lower than 40 $\mu g/mm^3$ and 7.5 $\mu g/mm^3$ respectively.[8] The water sorption and solubility values of all tested materials obtained in this study were considerably lower than the threshold values. High water sorption and solubility would adversely affect the physical and mechanical properties of the resin-based materials, including strength, modulus of elasticity, surface hardness, wear resistance, and color stability.[1,3,5,6,9]

Water sorption and solubility of resin-based materials can be affected by many factors, including material types, the efficiency of polymerization, matrix composition variations, filler size, and distribution[5,9] Monomer compositions in the matrix of the resin-based materials may influence their water sorption behavior. The more hydrophilic monomer presented in the material, the more amount of water gained was expected[4]. Most resin composite materials utilize bisphenol-A-glycidylmethacrylate (Bis-GMA) as the matrix resin. Since Bis-GMA is highly viscous, more fluid difunctional monomer such as triethylene glycol dimethacrylate (TEGDMA) needs to be added to dilute the viscosity of the material. The added diluent monomers coupled with Bis-GMA tend to adversely affect the properties of the matrix material including the increase of water sorption.[3] Previous studies revealed that the materials with Bis-GMA and TEDGMA tended to gain more water than the materials without them.[1,10] This may be the reason why Filtek™Z350XT, which contains Bis-GMA and TEDGMA, had higher water sorption value than Filtek™Bulk Fill in this study. However, MultiCore®Flow also contains Bis-GMA

and TEDGMA according to the manufacturer, its sorption value was observed to be lower than that of Filtek™Z350XT. A previous study suggested that the relative proportion of each monomer in the matrix should also be taken into consideration rather than the composition of the matrix alone.[5]

Fillers also play a role in water sorption of resin composites. Resin composite with small-size filler tends to have higher water sorption value because of higher volume fraction of polymer. Previous studies showed an inverse correlation between water sorption and the filler loading of the resin-based materials. The lowest water sorption was observed in the material with the highest filler loading percentage.[7,9,11] In contrast to previous studies, the result from this study showed that the lowest water sorption was found in the material with the lowest percentage of the filler, which is MultiCore®Flow. Moreover, Filtek™ Bulk Fill and Filtek™ Z350XT had almost comparable percentage by weight of filler, however, they showed different water sorption behavior significantly. Besides the monomers and filler components, the quality and stability of coupling agent also had an effect on water sorption of resin composites.[12]

All of the components in dental resin composites, including monomers and fillers, could be leached out from the material into water determining solubility properties of the material. However, monomers make up the large majority of the eluted components.[13] During polymerization process, monomers react to form cross-linked polymer chains. The enlarged polymer chains are found to be difficult to participate efficiently in the polymerization process, which consequently result in the presence of unreacted monomers. These unreacted monomers are the components that mostly found eluted from the resin composite. The percentage of reacted monomer after polymerization is known as the degree of conversion. Higher degree of conversion results in less unreacted monomers available to be leached out form the material and thereby lower solubility value. The degree of conversion depends on the monomer composition and does

not differ between chemically activated and light-activated materials containing the same monomer composition as long as adequate light curing is used.[14] Since the materials used in this study contained dissimilar monomer composition, the degree of conversion might be different. However, the degree of conversion of the tested materials was not determined in this study, so the difference could not be verified.

Filler size and type had some contributions on solubility of resin composites in that smaller molecules tend to have enhanced mobility and are expected to be eluted at a faster rate than larger and bulkier molecules.[12] However, there were limited studies generated on this topic.[3,13] Barium glass is more readily leached into water than silica particles resulting that resin composites with barium glass filler are more unstable than those with silica fillers.[9,15] Silica-containing resin composites could also have high solubility since water in contact with silica filler surfaces can break siloxane bonds and consequently induces debonding of the filler particles.[3] This study found the lowest solubility in barium glass-containing MultiCore®Flow, whereas higher solubility in silica-containing Filtek™ Bulk Fill and Filtek™ Z350XT. Solubility is affected by fillers to some extent, however its greatest influence comes mainly from monomers.

Additional investigation was done in this study by observing the tested specimens under light microscope at 100X magnification (CX31, Olympus, Tokyo, Japan). MultiCore®Flow specimens appeared to be more homogeneous than other tested materials, in which air-filled voids were to be noticed as shown in Figure 1. Voids in the material may increase the surface exposed to water and may lead to inhibitions zones with unpolymerized materials, thus, accelerate water sorption and solubility.[4,9,16] The ease of handling' s method of injectable MultiCore®Flow can be attributed to its ability to be homogeneously developed resulting in better water sorption and solubility behavior than other tested materials. Previous study in 1996 demonstrated that the application mode of resin composite influenced the presence of voids in the restoration. The number of voids in resin composite was found to be lower for the injection technique than that of the incremental condensation technique.[17]

The results of sorption and solubility are expected to be correlated in the same way since the unreacted components can only be leached out when water penetrates into the material.[7,10] This is in agreement with the result in this study that all the tested materials had the same ranking regarding their sorption and solubility behavior. MultiCore®Flow showed lower value of both water sorption and solubility, whereas Filtek™ Z350XT showed the highest in both aspects.

Although the result in this study did not entirely coincide with the previous studies, the mean sorption and solubility values of all tested materials were reported to be lower than standard value, implying that all tested materials in this study can be safely used as core build-up materials regarding their sorption and solubility properties. However, the technique of handling these materials in clinical practice should be of interest as having air voids or non-homogeneous layers in the materials may create higher chance of increasing water sorption and solubility besides the inherent property of the materials themselves.

This study aimed to compare water sorption and solubility of the commonly used core build-up resin composites to provide an information for dentists to assist in material selection regarding this aspect. To decide which materials are to be properly chosen does not depend only on the sorption and solubility behavior, however, other properties such as strength, wear resistance, microleakage, hygroscopic expansion and shrinkage or the ease of material's application should also be considered.

Figure 1 Representative images of MultiCore®Flow, Filtek™ Bulk Fill and Filtek™ Z350XT specimens under light microscope at 100X magnification from left to right respectively. Voids could be observed in all tested materials, however, fewer voids were noticed in MultiCore®Flow (left) compared with other materials.

Conclusion

Within the limitations of this *in-vitro* study, the conclusion could be drawn that after 30 days of immersion time in water, all tested materials had significantly different water sorption and solubility values. MultiCore®Flow and Filtek™Bulk Fill exhibited significantly lower water sorption value than Filtek™ Z350XT. Moreover, MultiCore®Flow showed the lowest solubility value, while Filtek™Z350XT showed the highest. All tested materials including MultiCore®Flow, Filtek™ Bulk Fill and Filtek™ Z350XT showed acceptable water sorption and solubility values according to ISO standards.

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การดูดน้ำและการละลายของวัสดุสร้างแกนฟันชนิดเรซินคอมโพสิต

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บทคัดย่อ

การศึกษานี้มีวัตถุประสงค์เพื่อเปรียบเทียบคุณสมบัติการดูดน้ำและการละลายของวัสดุสร้างแกนฟันชนิดเรซินคอมโพสิตได้แก่ มัลติคอร์ โฟล์ว ฟิลเทคบัลค์ฟิลล์ และ ฟิลเทคซี 350 เอ็กซ์ที ทำการศึกษาคุณสมบัติการดูดน้ำและการละลาย ด้วยการเตรียมชิ้นตัวอย่างกลุ่มละ 10 ชิ้นตามมาตรฐานไอเอสโอ 4049:2009 นำชิ้นตัวอย่างไปเก็บไว้ในโถดูดความชื้นที่อุณหภูมิ 37±2 องศาเซลเซียส เป็นเวลา 22 ชั่วโมง จากนั้น นำไปเก็บไว้ในโถดูดความชื้นที่อุณหภูมิ 23±1 องศาเซลเซียส เป็นเวลา 2 ชั่วโมง แล้วนำชิ้นตัวอย่างมาวัดมวล โดยใช้เครื่องชั่งวิเคราะห์ที่ความ แม่นยำ 0.1 มิลลิกรัม จนได้ค่ามวลคงที่ จากนั้นนำกลุ่มตัวอย่างไปแช่น้ำกลั่นที่อุณหภูมิ 37 องศาเซลเซียสเป็นเวลา 30 วัน จึงนำกลับมาวัดมวล อีกครั้ง แล้วนำกลับไปไว้ในโถดูดความชื้นตามกระบวนการข้างต้น จนวัดได้ค่ามวลคงที่ ที่มีการเปลี่ยนแปลงน้อยกว่า 0.1 มิลลิกรัม นำค่าที่ได้ จากการทดลองมาคำนวณหาค่าการดูดน้ำและการละลาย แล้วนำไปวิเคราะห์ข้อมูลโดยใช้สถิติวิเคราะห์ที่ระดับนัยสำคัญ 0.05 ผลการศึกษา พบว่ามัลติคอร์ โฟล์วและฟิลเทคบัลค์ฟิลล์มีค่าการดูดน้ำที่ต่ำกว่า ฟิลเทคซี 350 เอ็กซ์ทีอย่างมีนัยสำคัญทางสถิติ ($p<0.001$) ค่าที่ได้คือ 15.63±0.88, 14.46±1.72 และ 18.88±1.74 ไมโครกรัมต่อลูกบาศก์มิลลิมตรตามลำดับ และวัสดุทั้งสามชนิดมีค่าการละลายที่แตกต่างกันอย่างมี นัยสำคัญทางสถิติ ($p<0.001$) โดยมัลติคอร์ โฟล์วมีค่าการละลายต่ำที่สุด (2.34±0.59 ไมโครกรัมต่อลูกบาศก์มิลลิเมตร) ขณะที่ฟิลเทคซี 350 เอ็กซ์ที มีค่าการละลายสูงที่สุด (6.34±1.17 ไมโครกรัมต่อลูกบาศก์มิลลิเมตร) ส่วนฟิลเทคบัลค์ฟิลล์มีค่าการละลายที่สูงกว่ามัลติคอร์ โฟล์ว แต่ต่ำกว่า ฟิลเทคซี 350 เอ็กซ์ที (4.43±0.96 ไมโครกรัมต่อลูกบาศก์มิลลิเมตร) กล่าวโดยสรุปคือ วัสดุสร้างแกนฟันชนิดเรซินคอมโพสิตทั้ง 3 ชนิดนี้ มีค่า การดูดน้ำและการละลายแตกต่างกันเมื่อนำไปแช่น้ำเป็นเวลาหนึ่งเดือน แต่อย่างไรก็ตามค่าการดูดน้ำและการละลายของวัสดุทั้ง 3 ชนิดยังอยู่ใน เกณฑ์มาตรฐานที่กำหนด

คำไขรหัส: *การดูดน้ำ/ การละลาย/ เรซินคอมโพสิต/ วัสดุสร้างแกนฟัน*

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