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Evaluation Methods of Depth of Cure and Polymerization Shrinkage for Dental Resin Composite Restorative Materials

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Abstract: Resin composites are widely used for various restorative processes. In 1960's it was used in dentistry for esthetic restoration of anterior teeth. But, in 1970's composite was used as posterior dental restorations and UV cured composites were introduced. Limited light penetration led to difficulty in polymerization of deep cavities. This was solved by using incremental technique. The incremental technique has drawbacks as time consumption and the risk of contamination. Improvements led to the development of bulk-fill composites to reduce the disadvantages of the incremental technique. Bulk-fill composites speed up restorative procedures because they can be applied in layers up to 6 mm in a single application.

Keywords: Depth of Cure; Polymerization Shrinkage; Resin Composite

1. Introduction

Resin composites are widely used for various restorative processes. In 1960's it was used in dentistry for esthetic restoration of anterior teeth. But, in 1970's composite was used as posterior dental restorations and UV cured composites were introduced.

Limited light penetration led to difficulty in polymerization of deep cavities. This was solved by using incremental technique. The incremental technique has drawbacks as time consumption and the risk of contamination. Improvements led to the development of bulk-fill composites to reduce the disadvantages of the incremental technique. Bulk-fill composites speed up restorative procedures because they can be applied in layers up to 6 mm in a single application. Manufacturers claim that these materials show low polymerization shrinkage and an increased depth of curing [(1)].

Evaluation methods of depth of cure

Depth of cure

The thickness of a light cured resin that can be converted from a monomer to a polymer when exposed to a light source under a specific set of conditions. When light is transmitted through the composite resin, there is a gradual decrease in degree of conversion as the distance increases from the irradiated surface which leads to elution of monomer causing failure of the restoration [(2)].

Degree of conversion

The percentage of carbon-carbon double bonds (- C = C -) converted to single bonds (- C - C -) to form a polymeric resin [(2)].

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- **ISO 4049, Depth of Cure:**
  - Cylindrical composite specimens are prepared (diameter: 4mm, height: 6mm)
    - Photo-polymerizing from the top surface.
  - The specimens were removed.
  - Any uncured or loosely cured materials were scrapped from the bottom by a spatula.
  - The depth of residual material is measured by digital caliper (∓0.01).
  - Depth of cure = (recorded length divided by 2)
  - **Disadvantages:**
    - Subjective method
    - Inter operational differences
    - Affects the reliability of the results [3,4].

- **Hardness of the top and bottom specimens' surfaces:**
  - Hardness is an indirect approximation of the DC
  - Depth of cure can be evaluated by analyzing microhardness
  - Vickers hardness ratio is calculated by measuring the hardness of the top and bottom of the specimen.
  - VHR = $\frac{H\ (bottom)}{H\ (Top)} x100$
  - VHR ≥ 80% is regarded as the arbitrary unit for an adequately cured material as reported in literature [5].

- **Degree of conversion of the top and bottom specimens' surfaces:**
  - For measuring the degree of conversion several spectroscopic methods could be used as Fourier transform infrared spectroscopy (FTIR), Raman or micro-Raman spectroscopy.
  - A specified threshold cannot be determined for DC. Some researchers suggested the DC of 55% as the threshold.
  - $DC = \left[1 - \frac{R_t}{R_0}\right]$ x 100

R_0 = represents the ratio between the intensity of the reactive vinyl group (C=C) and the internal reference of the **unpolymerized material**.
R_t = represents the ratio between the intensity of the reactive vinyl group (C=C) and the internal reference of the **polymerized material**.[3]

Evaluation methods of polymerization shrinkage for dental resin composite restorative materials

- In polymerization reaction monomers in proximity react to establish a covalent bond. The distance between the two groups of atoms is reduced leading to volumetric shrinkage.
- The magnitude of volumetric shrinkage is affected by different factors such as:
  - Composition and degree of conversion of the matrix.
  - Percentage of the fillers.
  - Presence of organic fillers.
  - Curing time
  - Intensity of the curing light
  - Concentration of photo-initiators in composite resins [6]

Volumetric polymerization shrinkage

Pycnometer (Buoyancy method):
It is based on the Archimedes principle and relies on the buoyancy of a material in water [7].

Water Pycnometer:
ASTM: D792 specification:

- 1g of composite resin is placed in a Teflon split mold

Pressed manually with two flat glass plates resulting in a thin disc (± 2.0 mm thick).

Specific gravity = $\frac{a}{(b+a)-m}$

a = weight of the specimen
b = weight of the Pycnometer filled with water
m= weight of the Pycnometer containing the specimen and water.

- The volume of the specimens (pre and post polymerization) is calculated:

$$\text{Specific gravity} = \frac{\text{Density of the Specimen}}{\text{Density of water}}$$

Where Density of water is a constant at 1gm/cm^3

- Volume of the specimen is

$$\text{Volume} = \frac{\text{Mass}}{\text{Density of the Specimen}}$$

The percentage of volumetric contraction:

$$\text{Percentage shrinkage} = \frac{V_1 - V_2}{V_2} \times 100$$

V_1 =Volume of unpolymerized resin

V_2 = Volume of polymerized resin

- **Disadvantages:**

- Small pores entrapped in the material are not occupied by the water molecules
- Dissolved oxygen is not purged from the sample

values tend to be underestimated [8].

Gas Pycnometer

Gas pycnometer uses the difference in volume occupied by the material and helium gas molecules in a chamber of known volume.

Advantages:

- Temperature independent
- Helium molecules are much smaller than water. Therefore, it can occupy voids in the material.
- Displace some of the dissolved oxygen [7]

So, it gives True & Accurate measurement.

$$V = volume\ of\ the\ cured\ specimen$$

$$S = \frac{\Delta V}{V + \Delta V} \times 100.$$

$$\Delta V = change\ in\ volume\ between\ cured\ and\ uncured\ sample$$
$$S = the\ percent\ shrinkage$$

1. Dilatometer

The mercury dilatometer method uses the variation in height of a mercury column caused by composite shrinkage to calculate total volumetric shrinkage. After Curing, the change in height of the column is monitored in real time.

The composite specimen is placed on a glass slide and immersed in mercury filling a glass capillary tube. A linear variable differential transformer (LVDT) probe floats on the surface to measure the displacement, at the top of the mercury column. [7]

Hg Dilatometer:

Disadvantage:

- Temperature sensitivity of the mercury in the column

To correct this problem, a **thermocouple monitor**s the temperature variation of the mercury caused by the heat liberated by the curing lamp and the corresponding variation in column height is adjusted at the end of the test run.

- Potential health hazards [7].

AcuVol

- Video-imaging device developed for measuring composite shrinkage. That uses a CCD (charge-coupled device) camera to capture and analyze profiles of the specimen.

- Real time capturing.
- A 12 μL composite specimen is shaped into a hemisphere and positioned on a Teflon pedestal.
- The tip of the curing light source is placed 1 mm above the specimen.
- Shrinkage-time curves are obtained from a single view

or with the specimen rotating on the pedestal (multi-view mode).

- Multiview mode allows for correction of asymmetries on the specimen surface.
- The measured values are quite like those obtained with a mercury dilatometer [7].

2. Micro CT

- It provides fast acquisition of high-resolution 3D images even with small objects.
- It corrects the artefacts caused by air bubbles.
- It provides accurate images regardless of the shape of the object and its position.
- Non- destructive method.
- It enables measurement to be made on clinically relevant geometries.
- The variation of the shrinkages values is reasonable & acceptable.
- It enables us to visualize the real deformation vectors generated by curing contraction [9].

a. Linear shrinkage

1. Bonded disk method

- Resin composite disk (8 × 1.5 mm) is placed into a brass ring (16 mm x 1.5 mm) that is bonded to a glass slide.
- The composite is covered with a microscope cover slip (approximately 0.1 mm thick).
- LVDT probe is placed in contact with the center of the cover slip.
- The composite specimen is cured from below through the glass slide.
- As the composite shrinks, it pulls the cover slip down and its deflection in monitored by the LVDT probe.
- Displacement data (in μm) is obtained.
- Shrinkage is calculated by dividing the measured deflection of the cover slip by the initial height of the composite.
- Because the composite specimen must exhibit some stiffness to deflect the cover slip, this method likely measures post-gel shrinkage [7].

2. Electric resistance strain gauge

- Strain gauges are very sensitive to linear dimensional changes
- The gauge is bonded to a substrate
- The gauge is connected to a strain monitoring device
- Linear dimensional changes occurring in the substrate are transferred to the gauge and measured.
- Composite is placed on top of a strain gauge
- Shaped into a hemisphere
- Changes in the Electrical resistance performed by the gauge due to shrinkage of the resin are recorded
- Disadvantage

The difficulty of placing and maintaining samples of flowable materials with enough thickness on the strain gauge [10].

3. Linometer

- The composite specimen is placed between the glass slide and an aluminum disk.
- The disk and glass were greased to avoid adhesion of the composite sample.
- The linear polymerization shrinkage is calculated by the following formula

$$\textbf{Linear shrinkage (LS)} = \frac{\Delta L}{L+\Delta L} \times 100$$

L is the specimen thickness after polymerization

Advantages:

- Simple
- Fast

- Insensitive to temperature changes producing constant results.
- No significant differences between this method and dilatometry

Disadvantages:

- This technique is based on measuring linear shrinkage using contact displacement transducers.
- Potential errors may occur related to the effect of gravity or nonuniform shrinkage of the sample. [10]

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