

**Comparative Evaluation of Polymerization Shrinkage and Microhardness of Nano-ormocer With Posterior Composites - An In Vitro Study.**<sup>1</sup>Dr. Drisya L, Anugraha, PSM Dental College, Thrissur, Kerala<sup>2</sup>Dr. Athina Marium Thomas, PSM Dental College, Thrissur, Kerala**Corresponding Author:** Dr. Drisya L, Anugraha, PSM Dental College, Thrissur, Kerala**Citation of this Article:** Dr. Drisya L, Dr. Athina Marium Thomas, “Comparative Evaluation of Polymerization Shrinkage and Micro hardness of Nano-ormocer With Posterior Composites - An In Vitro Study”, IJDSIR- April - 2023, Volume – 6, Issue - 2, P. No. 338 – 346.**Copyright:** © 2023, Dr. Drisya L, et al. This is an open access journal and article distributed under the terms of the creative commons’ attribution non-commercial License. Which allows others to remix, tweak, and build upon the work non-commercially, as long as appropriate credit is given and the new creations are licensed under the identical terms.**Type of Publication:** Original Research Article**Conflicts of Interest:** Nil**Abstract****Aim:** To compare and evaluate polymerization shrinkage and micro harness of nano-ormocer with posterior composites.**Methodology:** Linear polymerization shrinkage - The restorative materials was placed in disc- shaped Mold of inner diameter 7mm and height 2mm, covered with mylar strip and pressed with a microscope glass slab and photo - activation was performed for 40 seconds. 14 specimens were prepared for each material. The surface of specimens was polished with sandpaper of decreasing grit. After 24 hours at 37°C, specimens were mounted on stubs, gold sputtered and the gap formed between Mold and material were observed using scanning electron microscopy (250 x). Images were taken and gaps were measured in micrometres.

Vickers micro hard ness – 14 - disc shaped specimens (10x2 mm) from each material were fabricated using Mold. The material was placed in one increment and excess materials were removed by compressing the

Mold with mylar strips and glass slides. The top surface of the specimens was light cured for 10s. The glass slide was removed and again light cured for another 10 s. The mylar strips were discarded and discs were removed from the Mold. Any excess material was removed gently with fine polishing discs. The specimens were kept at incubator at 37°C for 7 days prior to testing. For micro hardness measurement, a 100g load was applied for 10 s on the upper surface of the disc using a microhardness tester. Five Vickers hardness number were obtained from each sample.

SPSS statistical program (Version 23) was used to analyse the data. Descriptive data were expressed as mean [ $\pm$  SD]. Numeric values were compared with one-way ANOVA. All statistical analyses were carried out at significance level 0.05**Result:** For polymerization shrink age mean values obtained were Group I nano-ormocer 3.77, Group II: nano hybrid composite 14.73, Group III: bulk-fill composite 21.13 and Group IV: packable composite

34.50. In case of micro hardness mean values were Group I: nano-ormocer 55.02, Group II: nanohybrid composite 51.25, Group III: bulk-fill composite 45.82 and Group IV: packable composite 39.38.

**Conclusion:** The polymerization shrinkage was least for nano-ormocer and highest for packable composites. Whereas, highest microhardness was observed for nano-ormocer and lowest for packable composites.

**Keywords:** Polymerization shrinkage; Vickers micro hardness; Nano-ormocer; Nano hybrid composite; Bulk-fill composite; Packable composite.

### Introduction

Dental composite occupies a paramount position among various restorative materials and remains as an ideal Esthetic material. This can be attributed to following features: excellent esthetics, acceptable longevity and minimal preparation of tooth.<sup>1</sup> Due to these qualities, it has become a material of choice for both anterior and posterior restorations. Since its introduction by Bowen, these materials have undergone development in filler technology, resin and polymerization initiation systems, as well as improvements in the adhesion of dental composites to tooth structure have significantly improved their properties and expanded their clinical utility.

In spite of these improvements, polymerization shrinkage remains as a challenge.<sup>2</sup> During curing of resin composites they undergo dimensional shrinkage. When composite is placed inside a cavity and bonded to surrounding walls, the material deformation is restricted and thereby this leads to development of stress. The outcome associated with development of internal contraction stress can damage the marginal seal of restoration. This results in interfacial gap formation and causes post operative sensitivity; marginal discoloration and secondary caries.<sup>3</sup>

Polymerization shrinkage has always been a universal problem in resin based restorative materials.<sup>4</sup> Polymerization of the composite restoration is directly related to the material's chemical composition like resin matrix, type, morphology and filler contents. So the focus has been shifted to modify changes in the polymer matrix and fillers. As an outcome of this innovation, new resin composites with modified monomers, such as the ormocer and silo Rane resins also with nanofiller content have been developed to reduce long-term clinical problems caused by polymerization shrinkage stress.<sup>5</sup> In the present scenario, along with conventional resin matrix such as bis-GMA, ceramic particles and nano sized filler particles have been incorporated.

To evaluate the efficacy of polymerization in composite resins, several types of laboratory tests are documented. The direct methods include differential thermal analysis, infrared spectroscopy and Raman spectroscopy. Whereas, indirect methods are micro hardness measurement, optical microscopy and scraping test. Surface hardness is used to verify indirectly the degree of conversion of composite resins. Adequate surface hardness of composite restoration is important to obtain optimal clinical performance.<sup>6</sup>

To reduce shrinkage, the main approaches adopted so far are to change the monomer structure or chemistry, filler amount, shape or surface treatment. One of the latest novel material introduced is ormocer-based composite, which has a three dimension ally cross-linked co polymers. The composition of ormocer is ceramic poly siloxane, which claims to have very low polymerization shrinkage.<sup>7</sup> Ormocer is an acronym of organically Modified ceramic. The conventional composites were based on a purely organic matrix, whereas, ormocer have an inorganic as well as organic network. The organic segment has meth acrylate group and inorganic segment

have Si-O-Si network. On activation, the inorganic polycondensation and organic polymerization results in the formation of an inorganic co-polymer.<sup>8</sup>

In the past few years, it has been observed that the size of filler particles integrated in the resin matrix has continuously decreased. When inorganic phases in a composite become nano sized, they are called nano composites. Nano fillers can be prepared by techniques such as flame pyrolysis, flame spray pyrolysis and sol-gel processes. Nanofillers are extremely small filler particles of size 0.1- 100 nm. These fillers are able to increase the overall level due to small particle size. Increase in filler content cause significant reduction in polymerization shrinkage and improve the physical properties of composites.

Clinically, a composite restoration is placed in increments and is cured separately. This is due to the limited depth of cure and to reduce the polymerization shrink age. To overcome these difficulties, bulk-fill composites were introduced. They can be placed in a cavity and cured up to a depth of 4 mm.

Packable composites are based on the concept of PRIMM. This system consists of a resin and ceramic component. The inorganic filler particles are present as continuous network/ scaffold of ceramic fibres. These fibres are composed of alumina and silicon dioxide and the diameter is less than 2.0  $\mu\text{m}$ .

So, the purpose of this study was to compare and evaluate the polymerization shrinkage and micro hardness of nano-ormocer with other posterior composites such as nanohybrid composite, bulk-fill composite, and packable composite.

## **Materials & Methods**

### **Polymerization shrinkage**

The restorative materials were placed in increments in a disc-shaped mold of inner diameter of 7mm and height 2

mm. The materials were then covered with a mylar strip and pressed with a microscope glass slab. 14 specimens were prepared from each material. Photo-activation was performed with the curing tip positioned in close proximity with the Mold/ restorative material interface for 40 seconds. After 15 minutes, the surface of specimens was polished with sandpaper of decreasing grit. After 24 hours at 37<sup>0</sup> C, the specimens were mounted on stubs, gold sputtered and the gap formed between the Mold and restorative materials were observed using scanning electron microscope (250x). The gap formed between the mold and the resin composite was observed at 4 points located in positions corresponding to 3, 6, 9 and 12 hours of a clock face. Images were taken and the gaps were measured in micrometres.

### **Vickers microhardness**

14 disc-shaped specimens (10x2 mm) from each material were fabricated using the mold. The material was placed in increments and excess materials were removed by compressing the mold with mylar strips and glass slides. The top surface of the specimens was light cured for 10 seconds each using a LED curing light (Blue phase N) with a wave length of 385 – 515 nm and irradiance of 1200 mW/cm<sup>2</sup>. The glass slide was removed and again light cured for another 10 seconds. The mylar strips were sub sequent discarded and the discs were removed from the mold. Excess material was removed gently with fine polishing discs. The specimens were kept at incubator at 37<sup>0</sup> C for 7 days prior to testing. For micro hardness measurement, a 100g load was applied for 10 seconds on the upper surface of the disc using a microhardness tester. Five Vickers hardness numbers were obtained, and the mean value was taken from each sample.

SPSS statistical program (Version 23) was used to analyse the data. Descriptive data were expressed as mean [ $\pm$  SD]. Numeric values were compared with one-way ANOVA. All statistical analyses were carried out at a significance level of 0.05.

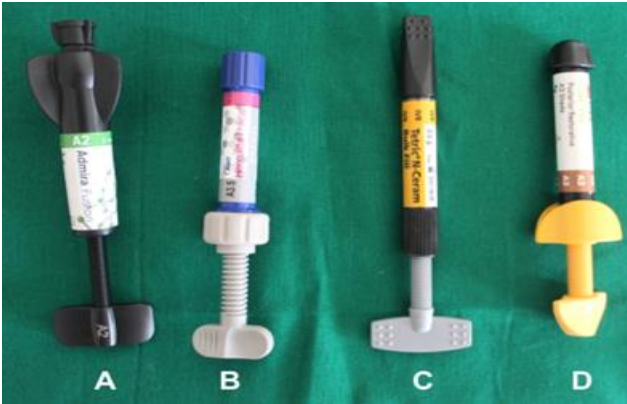


Figure 1:

- A) Group I: Nano-ormocer
- B) Group II: Nanohybrid composite
- C) Group III: Bulk-fill composite
- D) Group IV: Packable composite



Figure 2: Restorative material placed in increments within mold

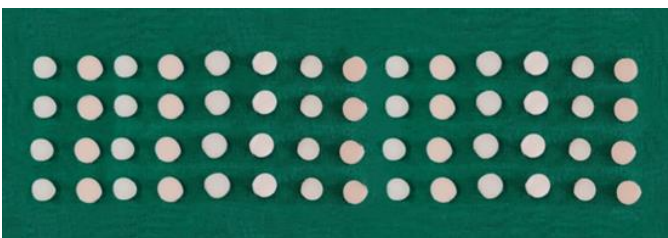


Figure 3: Specimens for polymerization shrinkage

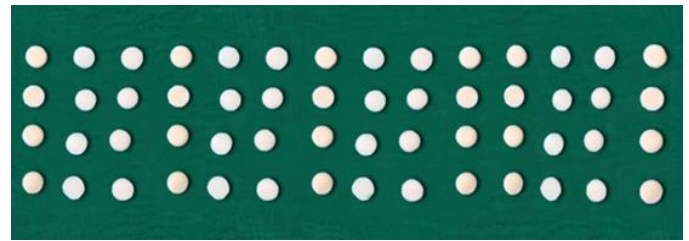


Figure 4: Specimens for Vickers microhardness



Figure 5: gold sputter



Figure 6: Scanning Electron Microscope

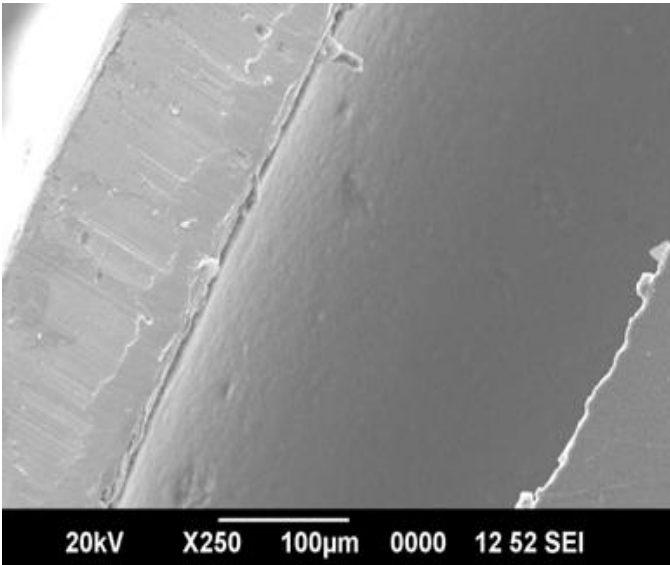


Figure 7: SEM image of Group I

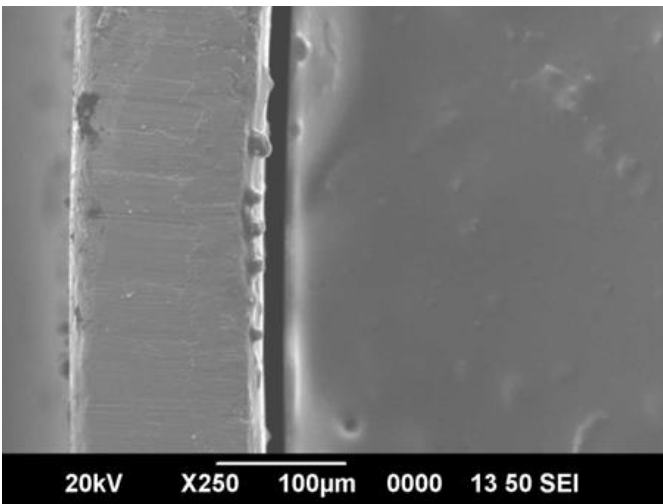


Figure 8: SEM image of Group II

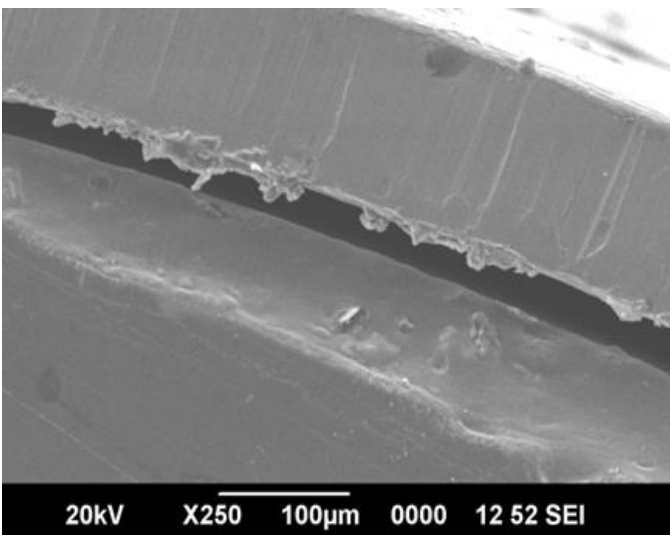


Figure 9: SEM image of Group III

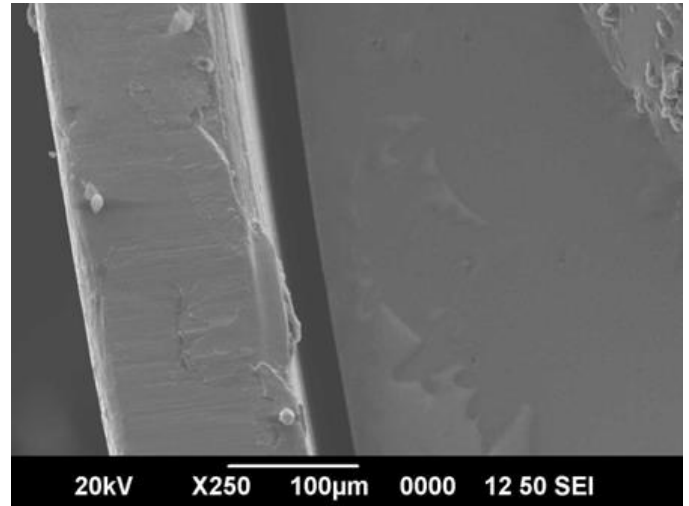


Figure 10: SEM image of Group IV



Figure 11: Vickers microhardness tester

### Result

The values obtained for polymerization shrinkage and microhardness for each restorative material have been tabulated (Tables 1 and 2).

The results are expressed as mean and standard deviation (Table 3 and 4). Numeric values were compared with one-way ANOVA.

All statistical analyses were carried out at significance level 0.05 (Tables 5 and 6).

Table 1: Mean and standard deviation of groups (Polymerization shrinkage)

| Polymerization Shrinkage |         |         |         |                |
|--------------------------|---------|---------|---------|----------------|
|                          | Minimum | Maximum | Mean    | Std. Deviation |
| Nano-ormocer             | 1.60    | 5.12    | 3.7786  | 1.05038        |
| Nanohybrid composite     | 9.12    | 15.55   | 11.8764 | 1.87522        |
| Bulk-fill composite      | 17.76   | 24.99   | 21.1314 | 2.58888        |
| Packable composite       | 26.83   | 42.44   | 34.5107 | 4.55296        |

Table 2: Mean and standard deviation of groups (Microhardness)

| Microhardness        |         |         |         |                |
|----------------------|---------|---------|---------|----------------|
|                      | Minimum | Maximum | Mean    | Std. Deviation |
| Nano-ormocer         | 52.85   | 57.46   | 55.0257 | 1.33502        |
| Nanohybrid composite | 48.61   | 52.86   | 51.2529 | 1.40072        |
| Bulk-fill composite  | 38.18   | 48.55   | 45.8279 | 2.66639        |
| Packable composite   | 37.32   | 42.33   | 39.3836 | 1.58531        |

Table 3: Analysis of variance (Polymerization shrinkage)

| ANOVA                    |                |    |             |         |         |
|--------------------------|----------------|----|-------------|---------|---------|
| Polymerization Shrinkage |                |    |             |         |         |
|                          | Sum of Squares | Df | Mean Square | F       | P value |
| Between Groups           | 7307.903       | 3  | 2435.968    | 304.109 | .0001   |

|               |          |    |       |  |  |
|---------------|----------|----|-------|--|--|
| Within Groups | 416.529  | 52 | 8.010 |  |  |
| Total         | 7724.432 | 55 |       |  |  |

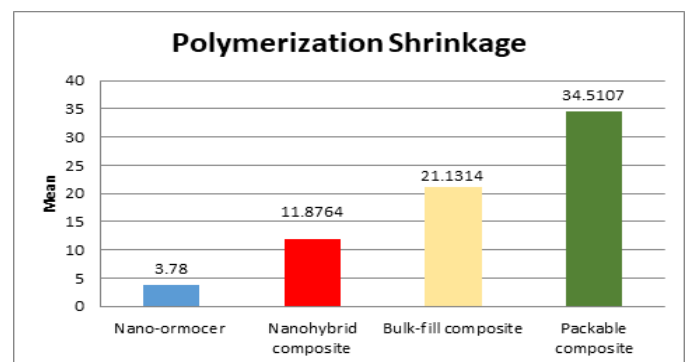
Table 4: Analysis of variance (Microhardness)

| ANOVA          |                |    |             |         |         |
|----------------|----------------|----|-------------|---------|---------|
| Microhardness  |                |    |             |         |         |
|                | Sum of Squares | Df | Mean Square | F       | P value |
| Between Groups | 1943.729       | 3  | 647.910     | 193.881 | .0001   |
| Within Groups  | 173.773        | 52 | 3.342       |         |         |
| Total          | 2117.501       | 55 |             |         |         |

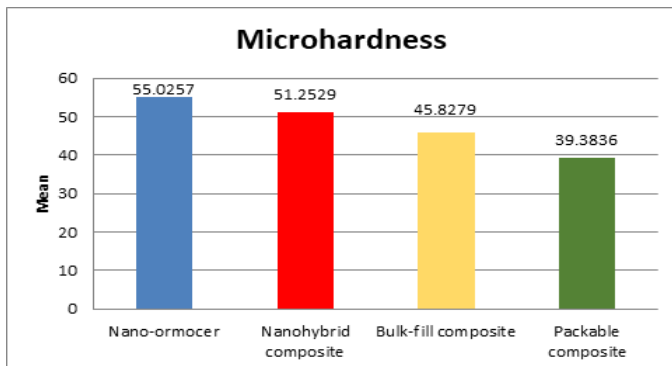
From the results obtained, it was observed that nano-ormocer showed the least polymerization shrinkage followed by nano hybrid composite, bulk-fill composite and packable composite (Graph 1).

In case of Vickers micro hardness, nano-ormocer had highest micro hardness and packable composites had the lowest micro hardness (Graph 2). It was also observed that statistically significant difference was present between all the groups for both parameters ( $p < 0.05$ ).

Graph 1: Mean Polymerization shrinkage ( $\mu\text{m}$ )



Graph 2: Mean Vickers microhardness (VHN)



## Discussion

Direct composite restorations are one of the most prevalent dental interventions in the human body, with more than five hundred million composite restorations being placed every year around the world.<sup>9</sup>The optimal performance of these restorations depends on proper polymerization of the resin component, which is characterized by transforming monomers into polymers, and it is accompanied by volumetric reduction of the material. Polymerization shrinkage of resin-composite occurs due to the conversion of the monomer molecules into polymer structure which is accomplished by replacing the van der Waals spaces by covalent bonds and consequently reducing the free volume. The defects that develop in the interfacial bonding are due to the polymerization stresses generated during the restorative placement procedure and later due to the functional, thermal and mechanical stresses.

Even with the advancements of restorative materials over the past few years, polymerization shrinkage still remains as a clinical dilemma. Typically, dental composites used in restorative procedures exhibit volumetric shrinkage ranging from less than 1% up to 6%, depending on the formulation and the curing conditions.<sup>10</sup> A consequence of shrinkage stress can be debonding along the restoration/tooth interface, which further leads to marginal gaps, micro-cracking within the restorative material or the tooth structure, marginal discoloration and cuspal flexure.

Several experimental methods have been developed to measure polymerization shrinkage of composites and resin cements. In this study, SEM was the method used to measure polymerization shrinkage. It was mainly due to its ability to magnify and reveal details at restorative margins and the gap created can be measured. In relation to other techniques, it is a simplified process and doesn't cause any additional errors in the specimen materials. The other parameter which was evaluated in this study was the microhardness of the restorative material. In this present study, we used Vickers microhardness test, since it is more suitable for determining hardness of brittle materials and also for measuring hardness of tooth structure.<sup>11</sup>

In this study, polymerization shrinkage and microhardness of nano-ormocer was compared with nano hybrid composite, bulk-fill composite and packable composite. From the results obtained, it was observed that nano-ormocer showed least polymerization shrinkage followed by nano hybrid composite, bulk-fill composite and packable composite ( $p = 0.0001$ ). Similarly, in case of microhardness, nano-ormocer showed highest value which was followed by nanohybrid composite, bulk-fill composite and packable composite ( $p = 0.0001$ ). So it can be inferred that nano-ormocer showed better polymerization efficiency compared to other posterior composites.

According to present study, ormoocer showed superior most properties among the tested materials. This can be attributed to the modified organic matrix, formed by monomers with a single polymerizable end. The other end is formed by an alkoxy group, resulting in an inorganic area, bonded to other monomers by a condensation reaction, converting the monomer precursors, creating a complex structure with the formation of the Si-O-Si chain in the inorganic area of

the polymer. The combination of this inorganic-organic matrix and filler particles in high concentration would result in lower polymerization shrinkage and superior micro hardness.

The nano hybrid composite used in this study have an organic matrix which is a combination of bis-GMA and TEGDMA. The filler content is aluminium - barium-silica based glass which is dispersed in silica dioxide. The filler particle size is 20-50 nm and the filler loading is 78.4 Wt% and 59 vol%. These contribute to increased polymerization efficacy of nano hybrid composite when compared to bulk-fill and packable composite.

In case of bulk-fill composite, the organic matrix is a combination of bis-GMA and UDMA and contain pre polymerized fillers. The filler loading is 60-61 vol% and 79-81 Wt %, of which 17% are pre-polymerized fillers. Due to presence of these pre polymerized fillers an inferior performance in comparison to nano hybrid composite was observed in this study.

The packable composite showed the least polymerization efficacy in this study. This could be due to its structural composition which includes bis-EMA, TEGDMA and UDMA as resin matrix and the filler particles are zirconia/silica of size 0.6µm. It has the least filler loading of 61%.

Bacchi et al in his study concluded that ormocer showed lowest polymerization shrinkage compared to methacrylate based composite resins, which is in accordance with this study. An ormocer is a hybrid molecular structure. This combines organic and inorganic components at nanoscopic scale through the sol-gel method, and the main characteristic of this type of material is the incorporation of organic groups linked to the inorganic backbone. Once the inorganic component of the ormocer structure presents no shrinkage during polymerization, the expected contraction and stress

would be reduced which could explain the lower shrinkage of the ormocer based restorative material.<sup>12</sup> Tomar H et al also stated that ormocer showed lower shrinkage compared to conventional resin-based composites.<sup>7</sup>

A comparative study was performed by Tagtekin et al, in which ormocer showed higher hardness compared to conventional hybrid resin composite. This was attributed to influence of the fillers themselves, the filler load level and the filler-matrix interactions. In ormocer the filler material consists of a special glass ceramic and highly disperse silica which was incorporated into cross-linked inorganic and organic matrix network.<sup>13</sup>

### Conclusion

From the results obtained, it was seen that Nano-ormocer had lowest polymerization shrinkage which was followed by nano hybrid composite, bulk-fill composite and packable composites. Due to better physical properties, nano-ormocer can be used as a restorative material in most of the scenarios to bring out better clinical performance as well as good longevity.

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