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ORIGINAL ARTICLE

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Evaluation of monomer conversion, Vickers hardness, compressive strength & water sorption of commercial dental composite resins

Avaliação da conversão de monômeros, dureza Vickers, resistência à compressão e sorção de água de resinas compostas dentárias comerciais

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ABSTRACT

Objective: The objective was to compare four commercially available resin-based composites so that clinicians can select an economic material that has better monomer conversion and improved mechanical properties with lower water sorption. **Material and Methods:** Four commercially available resin-based composites were used. These included "Z350" and "Z250" by 3M, "Charisma" by Heraeus, and "All Purpose" by Dentex. Fourier transform infrared spectroscopy was done in attenuated total reluctance mode before curing and after curing to evaluate the degree of conversion. For hardness and compressive strength, specimens (n=5) were cured from both sides followed by storing them in distilled water. Then they were placed in an oven at 37 °C for 24 h, and tests were performed. The water sorption study was done for 7 days. **Results:** One-way ANOVA and then post hoc Tukey's test ($p \leq 0.05$) were done to analyze the data. The pattern of degree of conversion was Z250>Z350>Charisma>All Purpose. The mean hardness value of Z250 was the highest followed by Charisma, Z350, and All Purpose. In the case of compressive strength, the pattern was Charisma>Z350>Z250>All Purpose. Z250 had less water sorption followed by All Purpose, Z350, and Charisma. **Conclusion:** According to the obtained results of this *in-vitro* study Z250 can be a resin resin-based composite of choice for clinicians as it has all the acceptable results and is a mid-range in price.

KEYWORDS

Compressive strength; Degree of conversion; Hardness; Resin-based composites; Water sorption.

RESUMO

Objetivo: O objetivo foi comparar quatro compósitos à base de resina disponíveis no mercado para que os clínicos possam selecionar um material económico que tenha uma melhor conversão de monômeros e propriedades mecânicas melhoradas com menor sorção de água. **Material e Métodos:** Foram utilizados quatro compósitos à base de resina disponíveis no mercado. Estes incluíam a "Z350" e a "Z250" da 3M, a "Charisma" da Heraeus e a "All Purpose" da Dentex. A espetroscopia de infravermelhos transformada de Fourier foi efectuada no modo de relutância total atenuada antes da cura e após a cura para avaliar o grau de conversão. Para a dureza e a resistência à compressão, os espécimes (n=5) foram curados de ambos os lados e depois armazenados em água destilada. Em seguida, foram colocados numa estufa a 37 °C durante 24 h e foram efectuados testes. O estudo da absorção de água foi efectuado durante 7 dias. **Resultados:** ANOVA de uma fator e, em seguida, teste post hoc de Tukey (p ≤ 0,05) foram feitos para analisar os dados. O padrão do grau de conversão foi Z250>Z350>Charisma>All Purpose. O valor médio de dureza do Z250 foi o mais elevado, seguido do Charisma, do Z350 e do All Purpose. No caso da resistência à compressão, o padrão foi Charisma>Z350>Z250>All Purpose. A Z250 teve menos sorção de água, seguida pela All Purpose, Z350 e Charisma. **Conclusão:** De acordo com os resultados obtidos neste estudo in vitro, a Z250 pode ser um compósito à base de resina de escolha para os clínicos, pois apresenta todos os resultados aceitáveis e tem um preço médio.

PALAVRAS-CHAVE

Resistência à Compressão; Grau de conversão; Dureza; Compósitos resinosos; Sorção de água.

INTRODUCTION

Dental caries is well-known as tooth decay, and it is a predominant disease that has an impact on the human population globally [1]. These defects may be rehabilitated by specially designed dental materials called dental restorative materials or filling materials which are manufactured to restore the function, integrity, and structure of the missing structure [2]. Previously, several novel restorative materials have been manufactured that have superior predictability and reliability for dental clinicians [3]. Commonly used dental restorative materials include resin-based composites (RBCs), glass ionomer cement, amalgams, compomers, and ceramics. Among these, RBCs have the advantage of esthetics as they are available in different shades and can resemble natural tooth color [4,5]. RBCs are primarily composed of dimethacrylate resin, fillers, and silane coupling agents to enhance the bond between non-organic fillers and organic matrix. Moreover, agents such as camphorquinone, diethyl amino benzoate, benzoyl peroxide, butylated hydroxy toluene, etc. are added which promote, enhance, or control polymerization reaction [6].

RBCs are now essential materials of contemporary restorative dentistry [7]. They are customized based on need as restorative, sealants, cement, or provisional materials because of their practical adaptability and aesthetic benefits [8]. There are many clinical situations such as closing of diastema, carious lesions in anterior regions, treatment of discoloration, and dental trauma of anterior teeth in which only RBCs can be used due to aesthetic reasons [9]. The main concern of RBCs is sensitivity due to monomer elution because of incomplete polymerization [10]. Besides this, the prime cause of restoration failure is the breakage of RBCs [11].

Although clinical outcomes have improved due to ongoing advancements in formulations of RBCs, choosing the best material remains a challenge for practitioners [12]. The performance and long-term success of these materials are greatly influenced by factors such as monomer conversion, mechanical properties, and water sorption [13].

Various in-vitro tests are done to analyze the properties of RBCs to evaluate their longevity and to judge the problems in present RBCs. These include water sorption, degree of conversion [14,15], hardness, and compressive strength [16]. The mechanical strength and durability of the composite are greatly influenced by monomer conversion, which is the degree to which resin monomers are polymerized during curing [17]. Low conversion rates may have detrimental effects on patient safety and the longevity of the restoration by causing less-than-ideal mechanical properties, increased wear, and the possible release of unreacted monomers [18]. Moreover, mechanical properties such as compressive strength, and hardness are crucial for withstanding harsh oral environments as variable chewing forces, food of various hardness, temperature and pH may contribute to further deterioration [19]. Additionally, water sorption is also crucial since too much moisture absorption can cause hydrolytic deterioration, dimensional instability, and discoloration, which will ultimately impair the appearance and functionality of RBCs [20].

Therefore, for long-term promising clinical applications of the materials, a high degree of conversion and superior mechanical properties are very important. Various companies have developed different RBCs which are available in the market under different brand names and different price ranges claiming all have optimal results and markets them with attractive strategies to catch the interest of clinicians. In this era of inflation for cost cutting many clinicians opt for cheaper available dental composite.

The aim of the study was to compare different properties of various commercially available RBCs in the market. With the help of evidence-based data, we hope to help clinicians choose the best restorative materials that maximize long-term success while considering the price in this state of inflation.

MATERIAL AND METHODS

Materials

Four commercially available RBCs are available in the market were analyzed. “Z350” and “Z250” by 3M, “Charisma” by Heraeus, and “All Purpose” by Dentex were tested. The composition of all the types of RBCs provided by the manufacturers used in this experiment are explained in Table I.

Sample preparation

The mold was placed on a glass slab and the material was poured into the mold carefully. A single increment layer was placed, and to prevent oxygen inhibition layer samples were covered with mylar strip. Then, the light curing unit having a wavelength of 470nm (LED, Woodpecker) was used to cure both sides of the samples. After curing, the samples were carefully retrieved from the mold followed by polishing with various grit papers (600, 800, and 1200) under a steady flow of water. Five samples for every test were made and subjected to testing. The sample size was calculated as per the following equation keeping the power of study equal to 90% and level of significance equal to 5% by the given formula below:

$$n = \frac{\left(Z_{1-\beta} + Z_{1-\frac{\alpha}{2}}\right)^2 \left(\sigma_1^2 + \sigma_2^2\right)}{\left(\mu_1 - \mu_2\right)^2} \quad (1)$$

$Z_{1-\beta}$ = Z score for power of study at90% = 1.28 ;

$Z_{1-\frac{\alpha}{2}}$ = Z score for level of significance at5% = 1.96 ;

μ_1 - μ_2 (176.45- 54.20) = mean difference (compressive strength) = 122.5;

σ_1^2 + σ_2^2 [$(50.59)^2$ + $(8.62)^2$] = standard deviations of the groups = 2633.64 [21];

Calculated sample size for each test = 2;

Sample Size is taken for each test = 5.

Characterizations

Degree of conversion (DOC)

Fourier Transform Infrared Spectroscopy (FTIR) was used to investigate DOC. It was done before as well as after the curing of samples. FTIR (Thermo Nicolet 6700, USA) with ATR cell (MTEC, USA) was used as a detector. The detector is placed on the sample and the scan is started. 256 scans were performed at a resolution of 8 cm^{-1} and spectra were collected over the region 4000–400 cm^{-1}. OMINIC software (8.1.11, Driver version 8.1, Firmware version 2.10) was utilized to analyze the data peaks which were matched from the software library. DOC was computed by the given formula below:

$$DC = 100 \times \left[1 - \left(R\ polymerized / R\ unpolymerized\right)\right] \quad (2)$$

where DC represents the degree of conversion, R denotes the ratio of the peak height of polymerized and unpolymerized samples [22,23].

Vickers hardness (VHN)

VHN was evaluated for samples which were disc-shaped samples having dimensions of 2 mm × 8 mm (height x diameter). A total of 5 samples

Table I - Information on the composition of dental materials is provided by the manufacturer

Composite Resin	Classification	Filler	Resin
Z350	Conventional Nanoparticle	Zirconia / silica 75wt. % or 59.5 vol%	*bis*-GMA, UDMA, TEGDMA, BISEMA
Z250	Nanohybrid	Zirconia/ silica 60 vol%	*bis*-GMA, UDMA, *bis*-EMA
Charisma	Universal hybrid	barium aluminum fluoride glass, pre-polymerized filler 61 vol.%	*bis*-GMA matrix
All Purpose	Nanohybrid	Barium Glass Nano Silica 34wt.%	*bis*-GMA matrix

Where *bis*-GMA: stands for Bisphenol A glycidal methacrylate; UDMA: Urethane dimeth acrylate; *bis*-EMA: ethoxylated bisphenol A glycol dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate.

were made in Teflon mold. After keeping samples in deionized water for 24 h and then in a desiccator for 1 hour, the hardness was determined according to ASTM E384-11 [24] when a 300 g load was applied for 10 seconds on each sample site via hardness tester (HVS 1000). The mean value was calculated to determine VHN for samples as three indentations were produced on each sample [25].

Compressive strength (CS)

CS was assessed for samples that were cylindrical shaped having dimensions of 4 mm x 6 mm (diameter x height). A total of 5 samples were made in Teflon mold. The mold was placed on a glass slab and the material was poured into the mold carefully. A single increment layer was placed, and to prevent oxygen inhibition layer samples were covered with mylar strip. Then, the light curing unit having a wavelength of 470nm (LED, Woodpecker) was used to cure both sides of the samples. After curing, the samples were carefully retrieved from the mold followed by polishing with various grit papers (600, 800, and 1200) under a steady flow of water. The samples were kept in deionized water for 24 h then in a dessicator for 1 hour and then subjected to testing. The CS was determined via a Universal Testing Machine having 0.5 mm.min^{-1} of cross-head speed, according to ISO 4049-49. Peak compressive stress (σ_c) was calculated after the peak compressive load (P) withstood by each sample was noted [26].

Water sorption

Water sorption samples (n=5) were made disc-shaped and it was determined in accordance with the procedure mentioned in ANSI/ADA Specification No. 27-1993 (ISO 4049). Samples were placed at 37 °C in an oven for 24 h. Then, they were taken out, placed for 1 h in a desiccator, and weighed using a balance having an accuracy of 0.1 mg. The weight of the sample was measured [Analytical Balance, SHIMADZU, JAPAN (10mg)] and recorded in dry and standard conditions. Then, the samples were submerged for 7 days in water at 37 °C. After 7 days they were removed followed by blotted drying and weighing. The percentage weight increase for each sample was computed by using by the given formula:

$$\Delta W = \left[\left(W_f - W_i\right) / W_i\right]$$

where W_f denotes the water saturation after 7 days of immersion (final weight) while W_i denotes the initial weight [25].

Statistical analysis

Data were statistically analyzed by IBM SPSS 23 (Armonk, NY, USA) with mean ± standard deviation. As the data was normally distributed One-way ANOVA followed by Post Hoc Tukey's test was used for analysis while keeping the significance ≤0.05.

RESULTS

Fourier Transform Infrared (FTIR) Spectroscopy

Z250 showed the highest DOC (69%) followed by Z350 (55.2%), and Charisma (32.6%) while the least degree of conversion was shown by All Purpose (15%) as shown in Table II. There was a statistically significant difference between DOC of all the RBCs compared. The DOC was calculated by graphs shown in Figures 1 and 2.

Vickers hardness (VHN)

The mean VHN of Z250 was highest (97.57 VHN) followed by All Purpose (93.55 VHN), Z350 (89.73 VHN), and Charisma (59.36 VHN) respectively. The mean hardness along with standard deviation is shown in Table II. There was a significant difference statistically between Charisma and all other commercial composites while no significant difference amongst the other three when compared between each other.

Table II - Mean Degree of Conversion, Hardness. Compressive strength and water sorption of commercial composite

Commercial Composite	Degree of conversion (%) ± SD	Hardness (VHN) ± SD	Compressive Strength (MPa) ± SD	Water sorption (µg/mm^3) ± SD
All Purpose	15 ± 3.2	59.36 ± 8.1	122 ±10.2	0.45 ± 0.28
Z250	69± 1.7	97.57 ± 7.5	184 ± 9.3	0.33 ± 0.28
Charisma	32.6 ± 2.4	93.55 ± 10.7	192 ± 12	0.82 ± 0.29
Z350	55.2 ± 1.4	89.73 ± 7.7	187 ± 7.7	0.53 ± 0.30

SD: Standard Deviation.

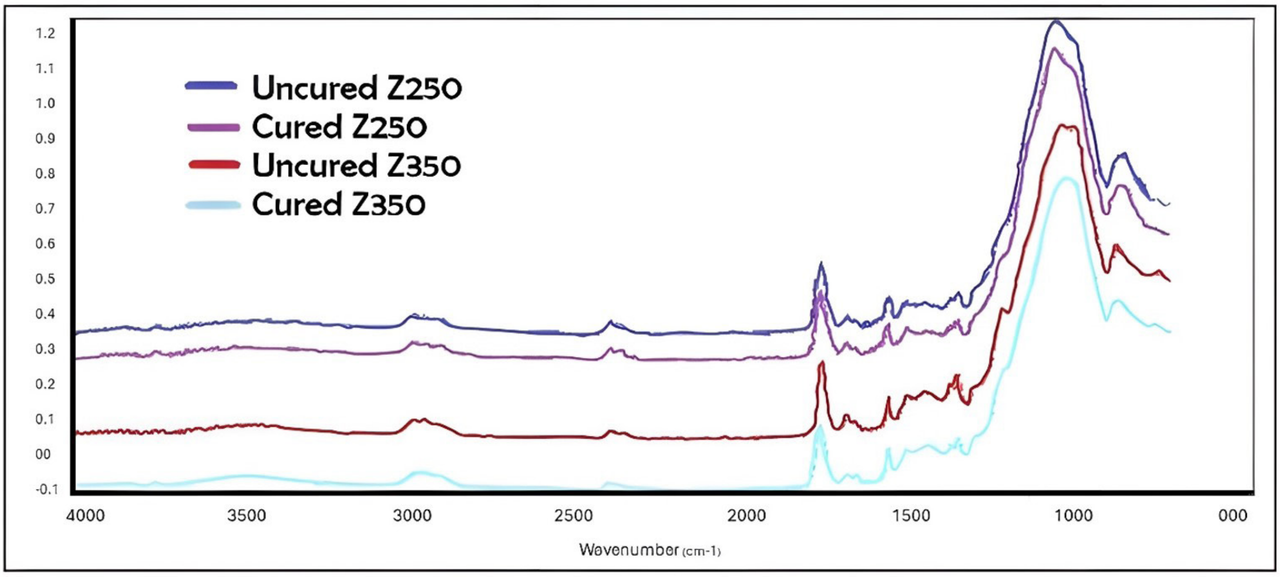

Figure 1 - Degree of conversion of All purpose and Charisma.

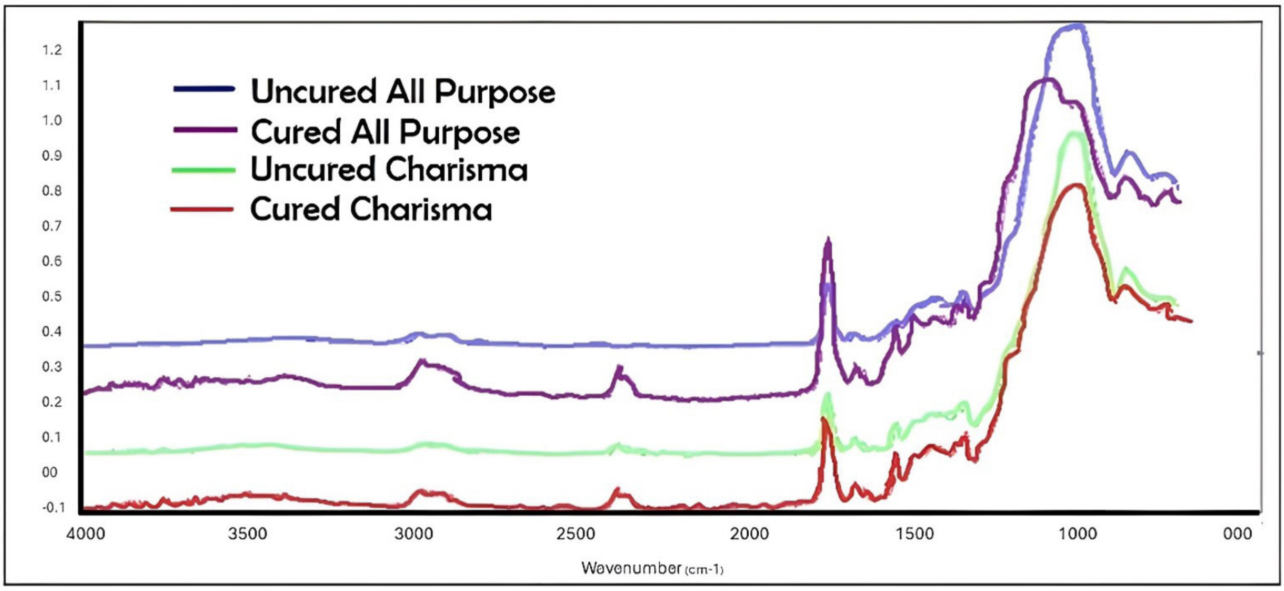

Figure 2 - Degree of conversion of Z250 and Z350.

Compressive strength (CS)

The mean CS values of Charisma were highest followed by Z350, Z250, and All Purpose. The highest compressive strength was seen in charisma which was 192± 12 MPa followed by Z350 (187 ± 7.7 MPa), Z250 (184 ± 9.3 MPa), and the least compressive value was observed in All Purpose (122±10.2 MPa). There was a significant difference statistically between All Purpose and all other commercial RBCs while no significant difference among the other three when compared between each other.

Water sorption

Z250 had the least water sorption (0.33 ± 0.28 $\mu g/mm^3$) followed by All Purpose (0.45 ± 0.28 $\mu g/mm^3$), Z350 (0.53 ± 0.30 $\mu g/mm^3$) while Charisma had the highest water sorption (0.82 ± 0.29 $\mu g/mm^3$) as shown in Table II. There was a significant difference statistically between Charisma and all other commercial composites while no significant difference amongst the other three when compared between each other. The mean water sorption of samples along with the standard deviation after 7 days is shown in Table II.

DISCUSSION

In dentistry, RBCs have been used by dental clinicians for many years. Data related to the properties of every product of RBCs is not available in black and white. As well as the ranking of various products in accordance with their laboratory findings does not indicate their clinical performance [27,28]. This *in-vitro* study was conducted to analyze the mechanical properties of different composites that are present in the market with different price range and are used in dental practices by our common dentist.

Both mechanical and physical properties of RBCs are directly proportional to the DOC in a polymerized sample. DOC depends upon the amount of double bonds present after curing in comparison to the double bonds present before curing [29]. A higher degree of monomer conversion means that there is a less unreacted monomer in the mixture which may reduce the chances of monomer leaching into the oral cavity, which in turn enhances the longevity of restoration [30]. Dental materials when placed in oral cavity absorb water and leach out unreacted monomer. Moisture in the restoration acts as a plasticizer resulting in detrimental mechanical properties and compromised biocompatibility [31]. A minimal degree of conversion for a dental restorative material has not been clearly recognized till now but the acceptable range is above 55% [32]. The FTIR results showed that DOC is the maximum for Z250 followed by Z350 and the relatively low value was shown by Charisma and All Purpose. Both Charisma and All Purpose had DOC less than the acceptable value. Kim et al. [33] also showed in their study that the conversion of Z250 is better than Z350.

Teeth are continuously being subjected to masticatory load and stresses in the oral cavity. So, when a restoration or a prosthetic material is placed it also shares the load applied. Continuously load and stresses sometimes result in restoration failure [34]. Vickers hardness test was done to measure the surface hardness of the selected dental composites. Surface hardness is an important virtue because the relationship between surface hardness and the physical properties of a material is well known for good longevity of restoration [35]. The mean hardness was maximum for Z250, “All purpose” value was comparable to Z250. Mota et al. [36] in their article evaluated Vickers hardness of Z250 and results as comparable with our study. Okulus and Voelkel [37] in their article compare Charisma with nanocomposites which gave good hardness results, but when compared with hybrid composite in our study the results were not comparable.

Compressive strength is the resistance to fracture under compression and shows the potential of a material to withstand vertical stresses. It is important in extreme stress-bearing areas such as the posterior region. Measuring compressive strength is very beneficial for determining materials such as RBCs as they are generally weak and brittle in tension. Compressive strength is deemed a key index of success as a greater compressive strength is essential to resist masticatory forces, though the accurate value is unknown. The composition of filler and filler load may have a substantial effect on the mechanical properties. The mechanical properties mostly increase with filler load for the similar type of materials [38]. “All purpose” showed relatively low compressive strength as compared to Z250, Z350, and Charisma. The low compressive strength of “All Purpose” may be due to low degree of monomer conversion. In a study where the compressive strength of Z250 was evaluated, the results were similar to the results in our study [36]. Saleem et al. [39], also evaluated the compressive strength of Z350, although the results were better comparable to our study. In another study, Sonwane and Ramachandran [40] evaluated the compressive strength of Z350 and Charisma and the mean values obtained were less than the mean values found in our study.

Water sorption to a specific degree is unavoidable in the oral cavity as restorative materials are in constant contact with saliva. Moreover, they are exposed to masticatory load, beverages, and food, thus changes in pH are also present. The sorption phenomenon of RBCs in an oral environment has adverse effects on them [41]. As, the bond between the resin matrix and filler particles is broken by the process of hydrolysis when water molecules infiltrate the resin/filler interface through the process of diffusion, therefore resulting in the deterioration of filler particles and resin matrix [42] and initiating adverse effects on the mechanical properties of RBCs, which consequently impacts the longevity of the RBCs. Moreover, allergic reactions may occur in the patients due to eluted monomers and additives in the oral cavity [43]. In RBCs, water uptake is a diffusion-controlled mechanism and mostly takes place in a resin matrix. The diffusion coefficient is inversely proportional to the amount of water in the resin matrix [44]. Thus, a decrease in the amount of water uptake is to be anticipated with an increase in storage time. Regarding sorption, just Z250 had a value which was in accordance with ISO 4049, i.e., water sorption was below 40 $\mu g/mm^3$ [45]. “Charisma” showed the highest water sorption followed by Z350, All Purpose and Z250. Kumar and Sangi [46] evaluated the water sorption of Z250 which was much more than the results obtained in our study. Similarly, in another study, Syed et. al assessed the water sorption of Z350 and the results were comparable to our study [25].

CONCLUSION

Z250 showed a better degree of conversion, hardness, and less water sorption. All Purpose showed comparable hardness to Z250, but its degree of conversion and compressive strength was low. Charisma has the maximum compressive strength, but its mean hardness value was very low and water sorption value was high. Z350 showed a comparable degree of conversion and mean hardness, compressive strength, and water sorption values. Thus, according to this study, Z250 can be the RBC of choice for clinicians as it has all the acceptable results and is a mid-range in price.

Author’s Contributions

SBL: Methodology, Formal Analysis. US: Conceptualization, Methodology, Formal Analysis. NA: Writing – Original Draft Preparation. ME: Writing – Review & Editing. SL: Methodology, Validation. AA: Supervision. ZK: Resources.

Conflict of Interest

The authors declare no conflict of interest concerning the publication of this article.

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Regulatory Statement

This was an in-vitro study which was conducted according protocols mentioned in ISO 4049:2021 for Resin based Dental Composites.

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