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Investigation of Mechanical Properties, Shade, and Water Sorption/Solubility of Commercial Composite Resins

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A B S T R A C T

Objectives: Assessment of mechanical properties of composite resins is important since they can affect the clinical performance and longevity of restorations. This study aimed to assess and compare different properties of dental composite resins available in the market.

Materials and Methods: In this comparative study, samples of four different dental composite resins (Edge COM, Saremco, FGM, and Kulzer) were tested according to ISO 4049:2019 guidelines. Flexural strength (by using a universal testing machine), depth of cure (using the ISO 4049 scrape technique), radiopacity (with aluminum step wedge), water sorption/solubility (by using immersion and drying cycles), and shade (by using Vita Easy Shade) of the samples were assessed. Data were analyzed by one-way ANOVA and Tukey's post-hoc test (alpha=0.05).

Results: No significant difference was found in flexural strength among the four groups (P>0.05). The depth of cure of FGM was significantly higher than that of other groups (P<0.05). Solubility and water sorption were the highest in Kulzer, and the lowest in Edge COM (P<0.05). All tested composites met the necessary radiopacity standards for precise radiographic diagnosis; radiopacity was higher in Saremco and FGM groups. None of the composite resins matched the reference A2 shade.

Conclusion: Although no significant difference was found in flexural strength of the tested composites, they had differences in other properties. Edge COM exhibited high flexural strength, while Saremco had optimal radiopacity.

Keywords: Composite Resins; Mechanical Tests; Flexural Strength

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INTRODUCTION

Dental composite resins are commonly used in restorative dentistry due to their favorable esthetics and direct-filling capability [1]. Composition of materials determines their strength and color stability. Fillers play a significant role in enhancing the strength, stiffness, and wear resistance of composite resins. However, excessive fillers can potentially diminish the translucency and adversely affect the esthetic appearance of composite resins [2]. The size, shape, and distribution of fillers also affect material properties [2]. Bis-GMA, TEGDEMA, and UDMA are the main matrix components of composite resins. The filler particle technology has undergone significant advancements to improve the

properties of composite resins [3].
Mechanical properties of dental composite resins such as their flexural strength, fracture toughness, and fatigue crack growth determine their clinical performance and longevity [4].
Evidence shows that the flexural strength of dental composite resins is significantly influenced by their filler content, resin chemistry, and filler morphology [5]. Additionally, the degree of conversion, polymerization mode, and microstructure play a significant role in determining the flexural strength of composite resins [6].
Water sorption and gradual dissolution in oral fluids are among other important parameters that can decrease the durability of composite resins [7]. Water sorption can adversely affect the flexural strength and modulus of elasticity of composite resins over time by plasticizing and disrupting the polymer network of the resin matrix [8]. Additionally, solvent sorption and release of components could lead to hydrolytic degradation and adversely affect the durability of composite resins in the long-term [9]. Decreased mechanical properties due to water sorption can contribute to greater susceptibility to wear, fracture, and secondary caries, leading to reduced clinical performance [10].
The depth of cure refers to the maximum thickness of a material that can be effectively cured, ensuring adequate polymerization throughout the restoration, which can affect its physical and mechanical properties [11]. The depth of cure is closely related to the polymerization shrinkage of composite resins, and is an inherent feature. Inadequate depth of cure can lead to development of marginal gap and compromise the integrity of restorations [8].
Radiopacity is essential for evaluation of the proximity of the restoration to the pulp chamber, its marginal adaptation, detection of secondary caries, and assessing the overall quality of restoration [9]. Radiopacity of dental restorative materials, including composite resins, should be higher than that of an aluminum filter of the same thickness according to ISO standards governing dental materials [12].
Color match of dental composite resins with the adjacent tooth shade is imperative in restorative dentistry to achieve natural-looking esthetically pleasant results. Shade guides, such as VITA Classical, VITA 3D-Master, and VITA Easyshade spectrophotometer are used to ensure accurate color match [10]. VITA Easyshade spectrophotometer has shown high accuracy, making it a valuable tool for color measurement in dentistry [13].
The dental materials market is rapidly changing, with new products consistently being launched; therefore, there is an ongoing need to assess and compare the properties of new products [14].
This study aimed to compare different properties (flexural strength, depth of cure, radiopacity, water sorption, solubility, and shade) of four dental composite resins commercially available in the Iranian market.

MATERIALS AND METHODS

In this comparative study, the A2 shade of four different dental composite resins (Edge COM, Saremco, FGM, and Kulzer) was used (Table 1). All tests for all composite resins were conducted according to ISO 4049 standard [12]. Each composite resin was handled according to the manufacturers' instructions.

Table 1. Composite resins evaluated in this study

Product	Manufacturer	Filler Type	Shade
Edge COM	Hamerz Medical Co., Iran	Nano-hybrid	A2
Saremco els	Saremco Dental AG, Switzerland	Micro-hybrid	A2
FGM Llis	FGM Dental Group, Brazil	Micro-hybrid	A2
Charisma Smart	Kulzer GmbH, Germany	Micro-hybrid	A2

Flexural strength:

Flexural strength was measured according to ISO 4049 [12] for polymer-based restorations. Ten specimens were fabricated for each group using 2mm×2mm×25mm stainless-steel split molds. The molds were slightly overfilled with composite resins, and compressed between two glass slides. While ISO 4049:2019 recommends five specimens for assessment, we doubled the number of specimens to improve accuracy.

The specimens were subsequently polymerized using a LED curing unit (Blue Phase G2, Ivoclar Vivadent, Schaan, Liechtenstein) by the overlapping technique according to the manufacturer's instructions. They were then placed in a water bath at 37°C for 15 minutes. Next, they were removed from the bath, and flashings were gently removed with a 320-grit abrasive paper. Subsequently, the specimens were placed in a water bath at 37°C for 24 hours. Their flexural strength was then measured using an Instron universal testing machine (SMT-5; Santam, Tehran, Iran). A load cell (Bongshin Loadcell Co., LTD, Seoul, Korea) was used to apply 20KgF load to the middle of the samples at a crosshead speed of 0.5mm/minute. The flexural strength was then calculated using the following formula:

$$\sigma=3FL/2bh^2$$

where σ= flexural strength (MPa), F= load at the fracture point (N), L= length of the support span (mm), b= specimen width (mm), and h= specimen thickness (mm). The modulus of elasticity was calculated using the following formula:

$$E=FL^3/4bh^3d$$

where E=the modulus of elasticity (MPa), F=load at the fracture point (N), L=length of the support span (mm), b=specimen width (mm), h=specimen height (mm), and d=deflection at load point (mm).

Water sorption and solubility:

Water sorption and solubility were assessed according to ISO 4049 for polymer-based restorations [12]. Five disc-shaped specimens from each composite resin were fabricated using a mold with 15.0±0.1mm internal diameter and 1.0±0.1mm depth, and cured with a light curing unit (Blue Phase G2; Ivoclar-Vivadent, Schaan, Liechtenstein) with a light intensity of 1200mW/cm^2 from both sides. The specimens were removed from the mold and placed in a desiccator (37±1°C) for 24 hours. Next, they were placed in a desiccator at 23±1°C for 1 hour and then weighed with ±0.2mg accuracy. This cycle was repeated until a constant mass(m_1) was obtained, i.e., until the mass loss of each specimen was no more than 0.2mg in a 24-hour period. The specimens were immersed in distilled water at 37±1°C and remained there for 17 days. After immersion, the specimens were removed from the water, washed with distilled water, and wiped off until the surface was free from visible moisture. They were then waved in the air for 15 seconds and weighed 1 minute after removal from the water (m_2). The specimens were then reconditioned to constant mass in the desiccator, following the same protocol mentioned above. The constant mass was recorded as m_3. The diameter and thickness of the specimens were measured at the center and at four equally spaced points on the circumference. Their volume (V) was also calculated (mm^3). The difference in mass was analyzed by one-way ANOVA. The following formula was used to calculate water solubility:

$$W_{sl}=\frac{m_1-m_3}{v}$$

where m_1 is the conditioned mass in micrograms (μg) prior to immersion in water; m_3 is the mass of the reconditioned specimen in micrograms; V is the volume of the specimen in cubic millimeters (mm^3)

Water sorption (W_{sp}) was calculated in micrograms per cubic millimeter (μg/mm^3) for each of the five specimens using the following formula:

$$W_{sp}=\frac{m_2-m_3}{v}$$

where m_2 is the mass of the specimen in micrograms after immersion in water for 7 days;

m_3 is the mass of the reconditioned specimen in micrograms; V is the volume of the specimen in cubic millimeters.

Depth of cure:

According to ISO 4049 [12], three specimens of each type of composite resin were conventionally fabricated in a cylindrical stainless-steel mold with 6 mm depth and 4 mm diameter. Composite specimens were cured with a light-curing unit (Blue Phase G2; Ivoclar-Vivadent, Schaan, Liechtenstein) with the light intensity and duration recommended by the manufacturer. After light-curing, the cylindrical specimens were removed from the mold, and uncured composite resin was removed with a spatula. The absolute length of the cylindrical specimens of cured composite resin was then measured with a digital micrometer (Guilin Guanglu, DingJiang, China), with 0.1mm accuracy. The obtained values were divided by two according to the standard, and the depth of cure was recorded.

Radiopacity:

Three cured disc-shaped specimens measuring 1x15mm were fabricated from each composite resin according to ISO 4049 [12]. Radiographs with D-speed dental film (Kodak Insight; Carestream Dental, Rochester, NY, USA) were obtained from each specimen separately with an aluminum step wedge; the specimens were placed upright and in contact with the center of each radiographic film. Exposure was performed using a dental X-ray machine (ORIX 70; Ardet Dental & Medical Devices, Milan, Italy) with 60±10 kV tube potential, 8mA tube current, and 50Hz frequency. The focal spot was 0.8mm with a target-film distance of 300mm to 400mm. The exposure time was 0.3 seconds. After processing, the region of the film next to the specimen and aluminum step wedge had an optical density between 1.5 and 2. The individual optical densities/grey values were plotted for each aluminum step wedge against the thickness of each step. The optical density/grey value for each specimen with a thickness of Ts was calculated to determine the corresponding value of aluminum (Ta) according to the plot. The radiopacity (aluminum equivalent) value of a specimen with 1.0mm thickness was then calculated using the formula Ta/Ts.

Colorimetry:

Four disc-shaped specimens were fabricated from each composite resin with the size of the device head and 1.0±0.1mm thickness and cured (Blue Phase G2; Ivoclar-Vivadent, Schaan, Liechtenstein) with a light intensity of 1200mW/cm^2. Next, they underwent shade assessment in a Vita Easyshade spectrophotometer (VITA Easyshade, H. Rauter GmbH & Co. KG, Essen, Germany), The spectrophotometer was calibrated by placing a probe tip on the calibration port aperture before assessment of each specimen. The shade of the specimens was measured by holding the probe tip at a 90-degree angle relative to the surface of each specimen against a white background. According to the manufacturer's instructions, the measurement was accepted when two consecutive identical readings were generated for each area, and the results were recorded. Each specimen's color shade was compared against the Vita shade system, documenting the closest match.

Statistical analysis:

The data were analyzed using SPSS (SPSS Inc., Chicago, IL, USA). Comparisons were made by one-way ANOVA followed by the post-hoc Tukey's test. $P<0.05$ was considered statistically significant.

RESULTS

Flexural strength:

The flexural strength values for the tested composites ranged from 91.27±27.53MPa to 100.40±26.48MPa. As shown in Figure 1, there was no statistically significant difference in flexural strength among the four groups (P=0.839).

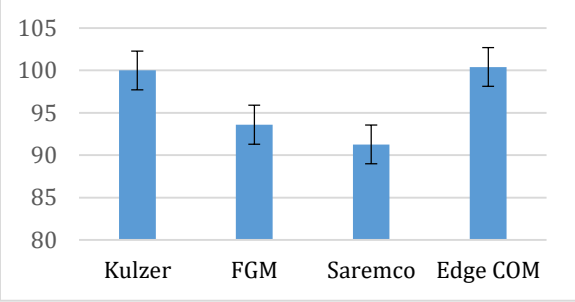

Fig. 1. Mean flexural strength (MPa) of the four groups

Radiopacity:
The radiopacity was 3.5mm for Kulzer, 3mm for FGM, 5mm for Saremco, and 4mm for Edge COM (Fig. 2).

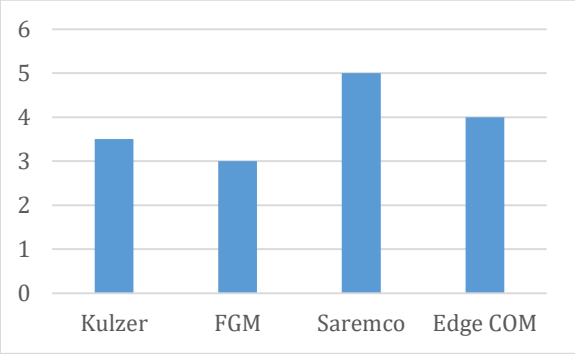

Fig. 2. Radiopacity of the four composite resins

Depth of cure:
Figure 3 presents the depth of cure of the four composite types. FGM (2.92±0.02mm) and Egde COM (2.17±0.04mm) showed the highest depth of cure. In contrast, Saremco (1.88±0.11mm) and Kulzer (1.78±0.09mm) exhibited the lowest depth of cure. A significant difference was found in the depth of cure among the four composite resins (P=0.000).

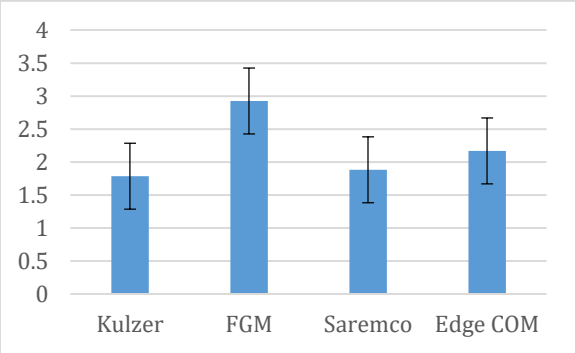

Fig. 3. Depth of cure (mm) of different composite resins

Water sorption and solubility:
There was a significant difference in water sorption among the four groups (P=0.000; Fig. 4). Kulzer exhibited a significantly higher water sorption than other groups (19.96±1.12µg/mm³), and the lowest water sorption was recorded in Edge Com (12.42±1.83µg/mm³).
A significant difference existed between FGM and Edge Com (P=0.009). FGM demonstrated no significant difference when compared to Saremco (P=0.248) and also, there was no statistically significant difference between Saremco and Edge COM (P=0.309).

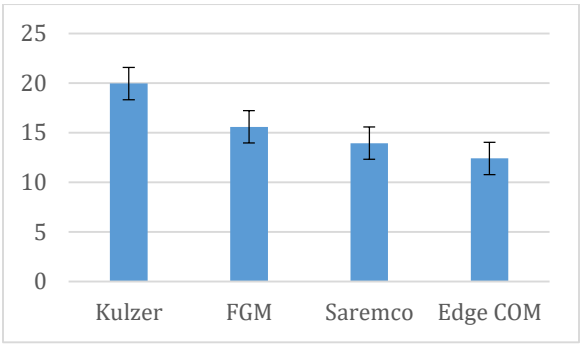

Fig. 4. Water sorption (µg/mm³) of different composite resins

In terms of solubility (Fig. 5), FGM and Edge Com demonstrated minimal solubility; whereas, Kulzer and Saremco did not. Although a significant difference was found among the four groups in this regard (P= 0.00), all experimental groups met the specifications outlined by ISO and were therefore suitable for clinical use.

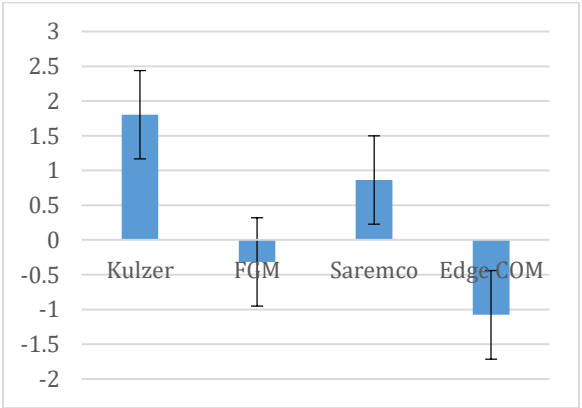

Fig. 5. Solubility (µg/mm³) of different composite resins

Colorimetry:
The results of colorimetry are presented in Table 2.

Table 2. Results of colorimetry

Group	Shade
Kulzer	A3.5
FGM	B3
Saremco	B3
Edge COM	B3

DISCUSSION

Currently, dental composite resins have several applications, and must possess acceptable mechanical properties. Therefore, this study aimed to assess the properties of four composite resins available in the Iranian market.

Composite resins contain various chemical compounds, including matrix components like monomers such as aromatic Bis-GMA, Bis-EMA, UDMA, carbonates, cyclic esters, acetals, and allyl sulfides. In addition to these essential components, most composite resins contain compounds with lower molecular weight such as HEMA, EGDMA, DEGDMA, and TEGDMA. Moreover, composite materials include initiators, activators, and additives, like light stabilizers required for the polymerization process [15]. Edge COM composite resin has different monomers in its matrix compared with other composite resins evaluated in this study, such as 1,4-butanediol dimethacrylate) and 12-diurethane dimethacrylate, which may affect its properties [16].

Dental clinicians should take into account the properties of composite resins before selection of a specific type for restorative procedures. Excellent mechanical properties such as high strength and fracture resistance, low wear, water sorption and solubility, and high radiopacity can maximize the longevity and clinical service of a composite restoration. Dental composites must have specific requirements for proper load transfer in the oral cavity [17]. Therefore, determination of chemical composition of a composite resin is preceded by thorough comprehensive examination of physicochemical characteristics of individual components and evaluation of their mechanical properties [18]. The mechanical properties of composite resins, including their flexural strength and modulus of elasticity, are predominantly influenced by the filler type, filler particle size, and amount of fillers loaded in the entire structure of the material [19].

The current study aimed to investigate some characteristics of four different types of composite resins. The results revealed significant differences in their radiopacity, depth of cure, water sorption, solubility, and shade.

High polymerization shrinkage stress and modulus of elasticity can potentially compromise the bond to tooth structure. Also, a lower modulus of elasticity does not always correlate with higher bond strength. However, it can promote a more uniform stress distribution at the tooth-restoration interface [20].

High flexural strength is essential to withstand masticatory forces without fracture in stress-bearing areas (Class I, II, and IV restorations). However, according to the present study, the difference in this regard did not reach statistical significance among the groups.

Radiopacity is a fundamental property for dental restorations. It facilitates the identification of defects such as fractures, voids, over-contouring, poor proximal contact, marginal gaps, and secondary caries [21,22]. The current results showed that Saremco had the highest radiopacity although all experimental groups demonstrated satisfactory radiopacity.

The depth of cure was the highest for FGM, and the lowest for Kulzer composite resin. Conventionally, to restore cavities with the incremental application technique, composite resins should be cured at a maximum thickness of 2 mm. The main advantages of the incremental technique include the optimal and deep curing of the material and reduction of polymerization shrinkage [23]. It should be noted that the depth of cure is affected by different factors, such as the light intensity, composition and transmission characteristics of the composite resin, depth of the cavity and restoration, duration of light exposure, and type of light source [24].

The solubility of resin-based composite materials has a critical importance in the field of restorative dentistry, as the inorganic ions incorporated as fillers in the composition of composite resins have the capacity to leach into the adjacent environment, ultimately leading to degradation of the restorative material. In the oral environment, polymer composites are commonly exposed to chemical agents found in the saliva, foods, and beverages, which may contribute to their chemical degradation [25]. As shown in the present study, solubility and water sorption of

all examined composite resins were within the standard range [12].

Shade assessment in the present study was conducted using the Easyshade Circuit Vita spectrophotometer. The four composite resins, regardless of their filler type and characteristics, had significant color shade differences, which could cause challenges in shade matching and illustrate inconsistencies in the desired color outcome in different applications. This finding heavily relies upon the specific pigments used by the manufacturer. Hence, future studies are required on the color stability of these pigments, both of which are critical factors when using composite resins in the clinical setting.

CONCLUSION

This study found no significant difference in flexural strength of the four tested composite resins. However, it revealed significant differences in their radiopacity, depth of cure, water sorption, solubility, and shade. These findings underscore the importance of future investigations aimed at discovering methods to enhance the clinical effectiveness of dental restorative materials, and improve their durability.

CONFLICT OF INTEREST STATEMENT

The authors declare that this research was funded by Hamerz Medical Company, and that Author 2 is an employee of this company. The funding source did not participate in the study design, data collection, data analysis, interpretation of data, or writing of the manuscript. The authors have no other conflicts of interest to disclose.

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