Journal of
Kerman University of Medical Sciences

https://jkmu.kmu.ac.ir
doi:10.34172/jkmu.4097
JKMU 2026;33:4097

Original Article

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Immediate and Delayed Repair Bond Strength of Amalgam -Composite Resin with Various Universal Adhesives and Surface Treatments

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Abstract
Introduction: Amalgam is a safe and effective option for direct restorations. For amalgam repairs, it is recommended to use composite resin along with an adhesive agent on pretreated amalgam surfaces. This study investigated the shear bond strength (SBS) between composite and both new and aged amalgam, using different universal adhesives on the pretreated alloy surfaces.
Methods: In this experimental in-vitro study, a total of 132 amalgam discs were prepared and divided into two main groups: fresh and aged (6 months water storage + 2000 thermal cycles). The aged amalgam specimens were randomly treated with sandblasting, pumice, or bur. Based on the type of adhesive used, each treated group was further divided into three subgroups: Single Bond Universal, Clearfil Universal Bond, and G-Premio Bond. Fresh amalgam groups were randomly divided into the Single Bond Universal and Clearfil Universal Bond subgroups. All samples were repaired with composite and then subjected to 2000 thermal cycles, followed by SBS assessment using a universal testing machine. One-way and two-way ANOVA, post-hoc Tukey's, Kruskal-Wallis, Mann-Whitney, and T-tests were used for statistical analysis.
Results: Single Bond Universal + pumice exhibited the highest SBS and Clearfil Universal Bond + bur was associated with the lowest SBS. Among the aged samples, groups prepared with bur had significantly lower SBS values compared to those treated with sandblasting or pumice. The SBS values for the Single Bond Universal groups were significantly greater than those for the Clearfil Universal Bond groups.
Conclusion: Pretreatment with pumice and sandblasting yielded the highest SBS between aged amalgam and composite. Furthermore, Single Bond Universal resulted in the best SBS values.
Keywords: Dental amalgam, Composite resins, Shear strength, Adhesives

Citation: Hoseinifar R, Shadman N, Mortazavi Lahijani E, Asadi L, Rezaee S. Immediate and delayed repair bond strength of amalgam -composite resin with various universal adhesives and surface treatments. Journal of Kerman University of Medical Sciences 2026;33:4097. doi:10.34172/jkmu.4097

Received: April 7, 2025, **Accepted**: July 19, 2025, **ePublished**: May 2, 2026

Introduction

Amalgam is a cheap, suitable, and effective material used in operative dentistry. Despite some disadvantages, including corrosion, a non-aesthetic appearance, and a lack of bonding to the tooth structure, it is still considered a good choice for dental treatments (1- 2).

In some cases, amalgam restorations must be replaced or repaired due to secondary caries, marginal defects, cusp fracture, loss of marginal adaptation, or improved esthetic appearance (3). The most common treatment for defective amalgam restorations is total restoration replacement. For older amalgam restorations with localized defects, however, repair with composite resin is an alternative treatment option (4). This technique preserves the tooth's structure and enables marginal sealing with minimal pulpal trauma (5).

The use of the amalgam-composite complex, in addition to its usage for repairing amalgam restorations, has also been popular in the deep Class II cavities, in which the deep section of the Class II cavity is filled with amalgam and the rest of the cavity is filled with composite (6).

The different methods for composite binding to amalgam are mechanical and chemical bonding mechanisms (applying multi-purpose adhesive systems or bonding systems with an alloy primer) (2, 7, 8).

To promote the bond between amalgam and

composite, some adhesives, such as universal adhesives, were introduced. In universal adhesives, there are phosphate monomers like 10-MDP (methacryloyloxy decyl dihydrogen phosphate), silane, and 4-META)4-methacryloxyethyl trimellitate anhydride(. There are numerous advantages to the existence of phosphate esters, such as 10-MDP, including the possibility of chemical bonding with metals, zirconia, and tooth substrates (9).

Old amalgams have changed over time due to the endurance of occlusal forces, corrosion, and phase shifts, so the bond between new and old amalgam and composite is probably different. The effect of the aging period of the existing amalgam on the bond strength has been previously evaluated. However, most of the studies used a short–term aging period, usually compared with new amalgam or during 21 days (10- 11).

Balkaya et al. (3) assessed how universal adhesives affect the micro-tensile bond strength in amalgam repairs, and the results showed that the application of an alloy primer before applying the universal adhesives significantly improved bond strength. Similarly, Kanzow et al. (12) studied the effects of universal adhesives on the SBS of composite to aged amalgam, finding that the use of universal adhesives and mechanical conditioning of the amalgam surfaces increases the SBS of amalgam-composite complex compared to conventional adhesives.

In addition, there is a lack of research on the immediate SBS of composite to amalgam using universal adhesives. Therefore, the present study aimed to evaluate the immediate and delayed SBS of composite to amalgam using different universal adhesives on the pretreated amalgam surfaces. The null hypothesis stated that there would be no difference in immediate and delayed SBS of composite to amalgam when using various universal adhesives on pretreated amalgam surfaces.

Methods

In this experimental study, a total of 132 discs of high-copper dental amalgam (SDI; GS - 80 - Australia) with a 6 mm diameter and a height of 3 mm were made in metal molds. The specimens were then randomly divided into two groups (fresh and aged). According to the design of the groups, $\frac{3}{12}$ of the groups were allocated to the fresh groups, and $\frac{9}{12}$ to the aged groups (n = 12) (Figure 1).

Fresh Groups

Five minutes after the placement of a new amalgam, fresh specimens were randomly assigned to one of the following groups. The adhesives used in the present study and their chemical compositions are mentioned in Table 1:

A1: A thin layer of Single Bond Universal adhesive was applied by a microbrush on the amalgam surface, gently air dried for 5 seconds, and then cured for 20 seconds using the LED (Light-Emitting Diode) curing device (Demetron, Kerr, USA). Afterward, composite Filtek Z250 XT (3M ESPE, St. Paul, MN, USA) was bonded using a plastic mold with a 4 mm diameter and 4 mm height. The composite was placed in two layers, and each layer was cured for 40 seconds. The light intensity of the LED was periodically checked with a radiometer (MONITEX, LM-100).

A2: A thin layer of Clearfil Universal adhesive was applied by a microbrush on the amalgam surface, gently air dried for 5 seconds, then cured for 20 s. The composite was subsequently bonded similarly to the A1 group.

A3: In this group, G-Premio Bond was applied, but all samples from this group failed at the early stages of composite bonding to amalgam, so they were excluded from the study.

Aged Groups

For the amalgam aging procedure, samples were stored in distilled water at 37°C for 6 months, and the water was replaced monthly (12). Afterward, they were thermocycled (Vafaei Industrial, Tehran, Iran) for 2,000 cycles at 5-55°C. The aged samples were randomly divided into three groups of 36 (B, C, and D). The surface conditioning methods of the specimens were as follows:

Sandblast method (B): The amalgam surface of all samples was sandblasted with 50 µm aluminum oxide particles using a sandblast machine (Microsandblaster, Dento, Denmark) from a 10 mm distance at a pressure of 2.5 bars for 4 seconds (13). The samples were washed, dried, and then randomly divided into three subgroups:

B 1: A thin layer of Single Bond Universal was applied on the amalgam surface by a microbrush, air dried, and then cured for 20 seconds.

B2: A thin layer of Clearfil Universal Bond was applied, and the remaining steps were completed as per the B1 group.

B3: A thin layer of G-Premio Bond was applied, and the remaining steps were completed as per the B1 group.

Surface roughness with a bur (C): Using a round diamond bur (Tizkavan, Tehran, Iran) and eight distinct motions, the amalgam's surfaces were roughened [13]. The samples were then divided into the following

Table 1. The adhesives used in this study and their composition

Material	Composition	Manufacture	PH
Single Bond Universal	10-MDP, Dimethacrylate resins, silane, initiator, filler, polyacrylic acid, polyalkenoic acid copolymer (Vitrebond copolymer), HEMA, ethanol, water, phosphoric acid	3M ESPE; St Paul, MN, USA	2.7
Clearfil Universal Bond	Bis-GMA, HEMA, MDP, hydrophilic aliphatic dimethacrylate, silica, silane coupling agent, ethanol, water	Kuraray; Okayama, Japan	2.3
G-Premio Bond	MDP, 4-MET, MEPS, methacrylate monomer, acetone, water, initiator, silica	GC; Tokyo, Japan	1.5
Filtek Z250 XT	Bis-GMA, Bis-EMA, UDMA, Zirconia and Silica Fillers	3M ESPE; St Paul, MN, USA	

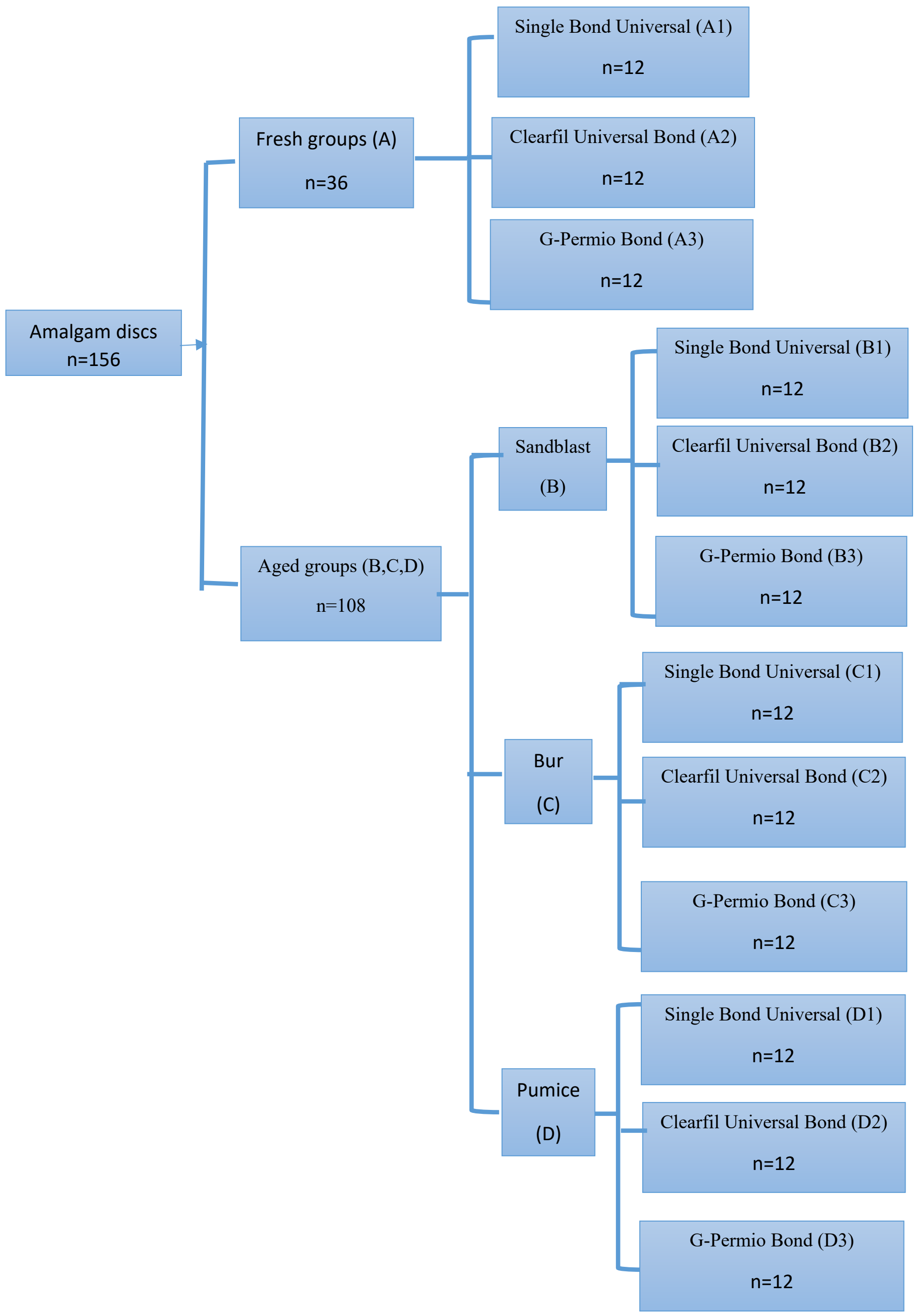

Figure 1. The sample grouping in the present study

subgroups:

C 1: Single Bond Universal was applied in the same manner as the B1 group.

C 2: Clearfil Universal Bond was applied in the same manner as the B2 group.

C 3: G–Premio Bond was applied in the same manner as the B3 group.

Cleaning with pumice (D): Pumice powder was used to clean the amalgam surfaces. Group D samples were also divided into the following subgroups:

D 1: Single Bond Universal was applied in the same manner as the B1 group.

D2: Clearfil Universal Bond was applied in the same manner as the B2 group.

D 3: G–Premio Bond was applied in the same manner as the B3 group.

Thereafter, the plastic mold (with a 4 mm diameter and height of 4 mm) was placed at the center of the dental amalgam surface, and the Filtek Z250 XT composite was inserted in two layers with a 2 mm thickness, and then cured for 40 seconds. To ensure complete curing, it was cured on all four sides for an additional 40 seconds.

After 24 hours of water storage, all specimens were thermocycled for 2000 cycles at 5-55°C. Finally, the SBS test was performed. To test the SBS, a universal testing machine (Testometric M350 – 10CT, England) was employed with a crosshead speed of 0.5 mm/min. A parallel stainless-steel blade with a 1 mm diameter was used at the interface between the amalgam and composite. The maximum force each specimen could endure before fracturing was recorded in Newtons, and the SBS was calculated in megapascals (MPa) by dividing this fracture load by the sample's cross-sectional area.

The obtained data were analyzed by the two-way ANOVA to determine the general effects of bonding types and preparation methods on the SBS of the composite to aged amalgam. Statistical comparisons of SBS values in different groups with various surface preparations or types of adhesives were performed with one-way ANOVA as well as post-hoc Tukey's, Kruskal-Wallis, and Mann-Whitney U tests. The difference in SBS values of composite to fresh amalgam under the conditions of curing and non-curing of bonding agents was investigated using a t-test. $P < 0.05$ was considered a statistically significant level.

Results

The effects of the surface conditioning method ($P < 0.001$) and bonding type ($P = 0.008$) on the SBS of composite to amalgam were significant.

The means and standard deviations of SBS values of composite to fresh amalgam are presented in Table 2. The results indicated that the SBS of Single Bond Universal was significantly higher than Clearfil Universal Bond ($P < 0.0001$).

The means SBS values of composite to aged amalgam in various groups are shown in Table 3. Comparing the different conditioning methods for Clearfil Universal Bond revealed that only the difference between the two subgroups of bur and pumice was statistically significant ($P = 0.001$). In the case of G-Premio Bond, no significant differences were found between different surface conditioning methods ($P = 0.18$). In the case of Single Bond Universal, the SBS of pumice and sandblast were significantly higher than bur ($P = 0.02$); however, no significant difference was observed between the sandblast and pumice conditioning subgroups ($P = 0.99$).

Regarding the effect of adhesive type, no significant differences were found between different adhesives in the pumice group ($P = 0.31$). In the bur and sandblast groups, however, the SBS values of Single Bond Universal were significantly greater than those of Clearfil Universal Bond ($P = 0.05$ and $P = 0.03$, respectively).

Discussion

Table 2. The means and standard deviations of SBS values of composite to new amalgam using various adhesives (in MPa)

Adhesive	Mean ± Standard deviation (MPa)
Single Bond Universal	8.96 ± 1.34
Clearfil Universal Bond	4.44 ± 0.95

This study investigated the effects of universal adhesives and surface conditioning methods on the immediate and delayed SBS of composite-amalgam. The results indicated that the highest SBS was achieved with pumice and sandblasting. Moreover, Single Bond Universal generated the highest SBS values; therefore, the null hypothesis was rejected.

The chemical bond to amalgam can be achieved using META or 10-MDP monomers; however, a reliable chemical bond between amalgam and composite is still debatable (3,7, 8). One of the most important factors affecting the bond strength of amalgam to composites is the surface condition (2, 13). Blum et al. (13) investigated the effects of surface conditioning on the tensile bond strength of amalgam repair and found that the maximum bond strength was achieved with sandblast surface conditioning + Panavia. They also reported that surface roughness has a significant effect on the bond strength of amalgam repair. Machado et al. (8) evaluated the immediate and delayed SBS of amalgam to composites and found that sandblasting produced the strongest bond. In addition, using the bur resulted in the lowest bond strength after one year, indicating that the surface created by the bur lacked the required roughness to provide adequate interlocking of the bonding agent.

Typically, sandblasting is used to clean the metal's surface and increase the micro retention and the surface area (8). These surface modifications are also produced by applying pumice powder. However, in a review of previous literature, no information regarding surface conditioning with pumice was found. Sandblasting with alumina provides a homogeneous surface by removing large surface defects, which ultimately leads to the formation of a stronger adhesive bond. In contrast, the use of bur creates stress concentration areas and large surface defects that prevent complete adhesive infiltration (2, 8, 10). In fact, sandblasting produces more regular and retentive surface topography than diamond bur (12). Moreover, it has been reported that the bonding agent can react with the oxide layer formed on the surface of amalgam, increasing bond strength (14). Khoroushi et al. (15) have reported that during the creation of surface roughness with a bur, some parts of the oxide layer were removed, so the bond strength was reduced.

When conditioning the surface with bur and sandblasting, the difference between Clearfil Universal Bond and Single Bond Universal was significant. However, when conditioning the surface with pumice, the difference

Table 3. The means and standard deviations of SBS values of composite to aged amalgam using different conditioning methods and various adhesives (in MPa), different superscript small or capital letters denote statistically significant differences in the columns and rows, respectively

Surface condition	Adhesive	Mean ± SD (MPa)	Surface condition	Adhesive	Mean ± SD (MPa)	Surface condition	Adhesive	Mean ± SD (MPa)
Bur	Single Bond Universal	$4.21 \pm 2.35^{a,A}$	Sandblast	Single Bond Universal	$9.31 \pm 4.88^{c,B}$	Pumice	Single Bond Universal	$9.23 \pm 3.89^{f,B}$
	G-Premio Bond	$4.17 \pm 0.74^{a,b,C}$		G-Premio Bond	$7.45 \pm 4.64^{c,d,C}$		G-Premio Bond	$6.86 \pm 1.99^{f,C}$
	Clearfil Universal Bond	$2.43 \pm 0.5^{b,D}$		Clearfil Universal Bond	$4.56 \pm 1.52^{d,D,E}$		Clearfil Universal Bond	$7.54 \pm 4.5^{f,E}$

between adhesive types was not significant, demonstrating the significance of the surface conditioning method.

Due to the new amalgam's high surface tension, which inhibits surface wetting, the composite was applied after a 5-minute delay for clinical simulation (16). In the present study, in the fresh group, all G-Premio Bond samples failed in the initial bonding steps to amalgam. The lack of an oxide layer combined with the high surface tension of the new amalgam may weaken the bond between the composite and amalgam (17). The failure of the G-Premio Bond samples may have been caused by the absence of HEMA in these adhesives, which prevents them from adequately wetting the amalgam surface. HEMA is a hydrophilic monomer that has been incorporated into the composition of a variety of universal adhesives. Due to its small molecular size, HEMA is an effective infiltrating agent that improves surface wetting and adhesive penetration (18).

In addition, the present study revealed that Single Bond Universal exhibited the highest repaired bond strength, while Clearfil Universal Bond exhibited the weakest one (both in the aging and fresh groups). Ercin et al. (19) evaluated the effect of adhesive systems on SBS between amalgam and composite and reported that the combination of universal adhesive and alloy primer has been reported to yield successful results in amalgam repair. In 2018, Balkaya et al. (3) assessed the effect of universal adhesives on the micro-SBS of amalgam to composite, with or without alloy primer, and reported no difference between the universal adhesives without the application of an alloy primer. This difference with the results of our study may be attributable to the type of test and amalgam employed. Hellak et al. (20) evaluated the bond strength of the orthodontic brackets to various substrates (including enamel, composite, and metal) with Single Bond Universal, iBond universal, and Transbond XT. The results showed that the highest SBS to the metal was obtained by Single Bond Universal. In 2019, Flury et al. (21) evaluated the immediate and delayed bond strengths of composite to six restorative materials. In this regard, the results showed that the silane existing in Single Bond Universal is effective on amalgam bonding as well as ceramic bonding. Additionally, in the preparation with Single Bond Universal, the failure of specimens was cohesive in amalgam, indicating a strong bond between Single Bond Universal and amalgam.

Various studies have reported the superior bond strength and performance of Single Bond Universal (12, 22,23), which is related to its low viscosity, better substrate wetting (12), the existence of non–inorganic nano fillers and polyalkenoic acid co-polymers, the high degree of conversion of filled hydrophobic resin, and its higher pH compared to the other adhesives (24,25).

Tsujimato et al. (25) showed that the fracture surfaces could be found in Clearfil Universal Bond and G-Premio Bond following fatigue bond strength testing. Moreover, Clearfil Universal Bond and G-Premio Bond had a greater number of bubbles, cracks, and gaps than other adhesives (Single Bond Universal and Clearfil SE Bond). It has been reported that the residual water after air drying in Clearfil Universal Bond and G-Premio Bond causes phase separation and the formation of blisters, which is known as the weak point for these adhesives and negatively affects their bond strength. Additionally, it has been demonstrated that the silane component in Clearfil Universal Bond forms a structural network that traps ethanol and water, which leads to more difficult solvent evaporation (23).

In 2019, Papadogiannis et al. (26) evaluated the performance of 6 universal adhesives containing 10–MDP. The difference of Single Bond Universal with Clearfil Universal Bond and G-Premio Bond was significant in terms of oxygen inhibition layer thickness. It has been reported that an oxygen inhibition layer is required for a proper bond before adding additional layers of the composite. This layer enhances adhesion between the substrate and the subsequent composite layer. The higher thickness of the oxygen inhibition layer in Single Bond universal could account for its superior strength (27). Single Bond Universal and Clearfil Universal Bond contain 10-MDP as the functional monomer, whereas G-Premio Bond contains 4-META, 10-MDP, and MEPS monomers. According to reports, the purity and concentration of MDP in adhesives affect their chemical bonding potential; therefore, the difference between the purity and concentration of 10–MDP may also affect bond strength. Tsujimato et al. (25) also reported that even though the manufacturers of Clearfil Universal use higher-purity MDP than other manufacturers, the fatigue strength of Clearfil Universal Bond was significantly less than that of Single Bond Universal.

Choo *et al.* (28) reported that an Alloy primer was bound to the non–precious alloys by the monomer of 10-MDP. 10-MDP creates a strong chemical bond with the oxide layer present on the alloy surface, resulting in a durable connection between the resin composite and the alloy.

The results of the present study showed that the mean of SBS calculated is less than the acceptable threshold under in vivo conditions (10 MPa). Thus, it seems that for clinical usage, in addition to bonding, it is necessary to obtain retention by mechanical methods (29). Hadavi et al. (30) reported that the bond strength of repaired amalgam is less than half that of an intact amalgam restoration; therefore, if the repaired amalgam is in occlusion, additional retention through the groove, undercut, etc., is necessary to increase the restoration's longevity. In addition, the oxide layer on the metal's surface serves as a bonding agent between resin containing 4-META and 10-MDP and base metal alloys such as amalgam. Since the oxide layer of an old amalgam in the mouth is thicker than that in vitro, greater bond strength is achieved than in vitro (31).

It must also be considered that the aging of the bond interface with thermocycling is ideal for materials with the same thermal expansion coefficient. Whereas the coefficient of thermal expansion of the composite is three times that of amalgam. As a result, amalgam has less tendency to increase in volume as the temperature rises, which causes a displacement between the materials at the interface and may separate the newly formed bond between the adhesive and metal. However, the variation of temperature within the mouth is not comparable to thermocycle, as it is highly unlikely that teeth or restorations would remain at 55°C for 20 seconds. Notably, the movement of food and drink in the mouth, as well as the effects of saliva, tongue, and mucus, play a significant role in the regulation of temperature equilibrium (32).

Conclusion

Considering the limitations of this study, it can be concluded that the method of surface conditioning and the type of universal adhesive had significant effects on the SBS of the composite to amalgam. The SBS of aged amalgam to composite was greatest when the amalgam surface was treated with pumice powder or sandblasting. Furthermore, specimens utilizing Single Bond Universal exhibited the highest bond strength values.

Acknowledgements

The authors would like to thank the Kerman University of Medical Sciences for their support and cooperation throughout the research.

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Competing Interests

The authors declared no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available upon request from the corresponding author. The data are not publicly available because of privacy or ethical restrictions.

Ethical Approval

The Research and Ethics Committee of Kerman University of Medical Sciences approved this study (Ethical code: IR.KMU.REC.1398).

Funding

This study was financially supported by Kerman University of Medical Sciences.

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