

Shear bond strength evaluation of resin composite to resin-modified glass-ionomer cement using three different resin adhesives vs. glass-ionomer based adhesive

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Abstract

Background: The clinical success of sandwich technique depends on the strength of resin-modified glass ionomer cement (RMGIC) bonding to both dentin and resin composite. Therefore, the shear bond strength (SBS) of resin composite bonded to RMGIC utilizing different resin adhesives versus a GIC-based adhesive was compared. **Materials and methods:** In this *in vitro* study, 84 holes (5×2 mm) were prepared in acrylic blocks, randomly divided into seven groups (n=12) and filled with RMGIC (Light-Cured Universal Restorative, GC). In the Group I; no adhesive was applied on the RMGIC. In the Group II, non-etched and Group III was etched with phosphoric acid. In groups II and III, after rinsing, etch-and-rinse adhesive (OptiBond Solo Plus); in the Group IV; a two-step self-etch adhesive (OptiBond XTR) and in Group V; a one-step self-etch (OptiBond All-in-One) were applied on the cement surfaces. Group VI; a GIC-based adhesive (Fuji Bond LC) was painted over the cement surface and cured. Group VII; the GIC-based adhesive was brushed over RMGIC followed by the placement of resin composite and co-cured. Afterward; resin composite (Point 4) cylinders were placed on the treated cement surfaces. The specimens were placed in 100% humidity at 37 ± 1°C and thermo cycled. The shear bond test was performed at a cross-head speed of 1 mm/min and calculated in MPa; the specimens were examined to determine mode of failure. The results were analyzed using one-way ANOVA and Tukey test. **Results:** The maximum (24.62±3.70 MPa) and minimum (18.15±3.38

MPa) SBS mean values were recorded for OptiBond XTR adhesive and the control group, respectively. The pairwise comparisons showed no significant differences between the groups that bonded with different adhesives. The adhesive failure was the most common failure mode observed. **Conclusion:** This study suggests that GIC-based adhesive could be applied over RMGIC as co-cure technique for sandwich restorations in lieu of employing the resin adhesives

Key words: glass-ionomer based adhesive, dental adhesives, resin-modified glass-ionomer cement, resin composite, shear bond strength.

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Introduction

Resin composites have gained considerable popularity in the restorative dentistry as a direct restorative material for both anterior and posterior teeth. However, they have several disadvantages, such as polymerization shrinkage, potential failure of adhesion leading to secondary caries, and a relatively high coefficient of thermal expansion (1, 2). Polymerization shrinkage, mechanical and thermal stresses create contraction, internal stresses and deformation in the

resin composite restoration and surrounding tooth structure. These stresses may result in de-bonding from the cavity walls or cohesive fractures of the restoration material or tooth structure (3, 4).

In contrast, glass ionomer cements (GICs), which were developed by Wilson and Kent (1971), provide long-term fluoride release, thereby decreasing the possibility of recurrent caries (5). They also have physicochemical adhesion to tooth structure, coefficient of thermal expansion similar to that of tooth structure, biocompatibility and low cytotoxicity (6,7). The reaction of GICs displays a similar behavior to dentin under thermal stimuli (8). Because of these favorable properties, it can be placed on dentin prior to the application of a resin composite. This restorative method is commonly referred to as the sandwich technique (laminar or bilayered technique) (9, 10). The rationale behind this technique is to combine the advantages of both GICs and resin composites to form one restoration in order to enhance the clinical serviceability (10-12).

The GIC considerably reduces the volume of resin composite used, subsequently minimizing the detrimental effects of polymerization shrinkage (13, 14). It also prevents the resin composite from bonding with the dentin, thereby restricting the adhesion of the resin composite to the enamel. The reduction in bonded composite surfaces decreases the configuration factor (C-factor) of the cavity (15). In addition, it acts as a stress absorbing interfacial layer between the shrinking resin composite and dentin (16). In contrast to resin bonding, the adhesion of glass-ionomer to tooth structure is less technique sensitive and its quality increases with time (8).

Two types of GICs, i.e., conventional or resin-modified GIC (RMGIC), can be used as base in the sandwich technique, although they show differences in the adhesion mechanism, setting reaction, and sensitivity to the moisture of the materials. The success of this technique depends on the bond strength of the GIC to both dentin and resin composite (17). Some studies have demonstrated that acid-etching of the cement surface enhances the bond strength due to increases in mechanical retention (18, 19); however, this has not been confirmed by other studies (20, 21).

Clinically, the etching procedure requires a two-to-seven minute waiting period for the initial setting of the GIC to be completed (18, 22). High technique sensitivity, low cohesive strength and slow setting reaction of conventional GIC, and also minimal of chemical bonding between it and resin have led to the introduction of resin-modified GIC (RMGIC) (18, 23). The RMGIC has been developed to improve tensile and fracture strengths, working time combined with a rapid set, chemical solubility, and polishing appearance (6,

12, 24, 25). The setting reaction of RMGIC follows two distinct mechanisms: resin polymerization and acid-base reaction. RMGIC also demonstrates significantly improved cohesive strength, lower modulus of elasticity and better bond strength to tooth than the conventional GIC. Nonetheless, these cements usually have somewhat inferior esthetic and abrasion resistance when compared to resin composite, thus limiting their use in high stress-bearing areas (6, 11, 25, 26).

The recent development of adhesive systems, including self-etch primers might overcome this disadvantage, as there is no need to rinse the GIC prior to application of the bonding agent (22, 27). In the last few years much attention has been focused on the development of adhesive systems. Therefore, light cured glass ionomer based adhesive (Fuji Bond LC) was introduced. It is the first bonding agent based on a dynamic glass ionomer adhesion; it is essentially a diluted version of the restorative RMGIC Fuji II LC and suitable for bonding all resin composites (28).

However, to date, only a few studies have evaluated the efficacy of different resin-based and glass-based adhesives on RMGIC surface before the application of the resin composite in sandwich technique and there is no consensus on the type of surface treatment modalities over RMGIC. Hence, the present study was conducted to compare the shear bond strength (SBS) of resin composite to RMGIC utilizing three different generations resin adhesives versus a GC Fuji bond LC. Moreover, this modified protocol would not only prevent moisture contamination or desiccation of the underlying GIC, but it could also save precious chair-side clinical time. The null hypotheses were that adhesive type and the acid-etching procedure have no effect on the SBS of the resin composite to RMGIC.

Materials and Methods

In this *in vitro* study, 21 acrylic resin blocks (Acropars, Tehran, Iran) were prepared using a cubical aluminum mould, 100×25×20 mm in dimension, which was polished with carbide polishing paper. Before applying the molding acrylic resin into aluminum mould, four water soluble tablets were located on glass slab in specified place and after the completion of the setting of acrylic resin, they were washed out. Thus, four holes of 5-mm in diameter and 2-mm in depth were made in each block. The holes were finally filled with RMGIC (Light-Cured Universal Restorative, GC Corporation, Tokyo, Japan) by mixing it according to the manufacturer's instructions. Every effort was made to ensure that the exposed cement surface has been flushed with the surrounding specimen container. A light-emitting diode (LED) light curing unit (Demetron A.2, Kerr Italia, S.p.A., Scafati, Italy) was used to

polymerize the cement for 20 s at 1000 mW/cm² light intensity. The tip of light cure unit was placed 1-mm above the surface of the cement. The RMGIC surface was not finished to a glass-smooth surface to mimic the clinical scenario.

Prior to cement surface treatment, the bonding area was demarcated with an adhesive tape with a punch hole of 3.5-mm in diameter. Thereafter, the 84 specimens were randomly divided into seven treatment groups (n=12) according to the surface treatment performed :

Group 1: No treatment was applied on the cement surface (control group).

Group 2: The cement surface was not etched .

Group 3: The cement surface was etched with a 37.5% phosphoric acid gel (Gel Etchant, Kerr Italia S.p.A., Salerno, Italy) for 15 s.

All specimens in group II and III were rinsed thoroughly for 20s and gently air dried for 5s to remove excess moisture without desiccation of cement. Then, OptiBond Solo Plus (Kerr Italia S.p.A., Salerno, Italy) was applied to the bond area marked on the cement surfaces and cured according to manufacturer's instructions (Table 1).

Group 4: OptiBond XTR self-etch adhesive (Kerr Italia S.p.A., Salerno, Italy) was applied on the cement surface and cured according to the manufacturer's instructions (Table 1).

Group 5: OptiBond All-in-One self-etch adhesive (Kerr Italia S.p.A., Salerno, Italy) was treated on the cement surface and cured according to the manufacturer's instructions (Table 1).

Group 6: Fuji Bond LC adhesive (GC Corporation, Tokyo, Japan) was treated on the cement surface and light cured according to the manufacturer's instructions before adding the resin composite (Table 1)

Group 7: Same as group 6, but the Fuji Bond LC bonding agent (GC Corporation, Tokyo, Japan) was not cured over RMGIC, the GIC-based adhesive and resin composite was co-cured for 20 s. On the other hand, immediately afterward resin composite was placed on the treated cement surface, two materials light cured together for 20s.

Immediately following these procedures, a transparent plastic tube with 3-mm inner diameter and 2-mm height was filled with a microhybrid resin composite (Point 4, Kerr Italia S.p.A., Salerno, Italy, A2 Body Shade) in a one-layer increment technique and centered over the RMGIC surface in the template. Subsequently, any excess uncured resin composite was carefully removed from periphery of tubes with an explorer and cured resin composite then with sharp surgical blade. The tube was exposed to the curing light for 20s vertically and for 20s circumferentially (10s from each side) to ensure complete polymerization. For

all the specimens, the tip of light cure unit was placed 1-mm away from the surface of the restoration materials. After the composite buildup, the plastic tube was carefully removed with scalpel blade, leaving the resin composite rod on the treated adhesive surface of the RMGIC block. All the experimental procedures were conducted at room temperature, and the manufacturers' instructions were precisely followed for the all materials.

After the preparation, each of the specimens were kept moist to avoid any dehydration changes and cracking during the laboratory procedures which might have affected the bond strength. Subsequently, all the specimens were stored in an incubator with 100% humidity at $37 \pm 1^\circ\text{C}$ for one month and then thermocycled (Vafaei Industrial Factory, Tehran, Iran) 1500 cycles between 5°C to 55°C to simulate clinical situation with a dwell time of 1-minute in each bath and transfer time 5s.

The shear bond test was performed using a Universal Testing Machine (Zwick GmbH & Co, Ulm, Germany) at a cross-head speed of 1 mm/min until fracture occurred. The shear load was applied as close as possible to the adhesive interface between the resin composite and RMGIC utilizing a blunt knife-edged apparatus and the maximum load required to debonding the two materials was recorded for each specimen (Fig 1). The SBS was calculated in Mega Pascal (MPa), which is derived by dividing the maximum load force (N) at the time of fracture by the bond area (πr^2).

After mechanical failure, the fracture modes in all the specimens were evaluated by one observer (MS) under an optical microscope (Olympus Optical Co., Tokyo, Japan) at 20x magnification. Accordingly, the specimens were classified into three groups: Fractures were called "adhesive failure" when the resin composite was removed from the glass-ionomer surfaces without residual debris, "cohesive failure" when fracture occurred inside the restorative materials, and "mixed failure" when combination of both cohesive and adhesive failures were observed.

Data were analyzed using SPSS-18 software (SPSS Inc, Chicago, IL, U.S.A.). Descriptive statistics including the mean, standard deviation, maximum and minimum of the SBS were accounted for each group. Normal distribution of the data and homogeneity of variances were confirmed using Kolmogorov-Smirnov and Levene's tests, respectively ($p > 0.05$). Therefore, one-way analysis of variances (ANOVA) was used for the comparison among all groups and post-hoc Tukey test for pairwise comparisons of the groups. The failure mode frequencies were analyzed using the fisher's exact test. P value < 0.05 was considered statistically significant.

Table 1. Various restorative materials used in the study and mode of their applications according to the manufacturers' instructions.

Material	Manufactures' Instructions
OptiBond Solo Plus (two-step etch-and-rinse)	<ol style="list-style-type: none"> 1. The cement surface was not etched in group 2. 2. The cement surface was etched for 15s in group 3. 3. Rinse thoroughly ensuring that all acid is removed. 4. Dry lightly (do not desiccate). 5. Apply the adhesive and rub for 15s. 6. Air thin for 3s. 7. Light cure for 10s. 8. Place composite and light cure for 20s.
OptiBond XTR (two-step self-etch)	<ol style="list-style-type: none"> 1. Apply the self-etch primer using a microbrush with a scrubbing motion for 20s. 2. Air thinning for 5s using medium pressure. 3. Shake the adhesive briefly. 4. Apply the adhesive using a light brushing motion for 15s. 4. Air thinning using medium to strong pressure for at least 5s. 5. Light cure for 10s. 6. Place composite and light cure for 20s.
OptiBond All-in-One (one-step self-etch)	<ol style="list-style-type: none"> 1. Shake the bottle for 10s. 2. Apply the adhesive and rub for 20s. 3. Apply a second layer of adhesive in the same fashion. 4. Air thinning lightly for 5s. 5. Light cure for 10s. 6. Place composite and light cure for 20s.
Fuji Bond LC (GC-based adhesive)	<ol style="list-style-type: none"> 1. Dispensing powder and liquid with one level spoonful of powder and two drops of liquid. 2. Mixing powder and liquid for 10s. 3. Apply Fuji Bond LC in a thin layer over RMGIC surface using a disposable brush. 4. Light cure for 20s. 5. Place composite and light cure for 20s.

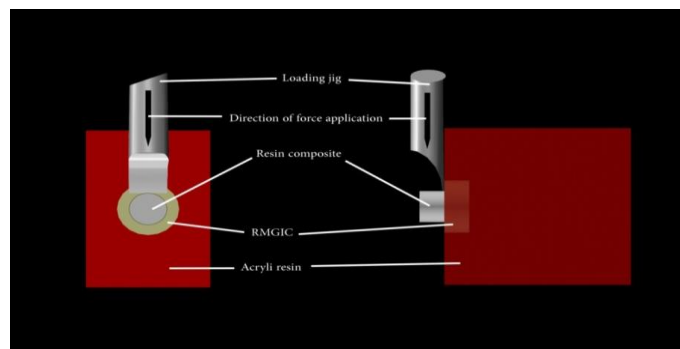


Fig 1: Schematic diagram of the specimen for shear bond strength test

Results

The descriptive statistics on the mean SBS (MPa) of resin composite to RMGIC utilizing different adhesives at the fracture for each group are tabulated in Table 2. The one-way ANOVA revealed that the SBS was significantly different among the experimental groups ($P < 0.001$). The maximum SBS mean values (24.62 ± 3.70 MPa) were recorded for Group 4, where

OptiBond XTR self-etch adhesive (Kerr Italia S.p.A.) was applied on the RMGIC surface; on the other hand, the control group which had not receive any pretreatment displayed minimum SBS values (18.15 ± 3.38 MPa).

Comparisons of the groups using post-hoc Tukey test indicated that the differences in SBS between

groups 3 (OptiBond Solo Plus without acid-etching) and 6 (Curing Fuji Bond LC before applying resin composite) with the control group was not significant ($p>0.05$); however, in the other groups (II, IV, V and VII) had a significantly higher SBS compared to the control group ($p< 0.05$). Based on the results of the present study no significant differences were found between groups that bonded with different adhesives ($p>0.05$) (Table 3).

Microscopic examination of interfacial debonding revealed that the majority of failure modes were adhesive failure followed by mixed failure, except for the group III that cement surface was etched with phosphoric acid that showed 58.3% mixed failure (Table 4). The fisher's exact test showed that significant differences were not observed in frequency of the failure mode across the test groups ($p=0.207$).

Table 2. Mean values, standard deviation (SD), minimum and maximum of the SBS (MPa) of resin composite bonded to the RMGIC by using different adhesives (n=12).

Group	Mean±SD	Min- Max
1	18.15±3.38	13.43-23.83
2	23.29±3.76	18.32-28.36
3	20.91±3.39	15.16-25.22
4	24.62±3.70	19.63-30.91
5	22.81±3.10	17.45-27.06
6	21.53±2.85	16.93-25.56
7	23.11±3.40	18.73-28.55

Table 3. Pairwise comparison between the groups and its statistical significance using post-hoc Tukey test.

Group	G1	G2	G3	G4	G5	G6
G2	0.007	-	-	-	-	-
G3	0.426	0.601	-	-	-	-
G4	0.001	0.960	0.115	-	-	-
G5	0.019	1.000	0.809	0.864	-	-
G6	0.193	0.862	0.999	0.289	0.967	-
G7	0.010	1.000	0.684	0.929	1.000	0.912

Table 4. Failure modes of the study groups (n=12)

Group	Mode of Failure		
	Adhesive n (%)	Cohesive n (%)	Mixed n (%)
1	9 (75)	0	3 (25)
2	7 (58.3)	2 (16.7)	3 (25)
3	4 (33.3)	1 (8.3)	7 (58.3)
4	7 (58.3)	1 (8.3)	4 (33.3)
5	7 (58.3)	2 (16.7)	3 (25)
6	11 (91.7)	0	1 (8.3)

Discussion

The sandwich technique using RMGIC layered with resin composite has been recommended as a viable restoration alternative in large and deep cavities to enhance adhesion and to limit micro leakage (10, 11). In this study, RMGIC (GC Corporation) was used as an under filling material, because most of the previous studies have shown that RMGICs have significantly higher bond strength and are more effective in preventing dye penetration than the conventional GICs, when bonded to tooth structures

and resin composites (8, 10, 12, 13, 29, 30). SEM photographs of a study indicated good interlocking adhesion between the RMGIC and resin composite (29). A strong bond between RMGIC and the resin composite is an important factor for the quality of sandwich restoration. Hence, the HEMA component forms higher chemical bond strength to the bonding agent/resin composite system (11, 23, 31). In this work, significant difference was observed among the study groups ($P<0.001$); the pairwise comparisons of the experimental groups showed that there was no significant differences in the SBS of the resin composite to RMGIC between the tested adhesives and acid-etching procedure. On the other hand, the type of adhesives and the acid-etching procedure is not effective on the SBS of the resin composite to RMGIC. Hence, the null hypothesis was accepted.

Although application of self-etch adhesive (OptiBond XTR) produced better SBS between resin composite to RMGIC, it showed no significant difference compared to the other groups ($p<0.05$) except for the control group ($P<0.001$). OptiBond XTR is a two-component self-etch, filled, fluoride releasing, light-cure bonding agent designed for the universal bonding of direct and indirect restorations. On the contrary, some studies have concluded that the

application of the self-etch adhesives between RMGICs and the resin composites increase the SBS significantly as compared to the total-etch type adhesives (32, 33). In the surface of set RMGIC there is a superficial catalyst rich with air-inhibited layer, which can copolymerize with resin composite. Also, the residual unreacted methacrylate groups on the polyacid chain within the polymerized RMGIC may form strong covalent chemical bonds with the resin bonding agent (33, 34).

The results of this study showed that application of OptiBond All-in-One improved the SBS more than the control group did ($p=0.019$); but no significant difference was observed with the other groups ($p>0.05$). Application of bonding agents improves the wettability of RMGICs to adhere to resin composite, thus promoting a strong shear bond between RMGIC and the resin composite (32). This is due to a similar chemistry between RMGIC and the resin composite, which allows the strong bonding of RMGIC to resin composite (11, 23, 33). Both RMGIC and the resin composite are cured by a free radical initiator system, which provides a potential for the chemical bonding between these two materials.

In Group III and II, the OptiBond Solo Plus was placed over RMGIC surface with and without acid-etching, respectively. According to the results of this study, the mean of SBS of resin composite to non-etched RMGIC was higher than the control group, without any pretreatment ($p=0.007$), however, there was no significant difference between acid etched and non-etched treatment methods ($p=0.426$). A study revealed that the application of self-etch adhesive systems (AdheSE and AdheSE One F) can improve the SBS between the composite and RMGIC (35). The acid-etching may remove the air-inhibited layer on the surface of RMGIC and decrease the potential for chemical bonding to the adhesive system (31). Consistent with the present study results, there is a consensus that etching of RMGIC is not required prior to the bonding of the resin composite (13, 12, 21, 36); It seems that RMGICs are not influenced by acid-etching due to their high resin content (12, 24). In another study, it was reported that acid-etching of one type of RMGIC can result in lower SBS as it may partially remove the HEMA and decrease the availability of oxygen-inhibited functional methacrylate groups which contribute to the adhesion to resin composite (31). However, this fact does not necessarily apply to all RMGICs (37). Also, it was indicated that acid-etching of RMGIC surfaces not only increases clinical application time, but also may enhance technique sensitivity but did not improve the sealing ability of sandwich restorations (13, 22, 27).

Specimen preparations in the majority of the studies done with the cement were allowed to set against a smooth surface, such as glass or mylar (9, 20). But in this study, the RMGIC surfaces were left uninstrumented in all the groups, and not finished to a glass-smooth surface, because resin composite will not bond to a glass smooth glass ionomer surface (9). In addition, glazed cement surfaces cannot be reproduced in clinical conditions (22). In this study, combinations of the various materials were selected based on the same manufacturer's products (Kerr Italia S.p.A.). The only exception was Fuji Bond LC bonding agent (GC Corporation).

Fuji Bond LC is a resin-diluted version of the restorative RMGIC. The bonding mechanism of Fuji Bond LC is based on both a micromechanical interlocking and a chemical interaction with the dentin and produce a hybrid layer with a thickness of about 0.5-1 μm . It is the only commercially available RMGIC adhesive, which can be used to bond resin composites to enamel and dentin. Fuji Bond LC also contains Hydroxy-Ethyl Methacrylate in its composition, similar to the fifth-generation and sixth-generation bonding agents, which provide for good wetting (38-40)

In the group VI, the GIC-based adhesive (Fuji Bond LC) was placed immediately over set RMGIC and then cured, while in the Group VII, it was placed and co-cured with resin composite. Although the SBS values of the latter group were superior to the group VI, there was no significant difference between them. The co-curing technique can improve the SBS between the composite and RMGI (35) and eliminates several steps in placing a sandwich restoration; notwithstanding this technique had not significant difference with other experimental groups but it produces a significantly strong SBS between the resin composite and RMGIC compared to the control group ($p=0.01$). The superior performance of GIC based adhesive could be attributed to chemical bonding between the resin composite and RMGIC (22). Mixing the RMGIC at twice the manufacturer's recommended liquid powder ratio creates a creamy consistency, similar to luting cement that is easily brushed over set RMGIC with a micro-brush. The clinical significance of this observation would be the recommendation to use co-curing technique rather than light curing separately when GIC based adhesive is used for bonding resin composite to RMGIC in sandwich restoration.

Analysis of the debonded surfaces revealed adhesive failure along the RMGIC/resin composite interface in 64.3 percent of specimens, cohesive failure in 7.1 percent, and mixed failure in the remaining 28.6 percent. According to the findings, no cohesive failure

was observed in the RMGIC and only a few samples had cohesive failure in the resin composite. It may be inferred that in spite of improvement of the bond strength between the resin composite and RMGIC surface through various surface treatments, this bond was not still strong enough and was less resistant than the cohesive resin composite and RMGIC. Also, it seems that difference in size of RMGIC surface (5-mm) and resin composite rod (3-mm) could be a reason for the higher number of adhesive failure in this study. Furthermore, the most mixed failure was observed only when the resin composite was bonded to the cement surface with OptiBond Solo Plus and acid-etching (group III). The correlation between SBS values and failure mode is controversial in the literatures (41-43).

Generally, this study provides practitioners with two alternative techniques for sandwich restorations instead of employing the traditional total-etch system. The application of one-step self-etch or two-step self-etch adhesive systems over RMGICs is recommended. Clinically, these systems would be useful, as it not only does away with the etch and rinse procedure, but it also saves valuable clinical time. The second alternative technique is employing a glass-ionomer adhesive system over RMGICs as co-cure technique. With this technique, clinicians can take advantage of glass-ionomer cement i.e., adhesion to tooth structure, increase the quality of time, fluoride releasing property, pH buffer capacity, it can turn out to the more reliable restorative material in minimal invasive dentistry, and forgiving, bio-active and intelligent materials (8).

Conclusion

This *in vitro* study showed that there was no significant difference in SBS of resin composite to RMGIC utilizing different generations of resin adhesives and Fuji Bond LC; therefore, the results suggest that glass-ionomer adhesive system (Fuji Bond LC) could be applied over RMGIC as co-cure technique for sandwich restorations in lieu of employing the resin adhesive systems. With this technique, clinicians can take advantage of RMGIC. Further studies are needed to understand the mechanism of adhesion between the resin composite and RMGIC bonded with different adhesive systems.

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Conflict of Interest

There is no conflict of interests in this research. The research was funded by Rafsanjan University of edical Sciences.

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