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EVALUATION OF MICRO-LEAKAGE OF INDIRECT RESIN COMPOSITE INLAY CEMENTED TO DENTIN WITH DIFFERENT RESIN CEMENT STRATEGIES

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A R T I C L E I N F O

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A B S T R A C T

***Objective*:** To evaluate micro-leakage of indirect resin composite inlay lutedto dentin after specimen's storage in distilled water and lactic acid.

***Materials and Methods*:** Standardized MOD Class II cavities were prepared in 48 intact human molars and restored with SR Nexco resin composite inlay restorations. The specimens were assigned into three sets (n=16) according to the luting resin cement used; group I: (etch-and-rinse (Variolink N), group II: self-etch (Panavia F2.0) and group III: self-adhesive (RelyXUnicem)). Each set was subdivided into two equal subsets (n=8) relative to the storage media either distilled wateror lactic acid. Half of the specimens of each subset were stored in each storage medium for 24h while the other half was stored for 168h (7 days).

***Results*:** There was significant difference ($p<0.05$) between all types of resin cement tested. Variolink N resin cement revealed the highest scores of dye pentration, followed by RelyXUnicem, then Panavia F2.0. In general, results showed, there was high significant difference ($p<0.05$) between both of storage media.

***Conclusion*:**Micro-leakage showed good results with SE luting resin cement strategy.association implying the importance of ruling out NTM in bronchiectasis patients.

INTRODUCTION

Due to the increased demands for esthetic dentistry and conservation of tooth structure, resin based composites are used as direct posterior restorations. Which have grown in popularity in combination with adhesive systems as the treatment of choice where esthetic is a primary concern.1,2 Sometimes clinicians were confused when dealing with restoration of posterior teeth, especially during rehabilitation of severely damaged or fractured ones. Where they have to select which material and technique is more adequate for restoration. Direct composite restoration may be inadequate in the long term due to insufficient wear resistance, imperfect proximal or occlusal morphology and deficient mechanical properties.3

Other resin based composite materials and curing systems have been introduced, one category is the indirect composite restorations such as inlays, which defined as single-tooth restoration that compensates a proximal-occlusal lesion with minimal or moderate extensions. These alternative esthetic restorations are developed to overcome the limitations of direct posterior composite and ceramic inlays. Laboratory-processed resin composite inlays are characterized by superior mechanical properties, high wear resistance, excellent esthetics, low polymerization shrinkage, better proximal contact and occlusal morphology in comparison to direct restorations. Also, they are lower in cost, lower wear for opposing teeth and easier in repair than ceramic inlays. But, they require two appointments and provisional restoration between visits.4,5

A brand of indirect resin composites, SR Nexco has been introduced in the dental market in 2012. SR Nexco paste is a light-curing laboratory composite indicated in the fabrication of the framework-free dental restorations (inlays and onlays). The combination of microfillers plus prepolymer enables a very high filling ratio and excellent physical properties. The use of the prepolymer allows the advantages of large filler particles to combine with those of microfillers. This technology allows for a superior strength of resin composite that if only inorganic

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microfiller were used.6 Cementation process is considered a critical step in ensuring the longevity of resin composite inlays. It may be difficult for choosing the appropriate resin cement to be used, because many dentin adhesives have been introduced in order to achieve a good bonding between resin cement and dental substrate.7-10 Resin cements can be classified according to tooth substrate pretreatment into: (1) etch-and-rinse, (2) self-etch, (3) self-adhesive.11-18

Etch & rinse resin cements are time consuming and sensitive to handling, efforts have been made to simplify the luting process and to provide a reliable as well as durable bond to dental tissues by producing self-adhesive resin cements. Which have attracted the interest of both manufacturers and clinicians, because they do not require any pretreatment of dentin surface.19, 20 The ideal resin luting cement should be impenetrable to oral fluids or acids produced by dental plaque and resist dissolution over the life time of restorations. In case of oral environment and presence of moisture or acids, there is increase risk of cement dissolution and bond degradation at the marginal gap leading to weakening and failure of restoration.21,22

Few studies evaluated the effect of acids produced by human dental plaque, and showed that lactic and other acids had detrimental effects on softening and surface degradation of polymeric resin materials.[21,22] Studies about the action of lactic acid on self-adhesive resin cement may increase the knowledge towards their durability in the oral environment. Therefore, it is necessary to evaluate the adhesion durability of indirect resin composite inlays both *in-vitro* and *in-vivo*.[23]

Microleakage is defined as undetectable penetration of oral fluids, bacteria, molecules and ions between the cavity wall and restoration, which affects the longevity of restoration performance.[24] The signs of microleakage phenomenon include immediate post-operative chronic sensitivity, and marginal discoloration.[25,26] Diffusion of cariogenic bacteria along tooth/restoration interface causes recurrent caries, pulpal inflammation, and later on, pulp necrosis.[27,28] Marginal leakage is attributed to many factors, including imperfect bonding at resin cement/tooth substrate interface as a result of polymerization shrinkage and stresses developed. So, marginal adaptation of resin composite inlays is an important factor and has a significant role in the long-term clinical performance of the restorations.[29]

Several studies have shown that, if there is occlusal marginal gap excessive wear of resin cement will occur, and if this gap is found in the proximal surface of the tooth nearby the gingiva, the risk of gingival inflammation and periodontal disease will increase. In addition, bacterial growth and their adhesion on resin cement causes recurrent caries, marginal discoloration and pulp inflammation. It is crucial to establish a strong and durable

Table 1 Indirect resin composite restorative system used in the study

Material	Composition	Manufacturer
SR Nexco liner	Dimethacrylates (48wt.%), barium glass filler, silicone dioxide (51wt.%), additional contents are catalysts, stabilizers and pigments (<1wt.%).	IvoclarVivadent AG Schaan, Liechtenstein
SR Nexco paste Layering materials (incisal & dentin)	Dimethacrylates (17-19wt.%), copolymer and silicone dioxide (82-83wt.%), inorganic filler (64-65wt.%), inorganic filler (64-65wt.%) (<1wt.%).	IvoclarVivadent AG Schaan, Liechtenstein
SR Nexco stain	Dimethacrylates (47-48wt.%), copolymer and silicone dioxide (49-50wt.%), additional contents are catalysts, stabilizers and pigments (2-3wt.%).	IvoclarVivadent AG Schaan, Liechtenstein
SR Gel	Glycerine, silicone dioxide and aluminium oxide	IvoclarVivadent AG Schaan, Liechtenstein

Table 2 Resin composite cements used in the study

Resin composite cement	Composition	Mechanism of adhesion	Manufacturer
N-Etch Syntac Heliobond Variolink N	Etchant: 37% phosphoric acid.-Syntac primer: 4% maleic acid, TEGDMA, PEGDMA, water, acetone.-Syntac adhesive: PEGDMA, glutaraldehyde, water.-Heliobond: Bis-GMA, UDMA, TEGDMA.-Variolink N base: Bis-GMA, UDMA, TEGDMA, fillers, ytterbium trifluoride, stabilisers, pigments.-Variolink N catalyst: Bis-GMA, UDMA, TEGDMA, fillers, tterbiumtrifluoride, stabilizers, pigments, penzoyl peroxide.	Dual-cured 3-step etch-and-rinse	IvoclarVivadentAGSchaan, Liechtenstein
Panavia F 2.0	ED primer:-Primer A (HEMA,10- MDP, chemical initiator, water, 5-NMSA)- Primer B (5-NMSA, chemical initiator, water panavia F2.0)-A Paste (quartz, glass,10- MDP, methacrylate, photoinitiator) -B Paste (silanated barium glass, NaF, methacrylate, chemical initiator)	Dual-cure one step self-etch	Kurary medical (Okayama, Japan
Rely X Unicem	-Powder (silica, glass fillers, calcium hydroxide, chemical curing initiators, light curing initiators) -Liquid (methacrylated phosphoric esters, dimethacrylates, chemical curing initiators)	Dual-cure self-adhesive Translucent aplicap	3M ESPE (ST Paul, MN USA)

Abbreviations: TEGDMA=triethylene glycol dimethacrylate; PEGDMA=polyethylene glycol dimethacrylate; UDMA=urethane dimethacrylate; Bis-GMA=bisphenol A di glycidylmethacrylate; HEMA=2-hydroxyethyl methacrylate; MDP=10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA=N-methacryloyl 5-aminosalicylic acid; NaF=sodium fluoride bond between resin luting cement and tooth structure to have an adequate marginal adaptation and sealing of inlay restorations in order to prevent microleakage.[30-34]The null hypotheses of this study wasthere is no difference between micro-leakage investigations of the three different resin cement strategies: etch-and-rinse Variolink N, self-etch Panavia F2.0 and self-adhesive RelyXUnicem for luting indirect resin composite inlays in MOD cavities.

MATERIALS AND METHODS

The present study was performed using a laboratory resin composite, SR Nexco (IvoclarVivadentAGSchaan, Liechtenstein), cemented with three different resin composite luting cements: an etch-and-rinse dual-cured Variolink N (IvoclarVivadentAGSchaan, Liechtenstein), self-etch dual-cured Panavia F2.0 (Kurary medical, Okayama, Japan) and self-adhesive dual-cured RelyXUnicem (3M ESPE, ST Paul, MN USA).

Teeth Selection

Forty-eight freshly extracted human molars from healthy individuals free from caries, or restorations were selected. Teeth were cleaned from the adherent soft tissues with a hand scaler (Zeffiro, Lascod, Florence, Italy) then stored and disinfected in 2% sodium azide solution for three days. After that, they were cleaned using a rubber cup and fine pumice water slurry then examined by binocular Stereomicroscope (30X magnification, SZ TP, Olympus, Tokyo, Japan) to exclude the cracked ones. Forty-eight molars were selected and finally kept in distilled water at 4^{o}C, which was changed periodically every 5 days throughout the study to avoid their dehydration, and teeth were removed only before their use.

The roots of selected teeth were embedded in a cylindrical polyvinyl chloride (PVC) rings up to 2 mm below the cementoenamel junction, using autopolymerizing acrylic resin (Acrostone, Cairo, Egypt) to complete stabilization of the teeth. A cylindrical Teflon mold, with a corresponding metal ring and two opposing screws at its top was used to hold the tooth in a centralized position, parallel to the long axis of the mold, during the setting of acrylic resin.

Cavity Preparation

At first, an impression was taken for each tooth before any preparation was done. Using equal amounts of the base and catalyst of high-viscosity impression paste (SHERATWIST 60) that were mixed together and seated in a sectional tray. A standardized MOD cavity was prepared using special kit for inlay preparation (Komet, Brasseler GmbH & Co. KG, Lemgo, Germany) and high speed handpiece with water coolant. To ensure high cutting efficacy, a new diamond instrument was replaced every five preparations.

The cavity dimensions were strictly standardized during preparation by securing the handpiece in a specially designed appliance that was constructed at Production Engineering and Mechanical Design Department, Faculty of Engineering, Mansoura University, Egypt. This device allowed accurate movements of the handpiece, resulting in approximately a standard divergence of cavity walls with a standard depth and width. The dimensions of the cavity preparation were 4mm buccolingually, 3 mm deep at the isthmus; 4 mm deep at the mesial and distal surfaces and the boxes were 1.5 mm at the base towards the pulp. The cavities were prepared 1-1.5 mm above the cemento-enamel junction. All the internal line angles were smoothed and rounded so as to reduce the possibility of stress concentrations.

Inlays Fabrication

Final impression was taken for each cavity using mix of light-viscosity impression paste (SHERATWIST 60), which syringed into the prepared cavity and over the high-viscosity primary impression which seated in the sectional tray. Then, every impression was sent to dental laboratory to be cast into a die stone. The technician fabricated all the restorations following the manufacturer's instructions.

Inlays Cementation

After try-in procedure, the 48 specimens were assigned into three groups (from group I to III and n=16) in relation to the resin cement used for luting the inlays. The cementation, finishing and polishing steps were followed according to the manufacturer's instructions. Each group was subdivided into two equal subgroups (n=8) relative to the storage media used (0.01M buffered lactic acid of pH 4 or distilled water of pH 7). Finally, half of the specimens of each subgroup (n=4) was stored in each storage medium for 24h while the other half was stored for 168h.

Group I: 16 inlays were cemented by Variolink N resin cement using etch-and rinse adhesive system.
Group II: 16 inlays were cemented by Panavia F2.0 resin cement.
Group III: 16 inlays were cemented by RelyXUnicem resin cement.

After specimens' storage in both media for both periods of time, all the 48 teeth in all groups were coated with two layers of nail varnish up to approximately 1mm from the restoration margins, and then were immersed in a 2% methylene blue solution for 24 hours. After that, every tooth was thoroughly cleaned by running tap water for 10 minutes, and sectioned in mesio-distal direction by an automated diamond saw (Isomet 4000, Buehler Ltd., Lake Bluff, IL, USA) under copious water coolant (Cool 2 water-soluble anticorrosive cooling lubricant, Buehler Ltd., Lake Bluff,IL, USA), with a concentration of 1:33, lubricant: water, so as to be observed for microleakage test by using Stereomicroscope and evaluated through scores.[35]The scores were collected and statistically analyzed using Pearson Chi-Square Test in order to compare the different study groups.

RESULTS

Concerning with microleakage results between all resin cements in general, it can be noticed that, there was significant difference ($p<0.05$) between all the types. Variolink N resin cement revealed the highest scores of dye pentration, followed by RelyXUnicem, then Panavia F2.0 which showed the lowest scores. According to the type of storage media either distilled water or lactic acid used in the study in general, results showed, there was high significant difference ($p<0.05$) between both of them. The highest scores of dye penetration were obtained with lactic acid and distilled water showed the lowest ones. In case of the different periods of storage time either 24h or 168h in general, there was high significant difference ($p<0.05$) between the two periods. Specimens stored for 168h showed higher scores of dye penetration than those which stored for 24h. Microleakage results showed that, no significant difference ($p>0.05$) between the three resin cements when stored in distilled water for 24h. However, results showed a significant difference ($p<0.05$) between them in case of using lactic acid as storage medium for 24h.

When comparing between all resin cements that stored in distilled water for 168h, it was noticed that no significant difference ($p>0.05$) between them. But, results revealed high significant difference ($p<0.05$) between them in case of specimens storage in lactic acid for 168h. Micro leakage results showed significant difference ($p<0.05$) between specimens of each resin cement that stored in distilled water and the ones stored in lactic acid for either 24h or 168h.Results revealed high significant difference ($p<0.05$) between the two periods of storage time (24h and 168h) when every resin cement stored within each storage medium in case of Variolink N, Panavia F2.0 and RelyXUnicem resin cement.Figs. 1,2,3&4.

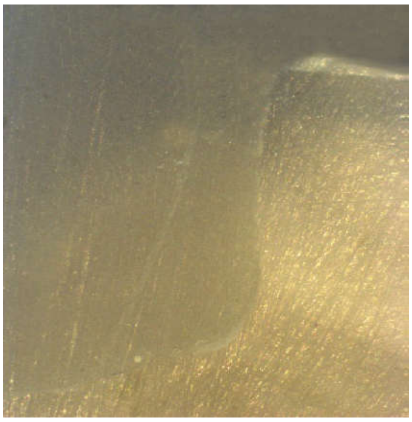

Fig 1 Score 0 of Panavia F2.0 stored in distilled water for 24h

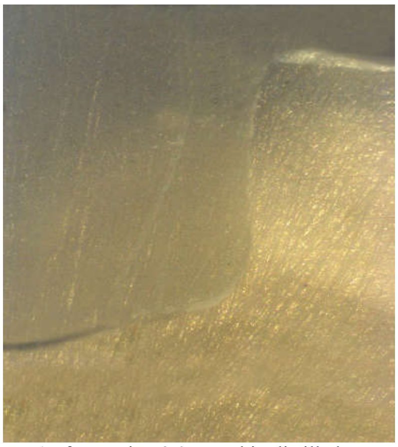

Fig 2 Score 1 of Panavia F2.0 stored in distilled water for 24h

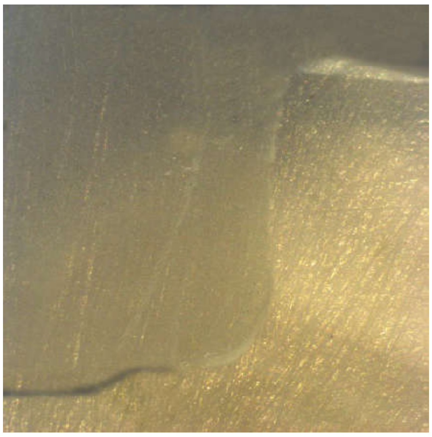

Fig 3 Score 2 of RelyXUnicem stored in lactic acid for 168h

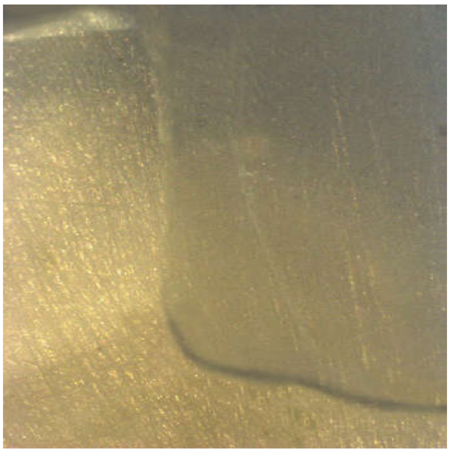

Fig 4 Score 3 of Variolink N stored in lactic acid for 168h

DISCUSSION

In the current study, Large MOD cavities prepared in molars were chosen because, they are considered to be the least durable design, due to high loads exerted in this region and the extensiveness of restoration.[36] A standardized cavity preparation was made using special inlay diamond instruments, fixed in a high-speed handpiece which attached to specially designed appliance in order to avoid bias and incorrect interpretation of the results.[37] Laboratory resin composite was chosen because of its relatively low cost, easy fabrication, ability to absorb loads and its improved physical and mechanical properties, that allow it to be used as an alternative to esthetic dental prosthetic treatment where masticatory load could be required.[38] Moreover, indirect resin composite exhibits less polymerization shrinkage, which is still the main drawback for direct composite restorations.[39]

Many dental adhesives have been introduced to provide adequate bond strength to both enamel and dentin substrates.[40] However, the clinical longevity of bonded restorations is still too short, due to tooth/resin cement interface degradation.[42] In laboratory studies, the durability of this bond is tested using different kinds of artificial aging methods like water, thermo-cycling, mechanical loading, degradation by enzymes and various chemical substances as acids. These *in-vitro* durability tests give detailed information towards the mechanisms of degradation. Although it is not right to generalize these laboratory studies to evaluate the durability of bond in clinical studies, there are some associations between laboratory and clinical information's on bonding effectiveness. Previous studies have evaluated the mechanical behavior of resin-based cements. However, until now little information has been available about their degradation in the oral environment.[41-46] Few published studies have shown that, durability of bond between resin-based cements and dentin can be affected by water immersion and over the course of time.[43-46]

Material degradation in the oral environment has two types; one is mechanical and the other is chemical degradation. Regarding to the clinical practice, two aspects are observed: firstly, after luting of the restoration, a film of resin-based cement will be exposed to the oral cavity. Secondly, this film is immediately subjected to severe conditions such as pH cycling, temperature changes and oral fluids, e.g., saliva, enzymes and organic acids produced by bacteria. Based on this, the chemical component would be the first to act on resin-based cement degradation, influencing the mechanical degradation at a later time. So, investigations about the chemical degradation could be important so as to predict the behavior of resin-based cement in the clinical service ability of indirect composite restorations.[44]

Water was chosen in the present study, because it is the main component of aqueous solutions and plays an important role in the oral environment as a weak solvent. Also, distilled water is considered an ISO-recommended solvent for resin cements. Although oral biofilm produces several acids, but lactic acid was used in this study, because it accounts for about 70% of the total acids present in the human dental plaque. Moreover, it was found that, there is a great increase in lactic acid after sucrose rinsing that undergoes for rapid degradation by lactate-producing bacteria, e.g., *S. mutans*. Based on this, during carbohydrate ingestion, there would be large amount of lactic acid in the oral cavity than the other organic acids produced by human dental plaque, such as propionic and acetic acids.[45]

Both water and lactic acid may act as solvents, their uptake by resin phase of luting cements may be caused by two processes. The first one may occur directly into the resin matrix by a diffusion-controlled process that is called free-volumetric theory. Which states that, molecules can enter inside the material via microvoids, structural defects or along filler/matrix interfaces without any actual binding to other polar molecules. The second theory states that, diffusion may be occurred by the actual binding to the hydrophilic groups forming hydrogen bonds. Concluding that, solvents can be absorbed by the unreacted monomers present in the polymer matrix of resin cement either physically by plasticization or chemically by hydrolysis and degradation. Creating stresses, crazes and finally forming microcracks that act as microscopic solvent reservoirs at filler/matrix interface, which considered the most likely route for solvents penetration. Leading to resin matrix softening and weakening which facilitate filler pull-out and finally decreasing the mechanical properties.[46]

The incorporated voids have oxygen-inhibiting areas of uncured monomers that increase the possibility for solubility of polymerized resin cement, and may also enhance the transport of liquid into and out of polymeric network resulting in increasing of sorption and solubility. These findings show that, although sorption and solubility are multi functional processes, they mainly depend on the hydrophilic nature and composition of monomer components of resin cement matrix.[46] They may considered the causes of many deleterious effects on other properties of resin cements such as marginal seal and bond strength. Cement at the marginal gap of luted restorations may be exposed to water from oral environment and lactic acid produced by colonized intraoral bactria.[45] Although water storage can compromise resin-dentin bond strength, but resin cements showed greater micro-morphological damage following storage in an acidic medium than storage in distilled water. Furthermore, low pH may increase the plasticizing effect of lactic acid and cause a reduction in the dimethacrylate matrix interchain interactions, thus accelerating the sorption rate within the material. Hence, Bond stability is clinically stressed by acids produced by bacteria.[47]

Several studies showed that, resin-based composites degradation was produced in low pH of acid environment used. Hence, confirming the role of a low pH of acids in the degradation phenomenon. So, low pH seems to have an influence on promoting the release of unreacted monomers and inorganic fillers and may additionally cause erosion at the filler surface. Resin cement solubility may be explained by different reasons. First, elution of unreacted components is more in acidic media compared to that in distilled water. Second, the amount of leachable components is dependent and inversely proportional to the degree of monomer conversion which may differ among the resin cements. Third, the method of mixing can be on paper pad (hand mixing) or auto mixing which may generate different amount and sizes of air voids within the resin which work as free surface areas.[47]

It has been shown that, during the first seven days, the main components released from resin cements are the residual unreacted monomers. Moreover, many solvents can leach out any component from the cured resin cement and this elution process can be occurred within 1-3 days. Therefore, the seven days (168 h) period was chosen in this study as the maximum time of storage for resin cements investigated in the study.[42] Although no relationship has been proven between the aging of inlay/tooth luting interface in the mouth and microtensile aged bond strength, but lengthy time of storage in water or lactic acid is considered a valuable method to evaluate the stability of the bond. Resin material's chemical composition, the hydrophilic components of the resin matrix, different storage media and aging time are important factors which affect resin cement degradation and hence reduce its properties.[48]

Results of the present study showed high degree of dye penetration in microleakage test and low values of microtensile bond strength with lactic acid more than distilled water immersion, especially for 168h more than 24h storage time. Moreover, Panavia F2.0 revealed the best results, followed by RelyXUnicem then finally Variolink N. This can be attributed to different rates of sorption by the resin matrices. High level of hydrogen bonds promoted by the functional groups that present in lactic acid molecule (-OH hydroxyl group and -COOH carboxyl group) with polar sites of dimethacrylate monomers (-OH- in Bis-GMA, -O- in TEGDMA and Bis-EMA and -NH- in UDMA) which found in the organic matrix of Variolink N resin cement. So, they can form strong hydrogen bonds with water and acids, increasing liquid uptake by resin matrix, increasing resin degradation and hence decreasing its mechanical properties.[48]

The ester groups in the resin matrix formed by phosphoric acid ester monomers (PO-O3R3) of RelyXUnicem resin cement, were hydrolyzed by lactic acid, leading to formation of alcohol and carboxylic molecules which may make the cement is more liable to moisture absorption, thus enhance resin matrix degradation. Also, the effect of acidic pH can cause chemical erosion of filler surface, facilitating their bonding and releasing of weak leachable ions. In case of Panavia F2.0, 10-MDP (10-methacryloxy decyl-dihydrogen phosphate) acidic functional monomer is found in its composition and has been rated as relatively hydrolysis stable because of its long carbonyl chain. It showed the best results than other resin cements in this study.[48]

The present study agreed with Da Silva *et al* trial where, lactic acid increased resin-based cements degradation more than water and significantly increased it with time.[49] Also, a study by Marghalani and others revealed results similar to this one where, a significant difference of sorption values between water and lactic acid, and the highest sorption exhibited after immersion in lactic acid for 168h.[50] But, Petropoulou *et al* were in disagreement with the present study where, SE and SA resin cements, which contain functional monomers, exhibited higher water sorption than conventional etch-and-rinse resin cement.[51]

CONCLUSION

Based on the results of the current study, and despite of the limitation of small sample size, it seems reasonable to conclude that micro-leakage showed good results with SE luting resin cement strategy.

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