ORIGINAL ARTICLE

Evaluation of Interface Adhesion Between Silicone Soft Liners and Denture Base After Immersion With Denture Cleansers by Scanning Electron Microscope

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ABSTRACT

Introduction: The compatibility of denture cleanser is crucial in the prevention of failure of adhesion between silicone soft liner and acrylic denture base, thus ensuring the durability of the prosthesis. This scanning electron microscope (SEM) study was to determine the mode of failure and measured the gap formation between silicone soft liners and Polymethylmethacrylate (PMMA) denture base after immersion in denture cleansers. **Methods:** A total of 135 specimens of PMMA denture base lined with three different silicone soft liners (GC Reline Soft, Mollosil and Tokuyama Sofreliner Tough) were immersed into denture cleansers (Polident® and Stearadent) daily and stored in distilled water at 37±1°C. Specimens were examined and sectioned at 2.5mm and 5.0mm from the margin after 1 day, 30 days and 90 days before analyzed. **Results:** No significant difference detected in the mode of failure and gap formation after one-day immersion. Adhesive failure was the commonest failure at the margin after 30 days (71.11%) and after 90 days (95.56%). However, 33.33% of specimens showed mixed failure at 5.0mm sectioned after 90 days. A significant difference of gap formation was demonstrated from Mollosil in Steradent at the margin and at 2.5 mm sectioned after 30 days and after 90 days ($p<0.05$). It was also observed at the margin and 5.0mm sectioned of specimens from Tokuyama in Steradent after 90 days of immersion ($p<0.05$). **Conclusion:** Specimen immersed in Steradent denture cleanser presented with wider gap formation as compared to Polident®, and adhesive failure is the commonest mode of failure.

Keywords: Denture cleanser, Failure mode, Gap of interface, Scanning Electron Microscopy, Silicone soft liner

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INTRODUCTION

Silicone soft liner is popular as denture liners because softer and absorbs shock better as compared to the acrylic liner. Previous studies have found that patients preferred soft lining materials as opposed to the acrylic base as it has better impact force reduction (1, 2, 3). The fitting surface of the denture base is well-formed by the liners as it has the elastic capacity and resilient capability (4, 5). This is improved by an adequate thickness of 2mm to 3mm of the soft liner that provides the cushioning effect (6). The silicone liner is designed to distribute loading stress more evenly by a dampening effect whilst maintaining its shape upon functions (1, 7). Clinical success of such a situation is determined by both the perceived softness of the lining materials and their durability (8, 9).

A denture made from two different materials with an adequate bond may provide a successful outcome. Although it has been a popular choice, the drawback of liner product is often inadequately surface adhesion bonded to the Polymethylmethacrylate (PMMA) denture base (7, 10, 11). The strength of the interfacial adhesion differently will influence and alter the failure of the crack at the interface. Debonding of interfacial occurred by a process of crack formation, propagation and subsequently joint failures (3, 12, 13). da Cruz Perez et al. (14) had found that the initiation of the delamination of the liner will happen at a weak interface adhesion, thus creating suitable conditions for staining and bacterial adhesion (p.67). This can accelerate the breakdown of a soft liner and lead to bacterial growth thus deterioration of the prosthesis. In the end, it will result in the loss of the reline material from the denture's borders and impression surfaces (13, 14).

Careful cleaning of the liner is important and should be kept simple yet effective and non-damaging. The original protocol of the post-insertion care and maintenance

of denture hygiene is essential in minimizing or eliminate adverse tissue reactions. However, they pose a challenge, as the improper care and handling by a patient may cause alteration in the physical property of the silicone soft liner after relining procedure (2, 14). Studies have been reported that certain types of denture cleansers cause significant deterioration of the liners and may undergo absorption resulting from the plasticization of the liner during immersion. Pisani et al. (15) had reported that certain types of denture cleansers cause significant deterioration of tissue conditioners. The union between the lining material and the acrylic resin had been influenced by the absorption of water, primer's action, and the nature of the denture base material (p.127). The patient may inquire about the choice of denture cleanser with their prescribed prosthesis in order to keep the longevity of the lining material (16-18). Thus, the denture cleanser should effectively inactivate or eliminates microorganisms without adversely affecting the fabricated material (19). Chemical cleaning by immersion type has been suggested as an efficacious method to prevent Candida albicans invasion and denture plaque formation (19, 20). An excellent denture cleanser should be able to reduce the risk of microbial colonization while minimally affect the physical properties of the liner.

Scanning electron microscopy (SEM) allowed qualitative visualization in three dimensions of dental materials (21, 22). However, not many studies measured the effect of denture cleanser on the adhesion between the denture base and liner using this tool (22). Although studies by Aydin et al. (22) and Muralidhar et al. (23) have been evaluated the influence of ageing in artificial saliva and distilled water, though the gap formed and severity of it was not quantitatively considered. This present study embarked on the gap formation at the adhesion interface and the characteristic of the failure after immersion into denture cleansers. It is important in determining an excellent denture cleanser that reduces the risk of microbial colonization while exerts an only minimal effect on the physical properties of the liner. Hence, suitable and clinically relevant of the denture cleanser and respective compatibility with denture soft liners may be recommended.

MATERIALS AND METHODS

Specimen preparation

This research was an interventional laboratory experiment study design. The detail and information regarding type of heat-polymerised polymethylmethacrylate (PMMA) denture base, three types of auto-polymerised silicone soft liners and two types of denture cleansers used and tested in this study is presented in Table I. Specimens in this study were made by using a mould from five separated stainless-pieces, which assembled to produce acrylic base and later for relining (Fig.1a). A total of 135 PMMA bases were prepared using the powder-to-liquid ratio of 2.34g:10ml as recommended by the manufacturer and process in a water bath (Acrydic10, Menfredi, Torino, Italy) using the curing cycle of 7 hours at 70°C followed by 1 hour at 100oC. All the PMMA specimen's surfaces were wet grounded with a twin grinding and polishing machine (Metaserv® 2000) on 600 grit silicon carbide paper until the thickness of each of the acrylic bases reached 10mm x 10mm with 3mm thickness. The acrylic base specimens were then stored in distilled water at 37 C in an incubator chamber (Memmert Gmbh) for 30 days before relining.

Relining phase

The acrylic bases were held in the mould and the soft lining materials when it is injected into the assembling part. This produced final specimens of size 10mm x 10mm x 6mm with a 3mm thickness of soft liners (Fig.

Table I: Materials used in this study

Brand Name	Material type	Main composition	Manufacturer
Impact Acrylic denture base	Heat polymerized polymethyl-methacrylate	Powder: polymethylmethacrylate Liquid: MMA, ethylene glycol dimethacrylate	Dental Export of London, London, England
GC Reline Soft	Auto-polymerized silicone soft liner	Cartridge type: Silicone dioxide, vynil dimethyl polysiloxane, hydrogen polysiloxane Filler: 37% Primer: Primer-R Ethyl acetate (> 90%)	GC Dental Products Corp, Tokyo, Japan
Mollosil	Auto-polymerized silicone soft liner	Tube type: Polydimethylsiloxane with functional group, filler, pigments, platinum catalyst Primer: polyacrylate, additives.	Detax GmbH & Co.KG, Ettlingen, Germany
Tokuyamma Sofreliner Tough	Auto-polymerized silicone soft liner	Cartridge type: Polyvorganosiloxane with 20% amorphous silica Primer: Sofreliner primer (Ethylacetate and adhesive polymer)	Tokuyama Dental Corp, Tokyo, Japan
Polident® denture cleansers	Tablets effervescent denture cleanser	Citric acid 20%, sodium carbonate 11%, potassium peroxymonosulphate 4.3% and sodium perborate monohydrate 10%	GlaxoSmithKline,UK
Steradent denture cleanser	Tablets neutral enzymatic denture cleanser	Sodium sulphate 10%, sodium carbonate peroxyhydrate 10%, sodium carbonate 10% And potassium peroxymonosulphate 10%	Reckitt Benckiser (UK) Ltd, Swindon

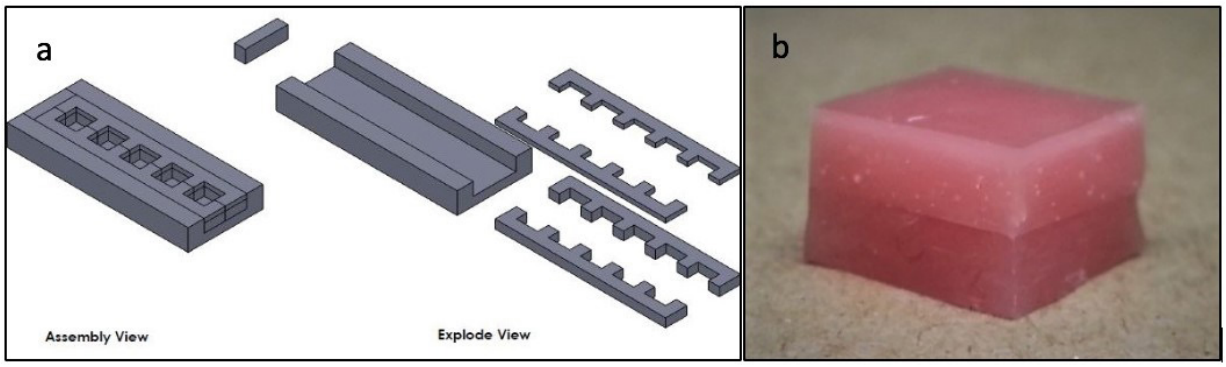

Figure 1: Preparing specimens and relining. a) Schematic design of mould for specimens construction. b) Specimen of silicone liner onto PMMA denture base (GC Reline Soft).

1b). A Perspex base was made to hold the components precisely during the relining procedure. The excess soft liner was removed with a sharp surgical blade. Finishing and polishing were done following a specific recommendation from the respective manufacturer. The denture bases-soft liners were divided into nine (9) groups with 15 specimens for denture cleansers combination group (Table II). All specimens were soaked and immersed in prepared solutions following the manufacture's instruction. The specimens were examined after one day, 30 days and 90 days.

Table II: Grouping of specimens

Group number	Group combination	Silicone liners	Cleansers or disinfectants	Number of samples
1	GC-DIS	GC Reline Soft	Distilled water	15
2	GC-POL	GC Reline Soft	Polident® denture cleanser	15
3	GC-STE	GC Reline Soft	Stearadent denture cleanser	15
4	MOL-DIS	Mollosil	Distilled water	15
5	MOL-POL	Mollosil	Polident® denture cleanser	15
6	MOL-STE	Mollosil	Stearadent denture cleanser	15
7	TOK-DIS	Tokuyama Sofreliner Tough	Distilled water	15
8	TOK-POL	Tokuyama Sofreliner Tough	Polident® denture cleanser	15
9	TOK-STE	Tokuyama Sofreliner Tough	Stearadent denture cleanser	15

N=135

Sectioning of specimens

Prior to SEM evaluation, the acrylic base-soft liner specimen was embedded in clear self-cure epoxy resin as a holder for cross-section cutting. Each specimen was sectioned with a high-speed precision sawing machine at 2.5mm and 5.0 mm distance from margin to examine the extent of gap formation at adhered surface and bonding interface between silicone soft liners and denture base.

Evaluation of the interface with SEM

The interface of each specimen was subjected to the field emission scanning electron microscope (FESEM, Quanta 250, FEI, Hillsboro, USA) examination. The SEM photographs were developed with x250, x500 and x1000 magnifications for inspection (Fig.2). Inspection of adhesion interfaces was performed to determine the nature of the failure, whether adhesive, cohesive or no failure. The following criteria were used: i) no failure, ii) adhesive failure (failure between the adhesive or soft liner and denture base), iii) cohesive failure (failure or tear within the soft liner and soft liner with adhesive) and iv) mixed failure (both adhesive and cohesive failure were present) (11, 12). The gap of the interface of SEM photographs was observed and measured using xT microscope Control software using Line measurement.

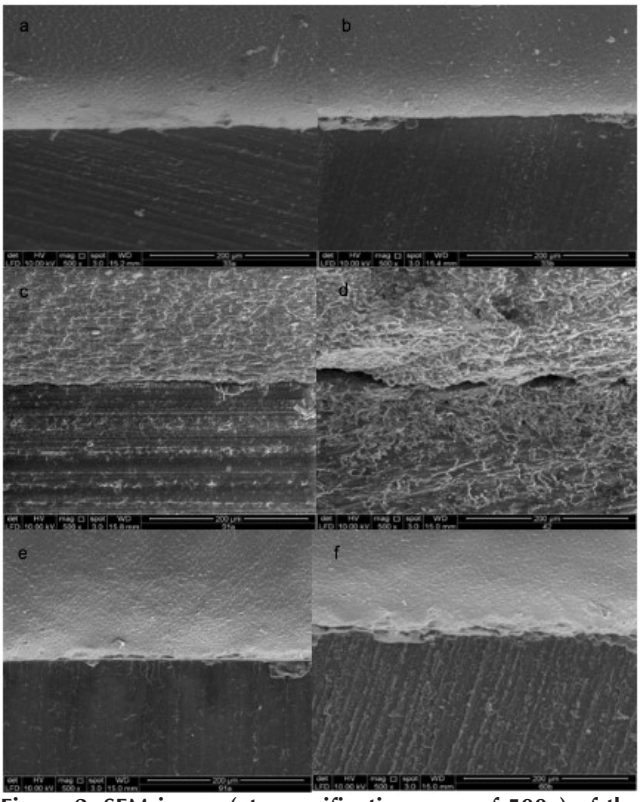

Figure 2: SEM image (at magnification zone of 500x) of the mode of failure and gap separation at the interface between silicone soft liners and denture base. a) GC Reline Soft in distilled water immersion after day 1 and b) after day 90. c) Mollosil in Steradent immersion after day 1 and d) after day 90. e) Tokuyama Sofreliner Rough in Polident® immersion after day 1 and f) after day 90.

Each photomicrograph was measured at three (3) different points and the mean average of separation (μm) was analysed.

Statistical analysis

All data were analysed using the Statistical Package for Social Science (SPSS) for Windows (Version 23.0, Incorporated USA). Mode of failure was described in number and percentage, then Fisher's Exact test was used to analyse the failure between silicone liners after sectioning at different days. The descriptive statistical analysis was performed to obtain the median and inter-quartile range (IQR) for gap measurements. Kruskal Wallis test was used to compare the differences in the interface gaps among the three soft liners following their immersion in denture cleansers. For all tests, the level of significance was set at $p \leq 0.05$.

RESULTS

Mode of failure

The mode of failure was observed at three different interfaces which were at the margin, 2.5mm sectioned and 5.0mm sectioned. After one day of immersion, there was no significant difference ($p>0.05$) indicating that the mode of failure between group samples after immersion was not greater than the other (Table III). After 30 days immersion, adhesive failure was the commonest failure in all groups at different sectioned (at the margin = 68.89%, at 2.5mm sectioned = 44.44% and 5.0mm sectioned = 15.56%) (Fig. 3). It also observed that the trend of failure increase as the ageing time was increased. A Fisher's Exact test recorded a significant difference of failure between studied groups at the margin ($p = 0.003$), at 2.5mm ($p = 0.000$) and 5.0mm sectioned ($p = 0.001$) (Table III). Group of Mollosil specimens immersed in Steradent denture cleanser showed 100% adhesive failure compared to other groups at the margin and 2.0mm sectioned.

Based on Fig. 3, the commonest mode of failure was an adhesive failure in all groups at the margin (86.67%), at 2.5mm (53.33%) and 5.0mm sectioned (42.22%) after 90 days of immersion. However, the differences at the margin, was statistically not significant $\chi^2(N=45) = 9.081$ $p=0.091$, indicating that mode of failure between group samples after immersion was no difference. It also observed that the trend of adhesive failure decreases as the depth of area increase whereby cohesive failure was higher at 2.5mm sectioned (17.76%). Mollosil and Tokuyama liners demonstrated 100% adhesive failure at the margin compared to GC soft liner (80%).

Gap at interface

There were also no significant differences of gap measured between groups of liner regardless of the denture cleansers used at the margin, 2.5mm and 5.0mm sectioned after one day (Table IV). However, samples from Mollosil liner immersed in Steradent denture cleanser (median, IQR= 4.362, 2.360) exhibited the highest gap of interface compared to other groups at the margin after 30 days immersion. A further analysis using multiple comparisons (Bonferroni post hoc test) revealed the gap of the interface was a significant difference between GC- DIS and MOL-STE ($z=-2.785$, $p=0.040$). When specimens were sectioned at 2.5mm, Mollosil soft liner immersed in Stearadent denture cleanser (median, IQR=2.720, 1.746) exhibited the highest gap between interface compared to other groups. There was a statistically significant difference in the gap interface between groups GC-DIS and MOL- STE ($z=-2.785$, $p=0.040$) and between GC-POL and MOL-STE (z=-

Table III: Comparison between liners and cleansers group combination by mode of failure after day 1, day 30 and day 90

Variables	Day 1		Day 30		Day 90	
	χ^2 stat	p	χ^2 stat	p	χ^2 stat	p
Mode of failure at the margin	19.976	0.257	18.358	0.003	9.081	0.091
Mode of failure at 2.5mm sectioned	9.646	1.00	34.278	0.000	35.340	0.001
Mode of failure at 5.0mm sectioned	9.646	1.00	22.246	0.001	30.646	0.019

N=135, *p-value ≤ 0.05 is statistically significant; Fisher's Exact Test (X^2)

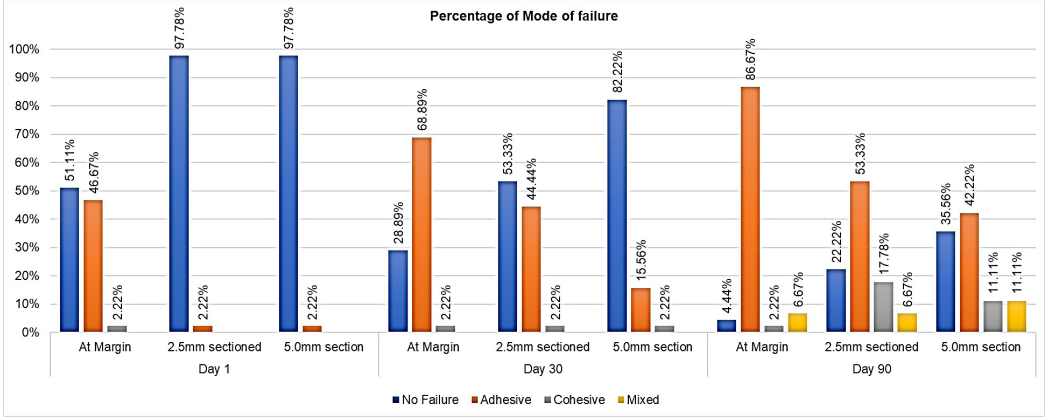

Figure 3: Mode of failure specimens in percentages after immersion and after sectioning at different days (n=45/Day).

Table IV: Comparisons of gap formation at the interface among all groups after day 1, day 30 and day 90

Variable	Day 1		Day 30					Day 90				
	H(df)	p	H(df)	p	Group (Median ± IQR)	z	p	H(df)	p	Group (Median ± IQR)	z	p
Interface gap at the Margin	9.172 (8) Cohen η^2 = 0.208	0.328	16.965(8) Cohen η^2 = 0.386	0.030*	GC-DIS (0.00±2.909) vs MOL-STE (4.362±2.360	-2.785	0.040*	25.203(8) Cohen η^2 = 0.573	0.001*	GC-DIS (2.727±3.590) vs MOL-STE (4.985±3.273) GC-DIS (2.727±3.590) vs TOK-STE (5.453±1.965)	-2.785	0.040*
Interface gap at 2.5mm sectioned	8.000(8) Cohen η^2 = 0.182	0.433	22.806(8) Cohen η^2 = 0.520	0.004*	GC-DIS (0.00±0.00) vs MOL-STE (2.720±1.746) GC-POLI (0.00±0.00) vs MOL-STE (2.720±1.746)	-2.785	0.040*	24.941(8) Cohen η^2 = 0.567	0.002*	GC-DIS (0.00±0.00) vs MOL-STE (3.885±2.469) GC-DIS (0.00±0.00) vs TOK-STE (3.608±3.431)	-2.785	0.040*
Interface gap at 5.0mm sectioned	8.000(8) Cohen η^2 = 0.182	0.433	26.283(8) Cohen η^2 = 0.597	0.001*	GC-DIS (0.00±0.00) vs MOL-STE (2.238±1.360)	-2.785	0.040*	18.756(8) Cohen η^2 = 0.426	0.016*	GC-DIS (0.00±0.00) vs MOL-STE (2.526±2.120) GC-DIS (0.00±0.00) vs TOK-STE (2.610±0.845)	-2.785	0.040*

N=135, *p-value ≤0.05 is statistically significant; Kruskal-Wallis test (H), Mann Whitney U Test (z).

2.785, p=0.040).

As shown in Table IV, specimens from group Mollosil and Tokuyama liners immersed in Steradent exhibited the highest gap at interface compared to other groups at the margin after 90 days of immersion. The Bonferroni post hoc test also recorded a significant difference in the gap of an interface at the margin when observed at all sectioned between GC-DIS to MOL-STE (z=-2.785, p=0.040) and GC-DIS to TOK-STE (z=-2.785, p=0.040).

DISCUSSION

Adhesive failure implies that bond strength among the liner molecules and between soft liner to denture base is nearly the same. The bond strength of silicone soft liners depends on the tensile strength between the material and adhesive used (24, 25). According to the research done, an adhesive failure at the margin indicated that bond strength between soft lining material and the denture base material is lower than the tensile strength of the soft lining material (25, 26). When failure was genuinely adhesive, the inability of adhesive in adapting to the substrate has probably attributed a cause (12, 27). Hence, the bonding agent was identified as the locus for bonding failure. The result of this study was consistent as reported previously that after one day, the mode of failure was mainly adhesive for all the soft lining materials with the PMMA base group, except for Tokuyama Sofreliner Tough immersed in distilled water. On the other hand, studies had reported that aged specimens demonstrated two types of the mode of failure, adhesive and cohesive at the interface between the soft liner and the acrylic resin (12, 27, 28). The same finding also reported by Muralidhar et al. (23), the mode of failure between GC Reline Soft and PMMA denture base was mainly adhesive and mixed with ageing significantly influenced the failure. There was probably due to the inherent physical property of the materials and sorption of liquid. This can be attributed to the hydrolysis of the polyorganosiloxane molecules when in contact with water and denture cleansers. Similarly in this study, increment in mixed failure mode is observed after 90 days of immersion. McCabe et al. (26) explained that in the mixed failure mode tensile strength of the soft lining material and the tensile bond strength are nearly equal. Mollosil has a significantly lowest tensile strength compared to GC Reline Soft and Tokuyama Sofreliner Tough. This justified the lowest bond strength obtained for this material in this study.

The manner in the previous study, they observed the integrity between soft liner and denture base interface in artificial saliva and distilled water with the influence of the ageing process with SEM (23, 29). In general, all denture cleanser demonstrated significant changes in gap formation after 90 days. At the margin of each specimen, all groups showed gap formation with the mean average between 1.9µm to 5.6µm. Silicone liner specimens immersed with Steradent denture cleanser

showed higher gap formation compared to others. In this present study, the immersion of specimens in distilled water showed the least gap formation compared to the others. Only after 90 days of ageing distilled water manifests influence in gap formation. This may be due to the high-water sorption of the denture base polymer which may result in leakage and may also explain the lower bond strength presented. The high permeability of silicone to water results in the rapid transfer of water to the bond site leading to bond breakdown by hydrolysis of the cross-linkages.

At the end of this study, gap formations occurred in all marginal sections as compared to assessment a day following immersion. Formation of gap or separation of various liners from denture base recorded a mean average value of up to 50μm following ageing had been reported (22). GC Reline Soft was found to be less influenced by immersion in denture cleansers. A similar finding was discovered by Mutluay and Ruyter (27) which concluded that GC Reline Soft had good bonding properties when compared to other soft lining materials. This implies that with time, immersion with denture cleanser may increase the penetration depth deeper into the specimen. As explained before, the active ingredient contents may be the cause for the detachment of polymeric and hydrolysis of the molecules (25, 30, 31). As similarly observed, separations at the interface are increased at the end of the study.

This study also demonstrated that Steradent had an influence on the failure of the bond between silicone liner and PMMA denture base, probably due to deep penetration of the cleanser into the specimen. The contents in the Steradent with active ingredient may be attributed to the significant changes in the mode of failure. The chemical agent may cause detachment in the polymer and hydrolysis of the molecules. This leads to the propagation of molecules and interfaces failure formation between the studied liner and base (6, 7). Therefore, the ideal denture cleanser when relining is performed could be a Polident® denture cleanser. It may preserve the longevity of the soft lining materials hence prolongs clinical usage.

The finding of this study proved that denture cleanser and ageing affect adhesion at the interface between silicone liners and denture base. The effect of ageing and immersion in water of soft lining materials also had been discussed by Al-Athel et al. (29,p.995). Several mechanisms had been proposed in the attempt to explain the increment in failure rate such as water absorption indirectly results in swelling of the lining material, stress concentration at the interface, and resulted in a reduction in bond strength (30-33). Kawano et al.(1) and Wright(6) demonstrated that silicone-based soft liner is highly permeable to water and it passes through silicone at approximately ten thousand times as fast as through acrylic thus, when combining with the active ingredient in cleanser it gives a double impact to the failure formation at the interface. The question could be raised here whether the difference in the time of immersion may be influenced in the gap formation. However, it is noted that manufacturer-specific procedures would need to be followed as an effective measure in infection control. Future studies may need to take into account the different immersion periods prescribed with different denture cleansers.

CONCLUSION

Through this study, it was observed that the adhesive mode of failure was the most common failure at the early phase of the study, but over time there was an increase in all types of failure. An immersion in Steradent denture cleanser demonstrated greater gap formation of the interface between silicone liners and denture base compared to Polident® cleanser. There was less gap formation between GC Reline Soft and PMMA base compared to others after 90 days. It indicates that GC Reline Soft may be suitable as long term soft lining material compared to Tokuyama Sofreliner Tough and Mollosil. This study could be used as a recommendation of the suitable denture cleanser after the relining of a denture with silicone soft liners. A more successful long-term relining durability is achievable with Polident® cleanser compared to Stearadent as it demonstrated the least change in the gap of an interface between liner and denture base.

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