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Original Article

The Effects of Eucalyptus Oil, Glutathione, and Lemon Essential Oil on the Debonding Force, Adhesive Remnant Index, and Enamel Surface During Debonding of Ceramic Brackets

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Main Points
- Ceramic bracket debonding is facilitated by immersing teeth bonded with them in glutathione for 10 minutes.
- There was no adhesive left behind on teeth treated with glutathione, thus exposing a clear enamel surface- exhibiting peel-off effect.
- Glutathione exhibited superior debonding effects, followed by eucalyptus oil and lemon essential oil.
- A clinically viable option of using a customized tray with glutathione, similar to tooth bleaching trays, to facilitate debonding of ceramic brackets is very much possible in the near future given the reduced immersion time of just ten minutes.

ABSTRACT

Objective: The present study aimed to find a chemical reagent that would reduce the debonding force to enable easier debonding of the ceramic brackets, thus reducing enamel damage as well as chair side time.

Methods: The study included 4 groups -control (distilled water), eucalyptus oil, glutathione and lemon essential oil for immersing teeth bonded with ceramic brackets. Samples (25 in each group), extracted first premolars, were mounted and immersed in their respective solution for a duration of 10 minutes following which they were tested to evaluate the debonding force using the INSTRON universal testing machine. The amount of adhesive left behind on the enamel surface was evaluated using adhesive remnant index (ARI) score and surface changes were checked using a scanning electron microscope.

Results: Teeth immersed in glutathione showed the greatest amount of reduction in debonding force (p=0.001) compared with other groups. ARI scores were low for specimens immersed in glutathione. SEM images showed that teeth in the glutathione group had a cleaner enamel surface, suggesting less or no adhesive was left behind and no sign of enamel damage after debonding ceramic brackets.

Conclusion: Specimens that were immersed in glutathione for a duration of 10 minutes before debonding of ceramic brackets showed the greatest reduction in debonding force compared with control and demonstrated peel off effect with no enamel damage. Glutathione can be used as an effective reagent during the clinical debonding of ceramic brackets.

Keywords: Debonding force, adhesive remnant index, ceramic brackets, eucalyptus essential oil, glutathione, lemon essential oil, peel off effect

INTRODUCTION

Ceramic brackets were introduced in clinical orthodontics to meet the increased demand for an aesthetic appliance and currently are clear alternatives to stainless steel brackets.[1,2] Despite the superior aesthetic advantage, clinicians are faced with challenges associated with increased bond strength and low fracture resistance while debonding, like enamel tear outs, pain, bracket failures, bracket fractures and cracks.[3,4] A systematic review and meta-analysis

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conducted to evaluate enamel micro cracks revealed that debonding was associated with increase in the number, length and width of enamel micro cracks irrespective of brackets.[5] A quantitative analysis of enamel loss during debonding of monocrystalline and polycrystalline ceramic brackets revealed that there was loss of 33µ and 21µ and post -clean-up loss of 18µ and 28µ of enamel respectively. Monocrystalline ceramic brackets left behind most of the adhesive while polycrystalline brackets fractured during debonding and fragments of brackets were left behind along with remnants of adhesive.[6]

Several techniques have been used to overcome the problems encountered during debonding of ceramic brackets like mechanical, ultrasonic, electro-thermal and lasers debonding.[7-10] The use of ultrasonic waves for debonding ceramic brackets can be advantageous over conventional methods in that bracket-adhesive interface is subjected to vibrations but the duration is significantly greater and may be uncomfortable for the patients.[11]

Electrothermal debonding (ETD) focuses on softening of the adhesive material leading to bracket removal with minimal force.[12,13] Laser-aided debonding of ceramic brackets is conceptually similar to electro-thermal approach and works by effectively controlling the thermal energy delivered.[14]

Clinical scenarios are always different from experimental set ups and irrespective of acceptable efficacies of any techniques previously described, there is always a risk of enamel damage, thermal injury to pulp and inhalation of ceramic debris by patients.

Larmour et al.[15] investigated the effects of certain essential oils (EOs) and phenolic agents on the debonding behavior of ceramic brackets. Peppermint oil was used as a debonding agent and it proved helpful in removal of residual resin from the enamel surface by a significant softening of resin.[16] Apart from peppermint oil the effect of ethanol, eucalyptus oil, and hot water have been studied. Among these, immersion of brackets for 10 minutes in eucalyptus oil significantly reduced the bond strength compared with control groups.[15-17] The effect of EOs like lemon oil and glutathione on the debonding behavior of ceramic brackets is yet to be researched and this study was an attempt to explore this possibility. The present study evaluated and compared the debonding force, adhesive remnant index (ARI) and damage to tooth structure following debonding ceramic brackets after being immersed in 3 different chemical reagents (eucalyptus EO, glutathione oil and lemon EO) and (distilled water) for ten minutes. The null hypothesis was "there was no difference in the debonding force, the adhesive remnant score and damage to enamel between groups bonded with ceramic brackets immersed in distilled water, eucalyptus oil, glutathione and lemon EO."

METHODS

Sample size was calculated using G*Power (ver 3.1.9.2) with data taken from a previous study.[15] A sample size of 92 was estimated with an effect size of 0.272, alpha error of 0.20 and 80% power of the study. However, sample size was increased to 100 anticipating tooth fracture or other eventualities, which would result in exclusion of the samples.

This study involved four groups (three experimental groups and one control group); with 25 samples in each group.

- Group I: Control group/Distilled water
- Group II: Eucalyptus oil
- Group III: Glutathione oil
- Group IV: Lemon EO

Premolar teeth were extracted, and teeth with sound enamel were selected for this study. Teeth with caries, hypoplastic enamel, or restoration on the buccal surface and teeth with enamel cracks were excluded. Samples were cleaned to remove debris and calculus by scaling and polishing, then stored in distilled water at room temperature for a period of one week. The teeth were then mounted in resin blocks along the long axis. The teeth with blocks were cleaned using pumice slurry, rinsed with water and dried with compressed air. Orientation marks on vertical and horizontal axes were marked on the buccal enamel surface to guide in bonding the brackets. All bonding procedures were performed by the same operator according to the manufacturer's instructions. The buccal enamel surfaces were etched with 37% phosphoric acid gel for 20 seconds, rinsed for 60 seconds, and dried with compressed air, bonding agent was applied using an applicator tip and cured, followed by the placement of ceramic bracket 3M Clarity, (3M, Monrovia, Calif, USA) using a bracket positioner on to the tooth and excess flash was removed. A positioning gauge was used to orient the ceramic bracket according to the marks placed on the buccal tooth surface and light cured. The samples were then placed in distilled water for 1 week to allow complete polymerization of the resin.[15]

EOs are usually used in 0.5%-5% concentration. However, 2% concentration is most commonly used as a safe dose to prevent any unwanted irritation of the oral mucosa. The concentration of the reagents used for this experiment was that of a mouth rinse since the purpose of the reagent is external application and not to be used systemically. A good rule of thumb when seeking to make 2% dilution is to add 10-12 drops of EO to each fl. ounce (30 mL) of carrier (water).[18] Teeth samples with bonded ceramic brackets were immersed for 10 min in their respective chemical reagents. Following the immersion, they were subjected to debonding.

A material testing machine was used to measure and record the force required during debonding procedure in this study. Each tooth was placed in a fixture provided in the Universal Testing Machine (Instron 5566, Norwood, Mass, USA) to simulate the firm grip of the tooth inside the bone socket in the oral cavity. This procedure was done to avoid experimental errors due

to specimen movement which may be caused by the force exerted by the material testing machine. The brackets were removed by applying shear load through cross head blades of testing machine as recommended previously.[19] The debonding force was displayed as a digital readout (in Newtons) in the universal testing machine, operating with a cross-head speed of 1 mm per minute. The magnitude of the debonding force which was recorded in newtons (N) was further converted to mega pascal (MPa) by dividing the newton value with the surface area of the bracket base (9.61 mm^2) as per manufacturer instructions.

The ARI scores were used to examine the site of bond failure and classify the distribution patterns of residual adhesive.[20] This index consists of the following scoring:

- Score 0= No adhesive left on the tooth surface
- Score 1= Less than half of the adhesive left on the tooth surface
- Score 2= More than half of the adhesive left on the tooth surface
- Score 3= All adhesive left on the tooth surface

Four specimens, randomly selected from every group, were examined with a scanning electron microscope (SEM) to obtain a micrograph of the surface enamel after debonding. The samples were sectioned using a rotary handpiece into small sections for ease of evaluation. Sectioning was performed distal to the buccal cusp of the premolar in the occluso- cervical direction in a smooth motion to maintain the integrity of the hard tissue. All samples were then conventionally metallized (Gold sputtering JEOL JFC 1100E) and observed under a SEM.

Statistical Analysis

The data were analysed using SPSS software (IBM SPSS software for windows, version 23.0, Armonk, NY) for normality using Shapiro-Wilk test, since the data was not normally distributed, Welch's one-way ANOVA was performed to compare debonding force between the groups. Multiple comparisons between the groups were carried out by Games-Howell post-hoc test.

The frequency distribution was used to present the categorical data representing the ARI scores. The mean rank is the average of the ranks for all observations with each group of the study, which implies that the group with the lowest mean rank has the least ARI scores. As the distribution was not normal, ARI scores were analysed using Kruskal-Wallis test followed by the Dunn test for pairwise comparisons between groups.

RESULTS

The mean debonding force of the groups are given in descending order- control was 121.44 Newton (N) followed by 68.44 N in the lemon EO group, 56.46 N in the eucalyptus oil group and the least was exhibited by specimens immersed in glutathione (50.56N). There was a statistically significant difference in the mean scores of the debonding force between the groups (p=0.001) (Table 1).

Table 2 indicates that there was a significant difference of debonding forces between glutathione and eucalyptus oil with other two groups. Thus, debonding force drastically reduced when the samples were immersed in glutathione and eucalyptus oil.

For ease of comparison and comprehension, debonding force values were converted into megapascals to arrive at shear

Table 1. Comparison of debonding force between different groups

	N	Mean (Newtons)	Mean (MPa)	SD	Std. error	F	Sig.
Control	25	121.44	11.56	28.781	5.756	77.361	p=0.001*
Eucalyptus	25	56.48	5.37	15.785	3.157		
Glutathione	25	50.56	4.81	10.775	2.155		
Lemon oil	25	68.44	6.51	12.583	2.517		

Level of significance at p<0.05
SD, Standard deviation; N, Number of specimens
*Statistically significant at p<0.01 using Welch's One-Way ANOVA (unequal variances)

Table 2. Results of Games-Howell post-hoc test

(I) Groups	(J) Groups	MD (I-J)	Sig.
Control	Eucalyptus	64.96	0.001*
	Glutathione	70.88	0.001*
	Lemon oil	53	0.001*
Eucalyptus	Glutathione	5.92	0.42
	Lemon oil	-11.96	0.024**
Glutathione	Lemon oil	-17.88	0.005*

Level of significance at p<0.05; MD, Mean difference
Statistically significant at p<0.05**and p<0.01 *using Games-Howell post-hoc test for unequal variance

bond strength during debonding (Table 1). It was found that specimens immersed in glutathione exhibited the least shear bond strength (4.81MPa) followed by specimens immersed in eucalyptus oil (5.37MPa), lemon EO (6.51MPa) and highest in control (11.56MPa).

Teeth immersed in glutathione had the highest proportion (76%) of specimens without any adhesive remnants (score 0), followed by specimens in eucalyptus oil (64%). About 12% of samples immersed in lemon oil had a score 3 that indicated half of the composite was on the enamel surface and rest half on the bracket base, 4% of specimens immersed in eucalyptus oil had a score 2. None of the specimens immersed in glutathione had either score 2 or 3. Control group had the highest percentage of specimens (20%) with score 3 suggesting that the entire composite was left on the enamel surface (Table 3).

There was a statistically significant difference in adhesive remnant scores between different groups, (p=0.001) with mean rank scores of 68.4 for group I, 42.16 for eucalyptus oil group, 36.20 for glutathione group and 55.24 for lemon EO group, respectively. The mean rank was lowest in the glutathione group, indicating poor ARI scores, implying that no adhesive was left behind after debonding, when specimens were immersed in glutathione (Table 4). There was a statistically significant difference between the mean ranks of the control and eucalyptus oil groups (p=0.003) and between the control and glutathione groups (p=0.000). However, there was no difference between the other groups (p>0.05) (Table 5).

When specimens immersed in distilled water were observed under 200X magnification, presence of enamel cracks were revealed (Figure 1). Thus, teeth immersed in distilled water

Table 3. Frequency and percentage of adhesive remnant index scores in different groups

		Adhesive remnant index scores				
		0	**1**	**2**	**3**	**Total**
Control	N	6	9	5	5	25
	%	24.00%	36.00%	20.00%	20.00%	100.00%
Eucalyptus	N	16	8	1	0	25
	%	64.00%	32.00%	4.00%	0.00%	100.00%
Glutathione	N	19	6	0	0	25
	%	76.00%	24.00%	0.00%	0.00%	100.00%
Lemon oil	N	10	11	3	1	25
	%	40.00%	44.00%	12.00%	4.00%	100.00%

N, Number of specimens; %, Percentage

Table 4. Mean rank of adhesive remnant index scores

Groups	N	Mean Rank	χ^2 value	Sig.
Control	25	68.40	22.153	p=0.001*
Eucalyptus	25	42.16		
Glutathione	25	36.20		
Lemon oil	25	55.24		
Total	100			

Level of significance at p<0.05 N, Number of specimens *Statistically significant at p<0.05 using Kruskal-Wallis test

Table 5. Pairwise comparison using Dunn test

Pairwise Comparisons of Groups					
Sample 1-Sample 2	**Test statistic**	**Std. error**	**Std. test statistic**	**Sig.**	**Adj. Sig.[a]**
Glutathione- Eucalyptus	5.960	7.463	0.799	0.425	1.000
Glutathione- Lemon oil	-19.040	7.463	-2.551	0.011	0.064
Glutathione- Control	32.200	7.463	4.315	0.000	0.000
Eucalyptus-Lemon oil	-13.080	7.463	-1.753	0.080	0.478
Eucalyptus- Control	26.240	7.463	3.516	0.000	0.003
Lemon oil- Control	13.160	7.463	1.763	0.078	0.467

Each row tests the null hypothesis that the sample 1 and Sample 2 distributions are the same.
Asymptotic significances (2-sided tests) are displayed. The significance level is 0.05.
[a]: Significance values have been adjusted by the Bonferroni correction for multiple tests

clearly exhibited the presence of enamel damage caused due to the higher debonding force. Teeth immersed in eucalyptus oil showed a clear enamel surface in the debonded site under 20X magnification. Under 200X magnification, teeth immersed in eucalyptus oil showed no signs of enamel damage, but the roughened enamel surface could be appreciated which suggested the presence of remnant resin at the microscopic level (Figure 2). Glutathione showed the cleanest enamel surface post debonding, magnification under 200X showed no sign of damage to the enamel surface (Figure 3). Specimens of the lemon essential oil group under 20X magnification showed a mesh pattern of the resin left on the tooth surface (ARI score of 3) which was further appreciated under 200X magnification. Though there was lot of resin on enamel surface, there was no sign of enamel damage (Figure 4).

DISCUSSION

Ceramic brackets are made of aluminum oxide and two types, mono- and polycrystalline, that vary in strength, translucency and fracture toughness.[21] Due to their natural brittleness, debonding of ceramic brackets is challenging because of fractured brackets and enamel damage.[21-23] The study was conceived with the aim of testing a new reagent that can reduce the debonding force of ceramic brackets so that enamel damage is prevented and patient discomfort during debonding is alleviated.

EOs are natural, volatile complex compounds that possess anti-oxidant and antimicrobial properties, are derived from aromatic plants, soluble in organic solvents but exhibit hydrophobic nature with density lower than water.[24-27] There has been no published research on the effect of glutathione on the bond strength of ceramic brackets. This study was conducted to evaluate the debonding force, Remnant Adhesive Index and damage to tooth structure as assessed by SEM, immersed in three different chemical reagents.

In this study, it was observed that teeth immersed in distilled water (control) had the highest debonding force, followed by teeth immersed in lemon EO, eucalyptus oil and the least force was required to debond brackets immersed in glutathione (Table 1). Though the force registered was similar for eucalyptus

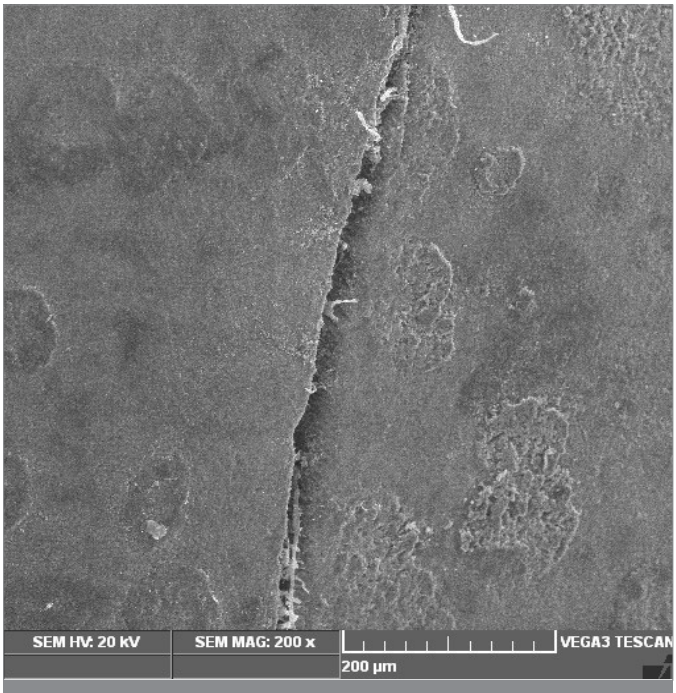

Figure 1. 200X magnification of control group showing crack propagation under SEM

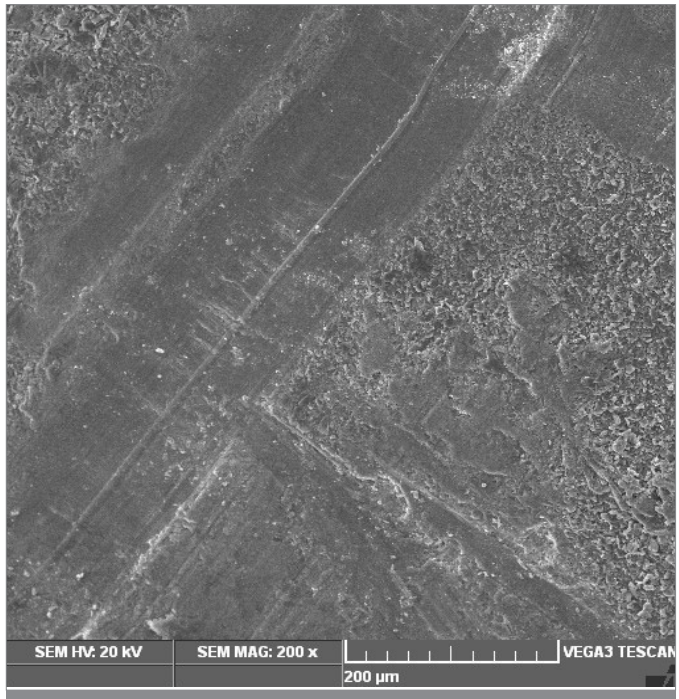

Figure 2. 200X magnification of eucalyptus oil group showing no cracks on enamel surface under SEM

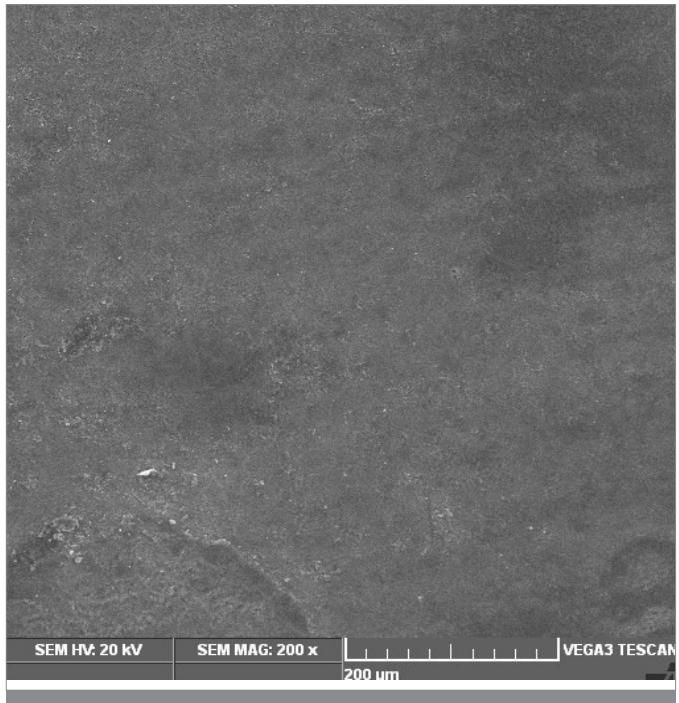

Figure 3. 200X magnification of glutathione group showing no damage to enamel structure under SEM

oil and glutathione group, there was a statistically significant reduction in the debonding force of brackets immersed in glutathione. Glutathione is a low molecular weight thiol and is one of the best antioxidants. It is probable that it enhanced marginal leakage between the tooth and cement interphase and thereby facilitated debonding of ceramic brackets.[18]

The specimen treated with eucalyptus oil had a mean debonding force of 56.48 N. Yu et al.[17] investigated the effect of eucalyptol on the debonding of metal and ceramic brackets and concluded that specimens immersed in eucalyptol gel for 10-15 in exhibited most reduction in shear bond strength. Larmour et al.[15] reported an immersion time of one hour in peppermint oil, whereas in this study, immersion time was reduced to a clinically acceptable level of ten minutes.[16]

A thorough analysis of various chemical compounds used in the formulation of EOs indicated a higher concentration of an organic compound 1-8 Cineole.[28] This may be responsible for lowering the shear bond strength of the composite resin by softening it. It can be the key ingredient in facilitating debonding of ceramic brackets, but this question requires extensive research. The mean debonding force of teeth immersed in lemon EO was 68.44 N, this value was the highest among the experimental groups. The effectiveness of lemon EO in debonding orthodontic ceramic brackets has not been previously studied; therefore, further research is necessary before a meaningful conclusion can be derived.

It was observed that 76% of specimens immersed in glutathione and 64% of specimens in eucalyptus oil had scores 0 in ARI, they did not have any remnant adhesive on enamel (Table 3). This result is more promising than the study by Devi Kanth et al.[29], where score 0 was observed for 60% of specimens that were treated with EO (peppermint oil) for 5 min before debonding. Lemon EO group had about 44% of the specimens with 50% of the adhesive still on the enamel surface. There was a statistically significant difference in the mean ranks of all groups (Table 4). Pairwise comparisons revealed no statistically significant difference between group II (eucalyptus oil) and group III (glutathione) (Table 5). Similarly, intergroup comparison of debonding force (Table 2) also did not reveal statistical differences between eucalyptus oil and glutathione, but ceramic brackets in the glutathione group exhibited peel-off effect, composite resin peeled off totally from the enamel surface (Figure 5). This observation was not evident in any other group. The ARI scores were lower in the experimental groups than in the control group, thus reflecting a reduction in resin retained on the enamel surface. The reason for the reduction in ARI scores can be an infiltration of EOs in enamel-adhesive interface, thus facilitating crack propagation, resulting in easy debonding of ceramic brackets.

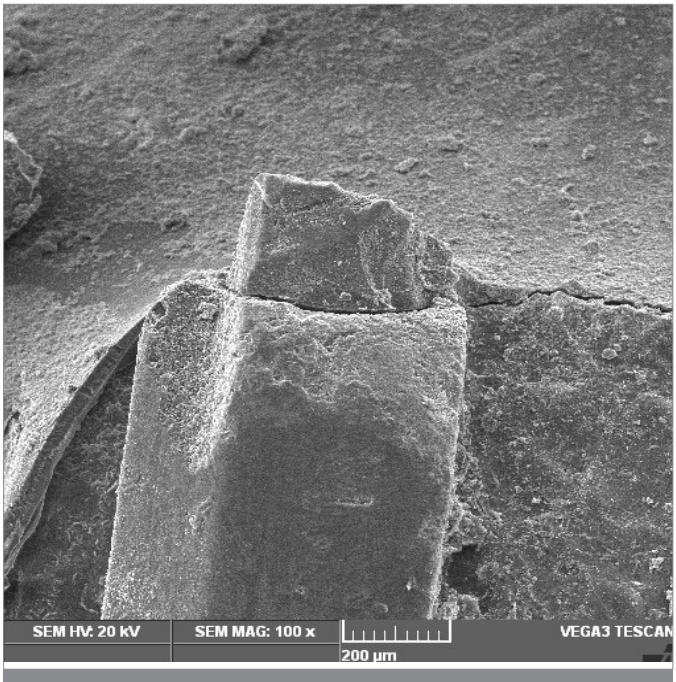

Figure 4. 100X magnification showing adhesive left behind on tooth surface after the use of lemon essential oil under SEM

Uysal et al.[30] evaluated the shear bond strength of ceramic and metal brackets and found that ceramic brackets bonded with the normal acid etch technique had the highest levels of shear bond strength (36.7MPa) among the groups investigated. Similar results were obtained in the research by Odegaard and Segner.[23] The mean values of shear bond strength obtained in this study were 11.56MPa in the control group and 5.37MPa, 4.81MPa and 6.51MPa in specimens immersed in eucalyptus oil, glutathione and lemon EO, respectively (Table 1). It was evident that, immersing the teeth bonded with ceramic brackets in EOs used in this study reduced the shear bond strength to a considerable extent. Thus, the null hypothesis was rejected. This finding was further reinforced by the ARI scores. In SEM images, the control group had a noticeable crack formation on enamel. Among the experimental groups, specimens immersed in eucalptus oil did not show any enamel damage but displayed minor surface irregularity, specimen in lemon EO group showed residual adhesive and the sample immersed in glutathione exhibited a smooth surface. There was no enamel pitting in the specimens in contrast to the results of study by El-Shourbagy and Ghobashy.[16]

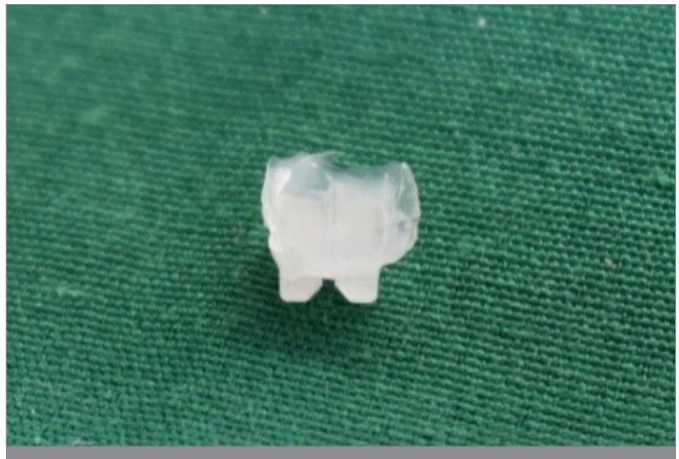

Figure 5. Peel off effect: entire composite is on the bracket surface after debonding of specimens immersed in glutathione (group III)

The findings indicated a definitive effect of softening resin when teeth bonded with ceramic brackets were immersed in chemical reagents. The antioxidant properties of glutathione might be the key reason for the excellent peel-off effect of the composite from the enamel surface, which can be considered beneficial as it would save clinical chair side time as well as eliminate the need to use rotary instruments to remove composite remnants. Glutathione holds promise as a better reagent compared with others, when the results of the three parameters tested are correlated.

Though this study was performed *in vitro*, further research with glutathione will improve the scope of using the same in day-to-day practice so that ceramic brackets can be debonded with ease, reducing chair side time, eliminating patient discomfort and obviating the chances of damage to surface enamel.

CONCLUSION

Immersion of teeth bonded with ceramic brackets in chemical reagents for a duration of 10 min reduced the debonding force, adhesive remnant on the enamel surface as well as prevented damage to the enamel structure compared to the control.

Of the three reagents used, specimens immersed in glutathione showed the highest reduction in debonding force that enabled easier debonding of ceramic brackets with peel-off effect. Thus, it can be concluded that immersion in glutathione for a duration of 10 min for debonding of ceramic brackets is a promising method to reduce debonding force, ARI scores and preventing damage to enamel surface.

Ethics

Ethics Committee Approval: This study was approved by the Scientific Review Board of SRM Dental College, Ramapuram, Chennai-89 (SRMDC/IRB/MDS/2018/No.108).

Informed Consent: Informed consent was obtained from patients before extraction of premolar teeth.

Peer-review: Internally peer-reviewed.

Author Contributions: Concept - S.M., P.J., R.K.; Design - S.M., P.J., R.K.; Supervision - S.M., P.J., R.K.; Funding - S.M.; Materials - S.M., P.J., R.K.; Data Collection and/or Processing - S.M., P.J., R.K.; Analysis and/or Interpretation - S.M., P.J., R.K.; Literature Review - S.M., P.J., R.K.; Writing - P.J., S.M., R.K.; Critical Review - P.J., S.M., R.K.

Declaration of Interests: The authors have no conflicts of interest to declare.

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