Research Article

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The Effect of Volatile Oils on Debonding of Polycarbonate Brackets Reinforced with Ceramic Filler: A Qualitative Study

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ABSTRACT

Introduction: *An effective debonding agent should be capable of reducing not only the debonding force, but also the amount of adhesive remaining with no enamel or bracket fracture. Aim: The aim of the present in vitro study was to evaluate the amount of the adhesive remnants, enamel fracture as well as bracket fracture after application of different volatile oils at different time of application using ARI system. Material and Method: Polycarbonate brackets reinforced with ceramic filler were bonded to one hundred and eighty (180) sound extracted human premolars using Transbond XT light curing adhesive. The teeth were then distributed into five groups. Teeth were then de-bonded using the Instron Universal Testing Machine. The enamel surface of debonded teeth and their bracket bases were examined under the stereomicroscope SEM for adhesive remnants (ARI), enamel fracture and bracket fracture. Result: At 5 minutes of application, no significant difference was noticed among volatile oils in all the qualitative results including; adhesive remnants, enamel fracture and bracket fracture. At 5 minutes of application, the peppermint volatile oil exhibited the highest percentage (55%) of score 1 whereas the clove oil revealed the least percentage (15%). At 30 minutes of application, the black seed exhibited the least percentage of score 1 and the highest percentage of score 2 (75%). At 5 minutes, the peppermint oil showed the least brackets fracture (20%) whereas the clove oil was in the second rank (25%) and the mixture of peppermint oil with black seed oil revealed the highest (55%). At 30 minutes, the clove oil showed the least bracket fracture (5%) whereas peppermint oil was in the second rank (20%) and the black seed oil depicted the highest percentage (45%). Conclusion: The present study supported that the 5- minutes of peppermint as well as the black seed volatile oil applications can be considered the best debonding agents whereas the clove oil can be used as a medication or mouthwash before debonding. The latter approach worth further investigations.*

Keywords

Volatile oil, Qualitative, Debonding, Remaining adhesive, Polycarbonate brackets.

Hight Light

To investigate if using volatile oils will reduce significantly the adhesive remnants after debonding of ceramic brackets without utilizing debonding burs.

Introduction and Literature Review

Most ceramic brackets are manufactured from aluminum oxide (alumina) particles, and present in polycrystalline and mono-crystalline forms [1].

Ceramic brackets are chemically stable (inert) in the oral environment and known for their hardness as it is harder than enamel [2,3].

This characteristic should be considered when there is contact between the enamel and ceramic. This is to avoid enamel damage. Hence, care must be performed with deep bite and/or Class II canine relationship and bite opening must be performed to prevent enamel damage [4].

Since, ceramic brackets do not flex; this makes that ceramic bracket are much more likely to fracture than metal brackets under the same conditions [5].

Further, it was reported that the chemical retention produced very strong bonds which will leads to cracks and enamel chipped off during bracket removal [2,5,6].

Furthermore, it is worth noted that the exposure of alumina to water or saliva decreases fracture toughness. This characteristic is important to remember when the researcher attempts to conduct *in vitro* study to the clinical oral environment [7].

The debonding problems of enamel and ceramic bracket fractures (esthetic brackets) continue to be of concern to clinicians.

However, few studies aimed in using chemical agents to soften the adhesive layer prior to debonding plier application. Larmour *et al.* [8] investigated the effect of application of peppermint volatile oil on debonding force and adhesive remnants one hour before debonding compared with two well-recognized softening agent acetone and ethanol when debonding ceramic brackets. They found that application of peppermint oil produced the lowest levels of retained resin and no evidence of enamel fracture with any of the groups, but bracket fracture remained a problem.

Winchester in 1992 carried out study on chemical agents that can contribute in easier mechanical debonding by applying peppermint oil and its derivatives around the bracket base and left for 2 minutes. He found that the peppermint oil facilitated bond failure at adhesive enamel interface without damaging the tooth surface [9].

Devikanth *et al.* [10] evaluated enamel surface characteristics site of failure and rate of bracket failure following debonding of ceramic brackets using different debonding techniques. They reported that chemical debonding technique though had bond failure at enamel-adhesive interface, SEM showed minimal enamel damage indicating it as better technique for debonding ceramic brackets.

On the other hand, Anita [11] conducted an *in vitro* study to determine the effect of 4 different debonding techniques using 3M ceramic bracket debonding plier, peppermint oil, diode laser and Er: YAG laser on enamel surface, and assess the remnant adhesive on the surface of brackets and also to determine the time taken to debond each bracket. He found that the Er. YAG laser debonding was the most effective, safest for the enamel surface and was least time consuming.

In 2017 Mohebi *et al.* [12] carried-out study evaluating the enamel surface roughness after orthodontic bracket debonding with atomic force microscopy. They compare the enamel surface roughness values after removal of the adhesive using 3 methods as well as the time needed to perform this procedure. The adhesive remnants were removed using a white stone bur, a tungsten carbide bur, or a tungsten carbide bur under loupe magnification. They concluded that the tungsten carbide bur is still recommended for composite removal.

However, Xiao-Chuan *et al.* [13] in his *in vitro* study to evaluate orthodontic debonding methods by comparing the surface roughness and enamel morphology of teeth after applying two different debonding methods and three different polishing techniques. They stated: “debonding pliers were safer than enamel chisels for removing brackets. Cleanup with One-Gloss polisher provided enamel surfaces closest to the intact enamel, but took more time, and Super-Snap disks provided acceptable enamel surfaces. The diamond bur was not suitable for removing adhesive remnant”.

AL-Bakry *et al.* [14] prepared non- toxic and safe composition comprising eugenol in gel form or as an “Emulgel” that showed an ability to reduce micro hardness of orthodontic adhesive bonding resins and that facilitated the safe removal of brackets as well as the removal of residual bonding resin from the enamel surface without damaging the enamel surface. Further, it was recommended to use the clove oil as a medication or mouthwash (safe and not toxic) for two days before debonding which reduced the debonding force and residual bonding resins. However, the latter approach might need further investigation.

As there were few studies on the effect of chemical agents on the debonding of ceramic brackets with no definite conclusion, it is the aim of the present investigation to study qualitatively the effect of different volatile oils on de-bonding of polycarbonate bracket reinforced with ceramic fillers and assess their effect on the number of adhesive remnants as well as enamel or bracket fracture after debonding.

Materials and Methods

Teeth Collection and Storage

Two hundred and forty (240) extracted human premolars were collected from adolescent patients undergoing orthodontic treatment. They were examined under a stereomicroscope (WILD Photo-makroskop M400, Switzerland) at 10X magnification to ensure the following:

Selection Criteria

a. Sound extracted premolars.
b. No caries, obvious defects, discolorations, or

Restorations that may affect the enamel strength.

c. Teeth with mild initial enamel crack were included but were recorded in the pre-operative records
Only one hundred and eighty teeth satisfied these

Selection criteria:

However, 130 were found to be without initial enamel cracks and the remaining 50 with initial enamel cracks.
The 180 selected teeth were then stored in distilled water. Each tooth was stored in a separate container, and the containers were then randomly numbered from 1 to 180.

Bonding Materials

a. Enamel conditioner: System 1+ (37%) phosphoric acid solution (740-0038, Armco. Corp. Glendora. California, USA).
b. Adhesive system: Transbond XT lightly filled light cure composite resin (15-17% mono- and di-methacrylateresin) (3M Unitek Corp., Monrovia, California, USA).
c. Brackets: premolar polycarbonate brackets reinforced with ceramic filler with (0.022) metallic slot and a mechanical retention base (Spirit MB, Ormoco Corp., Glendora, California, USA).

Debonding Materials

a. Debonding solvents: Clove oil (eugenol), Peppermint oil (menthol), Black seed volatile oil (Thymoquinone) and amixture of Peppermint and Black seed volatile oil were used as de-bonding agents. These solvents were pure extract from their seeds or plants. They were stored in tightly closed glass containers as they are volatile oils, and they can also affect plastic materials.

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b. Debonding instrument:
AEZ narrow blade debonding plier (803-0105, Ormoco Corp., Glendora, California, USA) was used (Figure 1).

The plier was mounted on an Instron Model TM universal testing machine (Instron 8500, England) by a customized jig (Figure 2).

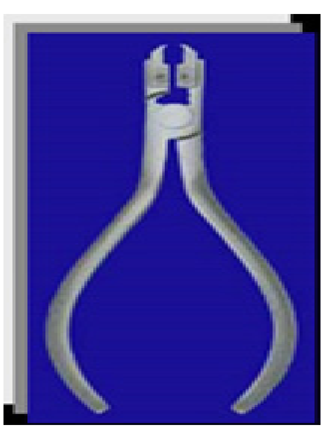

Figure 1: AEZ narrow blade debonding plier.

Figure 2: Debonding plier mounted on the Instron machine.

b. The buccal enamel surface of all teeth and 3 representative bracket bases were evaluated, before bonding, and photomicrographs were taken with the stereomicroscope at three magnifications: 10X(center)- 20X(center) -32X(center-mesial-distal-occlusalgingival) as a pre-bonding record.

c. A digital micrometer (Mitutoyo Digimatic micrometer, 29376530, 5-chome minato-Ku, Tokyo 108, Japan) was used to calculate the bracket base surface area of 20 randomly selected brackets. The average surface area of the bracket base was determined to be 10.6 mm2.

Method of Bonding

1) Enamel Surface Preparation The buccal enamel surface of each tooth was cleaned and polished with non-fluoridated pumice and rubber prophylactic cups for 15 seconds, rinsed with water spray for 10 seconds, and dried with oil-free compressed air for 10 seconds. Each buccal enamel surface was then etched with 37% phosphoric acid solution for 30 seconds according to the manufacturer's instructions. Then rinsed with water spray for 20 seconds and dried with oil-free compressed air for 20 seconds. All buccal enamel surfaces appeared chalky white in color after etching.

Bonding Procedure

Transbond XT Light Curing Adhesive was applied according to manufacturer's instructions. To ensure an equal adhesive thickness layer in all specimens, each bracket was then subjected to a 75-gm force using an articulator (Teledyne Hanau Series H2 & 145 Articulators, Buffalo, NY, USA).

This force was managed to be within the range of force used clinically by hand pressing to overcome having a very thin adhesive layer.

The articulator arm was kept pressing on the bracket till the excess bonding resin was removed from the edges of the bracket with an explorer.

a. The articulator arm was then removed, and the bracket was light cured for 20 seconds from the buccal surface according to manufacturer's instructions. This was performed using Ortholux XT Visible Light Curing Unit (3M Unitek Corp., California, USA).
b. The bonded teeth were then stored in distilled water at 37 °C oven (each in its container) for a period of one week to ensure complete polymerization of the adhesive resin before bond strength testing.

Method of Debonding

The teeth were divided into the following groups (Figure 3).

A drop of the below-mentioned solvents was injected on the buccal surface of its specified group of teeth in the interface between the bracket base and the adhesive. Each solvent was applied for its specified period immediately before de-bonding.

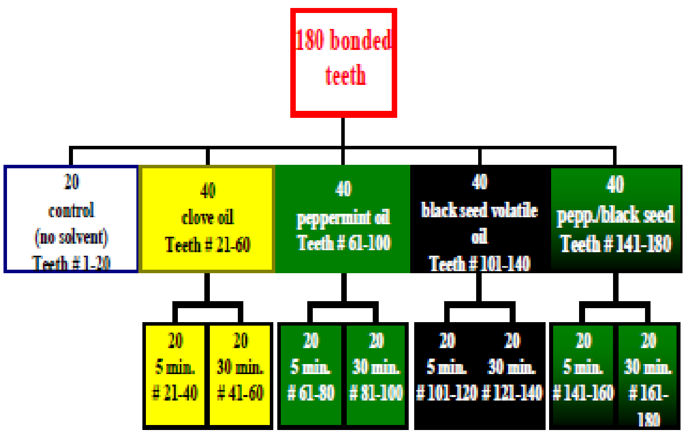

Fig. 3. Teeth distribution and solvents application.

Debonding Procedure

Each tooth was then debonded with the AEZ narrow blade de-bonding pliers mounted on the Instron Universal Testing machine. For consistency and to avoid slippage of the pliers on the Instron, the AEZ de-bonding plier was mounted on the Instron machine by a customized jig in its upper arm. While the lower arm of the pliers was kept, free so that a lower rod from the Instron with rounded tip will touch the lower arm at a concavity (in which the geometry of area of touch will not affect the inclination of force during compression). Thus, a bilateral load was applied on the pliers from the Instron machine (Figure 4).

This de-bonding technique represents the clinical
Situation. The bracket bonded to the tooth was then freely placed between the blades of the pliers in an occlusal-gingival direction at the bracket-adhesive interface (Figure 5).

The whole apparatus was then covered by a plastic sheet in a way that does not interfere with the movement of the Instron (Figure 6). The purpose of this plastic cover is to avoid any bracket loss after de-bonding for further assessment.

Figure 4: Compressive load applied by the Instron machine on the pliers arms.

Figure 5: Debonding pliers holding a tooth at the bracket-adhesive interface in an occluso-gingival direction.

The Instron machine slowly applied a squeezing (diametral) compressive force at a crosshead speed of 0.5 mm/min to the pliers at room temperature until bond failure occurred. The debonding plier was replaced after every 50 debonded Brackets, to assure blade sharpness (4 pliers were used).

The samples were randomly distributed among the pliers to reduce the effect of any difference in pliers mounting. The four pliers used, debonded equal number of samples (5 teeth) from each subgroup.

Figure 6: Plastic cover to prevent bracket loss.

The qualitative assessment

The qualitative assessment was to determine the amount of adhesive remnants and the presence of enamel and bracket fractures after debonding.

After the teeth have been debonded, they were further examined under a stereomicroscope (WILD Photo-makroskop M400, Switzerland) and confirmed by a scanning electron microscope (JSM-T330A scanning electron microscope, JEOL Ltd., Tokyo, Japan) on a representative sample from each sub-group.

Stereomicroscopic examination

The debonded teeth and bracket bases were re-examined after debonding and photomicrographs were taken with the stereomicroscope at 10X (center) -20X (center) -32X (center-mesial-distal-occlusal-gingival) magnifications.

The photomicrographs of the stereomicroscope were saved on CD. They were then analyzed through the computer using the ACD See version 3.0 software program specifically designed for photographic processing in the computer.

(a) Adhesive remnant assessment:

The residual adhesives remaining on the buccal enamel surfaces were recorded using the Adhesive Remnant Index (ARI) developed by Artun and Bergland14 as follows:

Score 0 = no retained resin.

Score 1 = > 0% - <50% retained resin on the enamel surface

Score 2=>50%- < 100%retained resin on the enamel surface

Score 3= all resin retained on the enamel surface with bracket imprint.

(b) Enamel and bracket fractures' assessment:

The presence of enamel and bracket fracture was assessed by comparing the photomicrographs of the stereomicroscope before bonding and after debonding. Comparing the pre- and -post debonding records is the best method to assure that any enamel damage was a result of debonding and not due to an original crack or developmental defect present on the enamel surface.

Scores for enamel fracture were:

Score 0: no enamel fracture,

Score 1: presence of enamel fracture.

Scores for bracket fracture were:

Score 0: no bracket fracture,

Score 1: presence of bracket fracture.

Compared to the bracket base before bonding (Figure 7), the deboned bracket bases were recorded as fractured when part of the base is fractured (Figure 8) and not just the tiny mechanical projections, which were unavoidable (Figure 9).

Figure 7

Figure 8

Bracket base before de-bonding (Figure 7) and after (Figure 8)

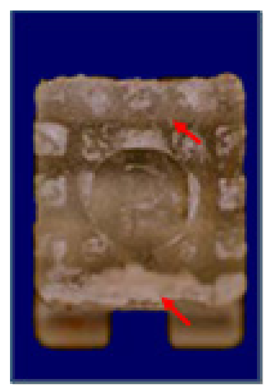

Figure 9: Un-fractured bracket base after debonding. **Red arrows** represent fractured tiny mechanical projections.

Scanning electron microscopic (SEM) examination

A random sample of 5 teeth from each sub-group with scores of 0, 1 or 2 based on the ARI system were further evaluated using SEM.

The buccal surfaces of the selected teeth and their companion bracket bases were set to dry. They were then mounted on copper stub by conductive carbon paints, with the buccal surface and the bracket base facing upwards, and sputter coated with gold in fine coat ion sputter (JFC-1100, JEOL Ltd., Tokyo, Japan). The failure surfaces were then examined under the SEM with 25KV at

15X and 35X magnifications and photomicrographs were taken. The results recorded from the stereomicroscopic examination were then confirmed with the photomicrographs of the SEM. Although the SEM gave better view, it was impossible to take a pre-bonding and after debonding photograph for the same tooth with the SEM machine used in the present study. This is because it must be gold coated before examining it. Thus, stereomicroscopic photomicrographs were important especially for pre-bonding records.

The higher magnifications and detailing of the photomicrographs of the SEM especially in examining the enamel and bracket fractures have drawn the attention of the examiner to re-examine the stereomicroscopic photomicrographs of all the teeth and bracket bases to ensure proper assessment. It was found that 15X magnifications with the SEM gave almost similar picture to 32X magnifications with the stereomicroscope as shown in (Figures 10 and 11).

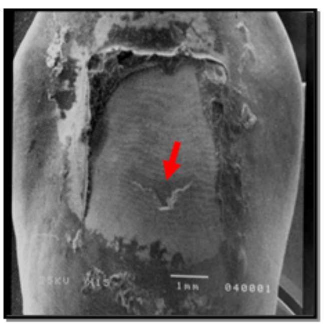

Figure 10

Figure 11.

Figure 10: SEM photomicrograph at 15x magnification showing a buccal enamel surface with enamel fracture (red arrow).

Figure 11: Stereomicroscopic photomicrograph of the same tooth at 32x magnification showing the same enamel fracture (red arrow).

Intra-examiner reproducibility

The examiner viewed the photomicrographs of the stereomicroscope and SEM four times to ensure reproducibility. The results of the ARI, and the presence of enamel and bracket fractures were then recorded.

Assessment of measurement error

For intra-examiner reproducibility, the examiner re-evaluated a random sample of 36 teeth and their bracket bases (4 from each sub-group) after two weeks from the first assessment. The ARI and the presence of enamel and bracket fractures were recorded again.

Statistical Analysis

Assessment of measurement error

The assessment of the adhesive remnants on the enamel surface after debonding (using the adhesive remnant index, ARI system) and the presence of enamel and bracket fractures were subjective. Thus, a measurement of agreement test for categorical data (Kappa statistic) was applied to assure intra-examiner reproducibility.

Qualitative assessment:

By cross-tabulation, descriptive analysis by percentage was performed for different types of volatile oils (solvents) and at different times of application. The variables assessed were; the amount of the adhesive remaining on the enamel surface (ARI), and the presence of enamel and bracket fractures. For statistical inferences, the following was performed for each qualitative variable:

Table 1. The frequency and percentage distribution of the combined ARI scores, the enamel and the bracket fracture at the two times of application.

Groups	***Time (min)***	***Sample size***	***Combined ARI Scores*** (0+1) 1	(2+3) 2	***Enamel fracture***	***Bracket fracture***
Control	5	20	7 35%	13 65%	3 15%	8 40%
Clove oil	5	20	3 15%	17 85%	0 0%	5 25%
	30	20	10 50%	10 50%	4 20%	1 5%
Peppermint oil	5	20	11 55%	9 45%	2 10%	4 20%
	30	20	11 55%	9 45%	2 10%	4 20%
Black seed volatile oil	5	20	5 25%	15 75%	1 5%	8 40%
	30	20	1 5%	19 95%	0 0%	9 45%
Peppermint + Black seed volatile oil	5	20	5 25%	15 75%	3 15%	11 55%
	30	20	8 40%	12 60%	3 15%	5 25%

A. Adhesive Remnant Index (ARI)

Scores 0 + 1 were combined as score 1 which represents minimum adhesive remnant. Scores 2 + 3 were also combined as score-2, which represents excessive adhesive remnant. Then, the Chi-square test was applied for different volatile oils (solvents). The test was performed for each solvent at different times of application.

Presence of enamel fracture

The exact distribution of permutation statistic was applied using the StatXact-5 software program to test the statistical difference among different solvents at both times of application.

Presence *of bracket fracture*

The Chi-square test was used to test the statistical difference among different solvents at both times of application.

Results

Assessment of measurement error

Kappa statistic (measurement of agreement test for categorical data) was conducted to test the intra-examiner reproducibility in determining; (i) the amount of adhesive remaining on the enamel surface after debonding using the adhesive remnant index (ARI system), (ii) the presence of enamel fracture and, (iii) the presence of bracket fracture. The result showed very good agreement level (>80%) in measuring the above-mentioned qualitative variables.

Qualitative measurements

Table 1 showed the frequency and percentage distribution of the combined ARI scores as well as the enamel and the bracket fracture at the two times of volatile oils application.

Adhesive remnants

In Figure 12, at 5 minutes of application, the peppermint volatile oil exhibited the highest percentage (55%) of score 1 whereas the clove oil revealed the least percentage (15%) of score 1. On the other hand, the peppermint showed the least percentage of score 2 (45%) whereas the clove oil depicted the highest percentage (95%). No significant difference (Pearson Chi-square = 0.072) was observed between the different solvents (Table 1).

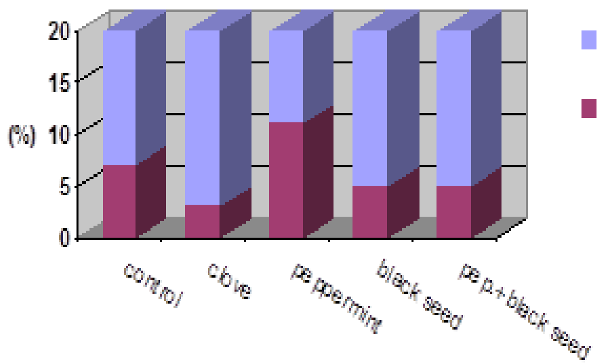

Figure 12: The percentage distribution of ARI scores for each solvent at 5 minutes of application (Pearson Chi-square = 0.072).

In Figure 13, at 30 minutes of application, the black seed exhibited the highest significant percentage of score 2. (Pearson Chi-square = 0.005) compared to other volatile oils. On the other hand, the peppermint and clove oils demonstrated the highest percentage of score 1 (75%) (Table1).

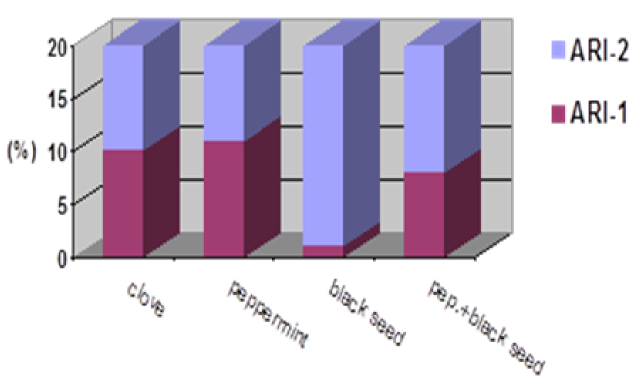

Figure 13: The percentage distribution of ARI scores for each solvent at 30 minutes of application. (Pearson Chi-square = 0.005).

Enamel fracture

Figure14, at 5 minutes of application: the clove oil showed no enamel fracture (0%) whereas the black seed oil was in the second rank (5%) and the mixture of black seed oil with peppermint oil demonstrated the highest percentage. (15%). No significant difference was reached between the groups. (P-value = 0.922) (Table1).

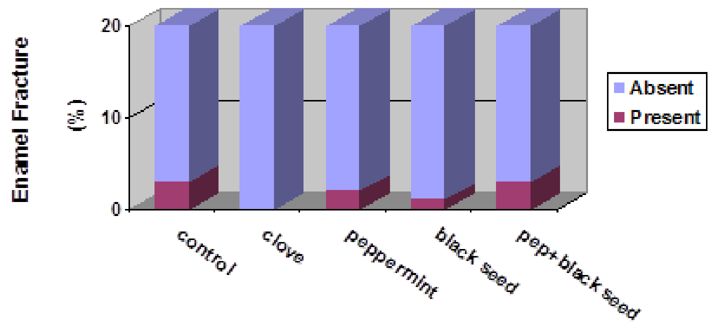

Figure 14: The percentage distribution of enamel fracture among the different solvents at 5 minutes of application. (P-value = 0.922).

Figure 15, at 30 minutes of application: the black seed oil does not exhibit enamel fracture whereas the peppermint was in the second rank (10%). On the other hand, the clove oil demonstrated the highest percentage of enamel fracture (20%). No significant difference (Pearson Chi-square = 0.922) was noticed between the different solvents (Table 1).

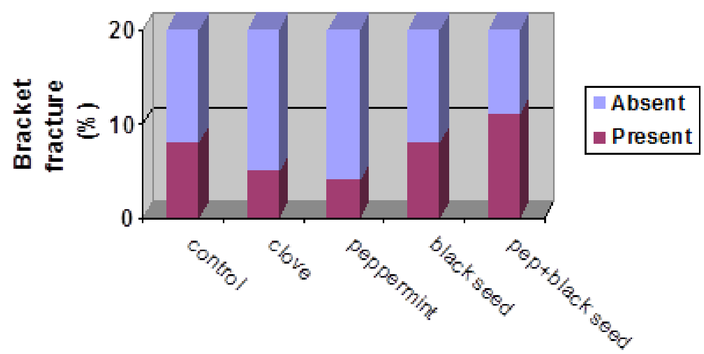

Figure 15: The percentage distribution of enamel fracture among the different solvents at 30 minutes of application P-value = 0.9222).

Bracket fracture

Figure 16, at 5 minutes of application, the peppermint oil showed relatively the least brackets fracture (20%) whereas the clove oil was in the second rank (25%). On the other hand, the mixture of peppermint oil with black seed oil revealed relatively the highest (55%) and no significant difference was observed between the groups. (Pearson's Chi-square = 0.154). (Table 1).

Figure 17, at 30 minutes of application, the clove oil showed relatively the least bracket fracture (5%) whereas peppermint oil was in the second rank (20%) and the black seed oil depicted relatively the highest percentage (45%) of brackets fracture. No significant difference was reached between the groups. (Pearson's Chi-square =.029) (Table 1).

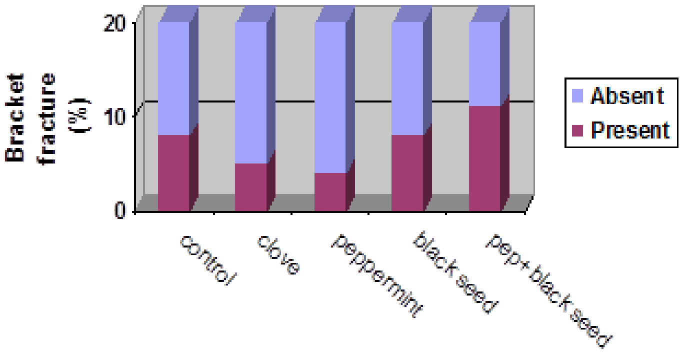

Figure 16: The percentage distribution of bracket fracture among the different solvents at 5 minutes of application (Pearson's Chi-square = 0.154).

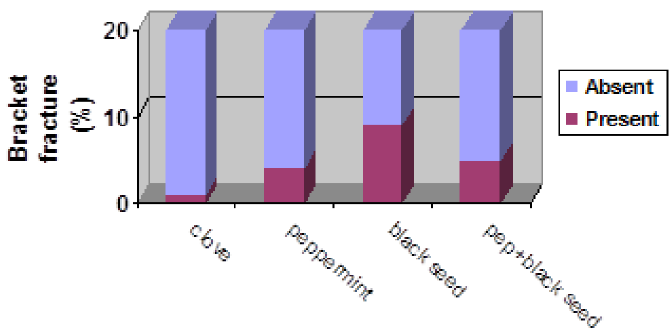

Figure 17: The percentage distribution of bracket fracture for each solvent group at 30 minutes of application (Pearson's Chi-square = 0.029).

Discussion

The main goal of the orthodontist when removing the adhesive remnants is to obtain an intact enamel surface as possible [15]. Hence, when choosing an effective debonding agent it should be capable of reducing not only the debonding force, but also the amount of adhesive remaining after debonding and addition to that avoiding enamel or bracket fracture. Thus, investigating the quantitative and qualitative results for each volatile oil is significant in determining the best debonding agent. However, the present study indicated that no significant association was noticed between the quantitative and the qualitative results except for the clove oil versus bracket fracture (Table 2).

This agreed with the findings of O'Brien *et al*. [16] who found that no statistically significant differences observed between the shear bond strengths at the adhesive/enamel and adhesive/bracket base interfaces. They stated, "These results suggest that the number of residual debris following removal of the bonded bracket is not related to the shear bond strength at the separate interfaces but is governed by factors caused by bracket base design and properties of the adhesive used".

Table 2. The association between the mean debonding force and the different qualitative variables using the independent t-test. (P<0.05)

Group	Mean force (Mpa) *versus* ARI-2	Mean force (Mpa) *versus* Enamel fracture	Mean force (Mpa) *versus* Bracket fracture
Control	0.637	0.409	0.019 S
Clove oil	0.106	0.740	0.003 S
Peppermint oil	0.171	0.341	0.555 NS
Black seed oil	0.441	0.093	0.846 NS
Peppermint + Black seed oil	0.757	0.932	0.098 NS

Montassera and Drummond [17] evaluated the reliability of the Adhesive Remnant Index (ARI) Score System with different magnifications with the naked eye, under 10x Mag, and under 20x Mag. They found that higher magnification offers accurate evaluation of adhesive remnant and recommended further investigation to reach standard magnification. However, in the present study the debonded teeth and bracket bases were examined under the stereomicroscope at 10X (center) -20X (center) -32X (center-mesial-distal-occlusal-gingival) magnifications.

The ARI score system has proved to be of great value in studies of orthodontic adhesive systems. This is since it is a quick and simple method, which does not need special equipment's. However, its reliability requires investigation, with special attention on the effects of magnification on evaluation of the adhesive remnant [18].

Studies testing the reliability of the ARI score system were uncommon. However, Oliver [18] conducted study on the bond strength of orthodontic brackets to check the reliability of the ARI system. He reported that inter-observer and intra-observer variability is low when the ARI system is used although the reliability was assessed under the same magnification. In the present study Kappa statistic (measurement of agreement test for categorical data) was conducted, and the result showed very good agreement level of (>80%) ARI Score in measuring the qualitative variables. Few studies [8,14] aimed at using chemical agents to soften the adhesive layer prior to debonding. The debonding problems of enamel and ceramic bracket fractures remain to be of concern to clinicians. However, no definite conclusions on the effect of chemical agents on the debonding of ceramic brackets have been established.

Since there is an interaction between the solvents' application and the time of application, the results will be discussed based on the time of application. The qualitative result at 5 minutes of application indicated that no significant difference was observed between the different volatile oils with respect to the number of adhesive remnants, enamel fracture and bracket fracture.

However, at 30 minutes of application, the black seed volatile oil revealed significantly the highest percentage of bracket fracture and the highest percentage of excessive adhesive remnants (ARI-2) compared to the other volatile oil's groups. Contrary, the clove oil showed significantly the least percentage of bracket fracture at 30 minutes compared to the other groups. On the other hand, no significant difference was recorded between the volatile oils and the presence of enamel and bracket fracture at 5 -minutes. This agrees with the result obtained by Hajrassie and Khier [19] who conducted an in-vivo and in-vitro comparison of bond strengths of orthodontic brackets bonded to enamel and debonded at various times and reported that bond strength values are not time dependent.

Therefore, the criteria for the selection of the best volatile oil as a debonding agent should have an acceptable application time, acceptable debonding force, minimal or/ no adhesive remnants (ARI-1) and no enamel fracture and no/or minimal bracket fracture. Fulfilling all these criteria simultaneously remains a challenge. However, within these criteria the mixture of the peppermint volatile oil with the black seed volatile oil was excluded, whereas the peppermint oil, clove oil, and black seed volatile oil worth further investigation and comparison.

From clinical point of view, the 5 minutes of application is more clinically and practically acceptable. Accordingly, at this period, the results of the present study revealed that the peppermint and the black seed volatile oils when used separately gave the best results and were considered as the best debonding agents in addition to clove oil.

In the present study, the procedure of applying the peppermint, black seed and clove was different and in oil forms and applied once and only for either 5 minutes or 30 minutes, which is from clinical point of view, is acceptable. However, in AL-Bakry *et al*. [14] the eugenol was in the form of gel form. However, they did mention the time of application of the eugenol in the gel form or as an "Emulgel; (Derivative of Clove oil) ranging at least 5 to 60 minutes or 1 to 24 hours and sometimes reapplied once or more than once at intervals.

As mentioned earlier; peppermint volatile oil was the only solvent that was studied previously by Larmour *et al.* [8]. They investigated the effect of application of peppermint volatile oil on debonding force and adhesive remnants one hour before debonding compared with two well recognized softening agent acetone and ethanol when debonding ceramic brackets. In addition, found that the application of peppermint oil produced the lowest levels of retained resin and no evidence of enamel fracture with any of the groups. The same observation was reported by Winchester in 1992 that peppermint oil facilitated bond failure at adhesive enamel interface without damaging the tooth surface [9].

However, the comparison between our study and Larmours *et al.* [8] study differ in several variables, the most being: (1) the conditioner –adhesive – bracket combination; (2) the type of loading (shear, using debonding plier); (3) the configuration of specimen testing jig; (4) the crosshead speed of mechanical testing machine; and (5) the significance level. (Table 4). Thus, a valid comparison between the two studies is limited, if not impossible. The only worthwhile comparison is to compare within each group in the same study e.g to investigate the effect of peppermint oil at different times of application and compared to the control group of the same study.

In Larmour's study [8], the application of peppermint oil gave more favorable qualitative results than the control group, regardless of the time of application. The same finding was observed in the present study at both time of application. Further, in previous work conducted by Winchester [9], Larmour and Chadwick [20] as well as Waldron and Causton [21] reveal that peppermint volatile oil acts as a crazing agent facilitating crack propagation when debonding ceramic brackets. The same observation was noted in the present study.

However, it is worth noting that; not only the number of adhesive remnants on the enamel surface that is important, but also the hardness of the adhesive is of equal importance. A soft easy to remove adhesive is more favorable to accept than a hard one, even if it is slightly less in amount. This point, which was not investigated in the present study, but our incidental observations showed that clove volatile oil had a clear softening effect on the adhesive and the ceramic bracket itself (Figure 18).

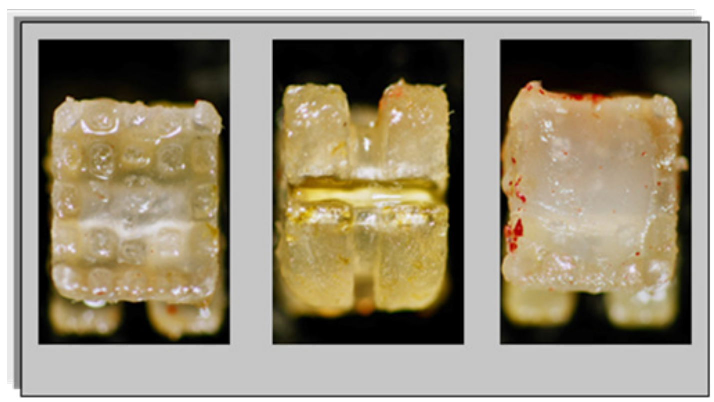

Figure 18: Effect of clove oil on the adhesive resin using a polycarbonate bracket reinforced with ceramic filler.

This observation is in line with the earlier finding of Powell and Hugest [23] and AL-Bakry *et al*. [14]. On the other hand, peppermint volatile oil work differently by increasing or at least did not change the hardness of the adhesive and the ceramic bracket (Figure 19).

The same findings were noticed by Waldron and Causton [21] and Larmour and Chadwick [20] who reported that peppermint volatile oil did not significantly affect the micro-hardness of concise composites except after 180 seconds application. This finding was observed in the present study when applying black seed volatile oil. (Figure 14).

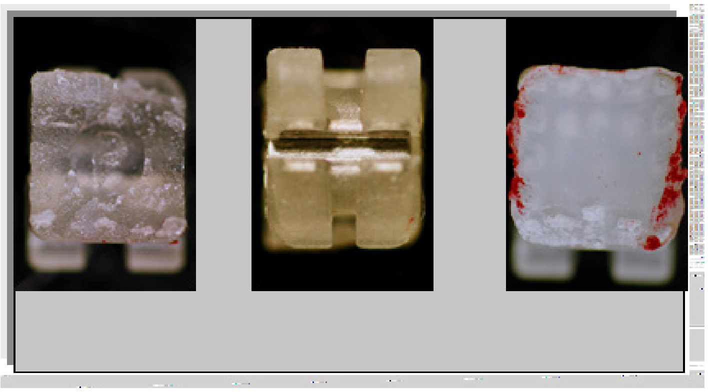

Figure 19: Effect of peppermint oil on the adhesive resin using a polycarbonate bracket reinforced with ceramic filler.

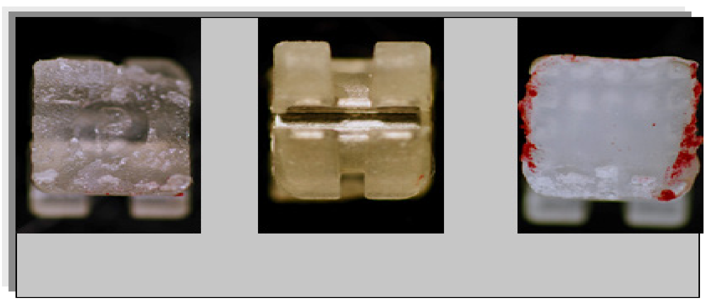

Figure 20: Effect of black seed volatile oil on the adhesive resin using a polycarbonate bracket reinforced with ceramic filler.

Extrapolation of laboratory data to the clinical situation should always be undertaken with care. This is because of the complexities of the oral environment. Therefore, it is reasonable to suggest that the comparison between standardized *in vitro* studies could be extrapolated to predict clinical results and that laboratory testing could be used as a screening mechanism for predicting clinical performance [22].

Finally, the present investigation and the few previous works on chemical solvents opened the door for a new challenging area of research that awaits further investigations. It will be highly appreciated to find a debonding agent that can be utilized as an adjunct during mechanical debonding of ceramic brackets, decreasing the force required for bracket removal upon which enamel surface damage and bracket fracture will hopefully no more be a clinical problem. [8,17,18,20].

Further, it is advisable to follow the manufacturer's recommendation method of debonding each bracket type and that the risk can be decreased by assessing the patient dentition. This can be performed by avoiding heavily restored teeth with pre-existing enamel cracks [8].

Conclusions

Within the limitations of the present *in vitro* study, the following conclusions may be drawn:

1. At 5 minutes of application, the peppermint oil showed the highest percentage of ARI-1 whereas in case of enamel and bracket fracture was located in the second rank compared to the other volatile oils.

2. At 5 minutes of application, the clove oil revealed significantly the least percentage of bracket fracture compared to the other groups. The highest percentage of score 2 and none enamel fracture.

3. At 30 minutes of application, the black seed volatile oil showed significantly the highest percentage of bracket fracture, none enamel fracture and the highest percentage of score ARI-2 compared to the other volatile oil's groups.

4. The present study supported the assumption that the 5- minutes of peppermint volatile oil applications can be considered as the best debonding agents, and that clove oil can be used as a medication or mouthwash before debonding. However, the latter approach might worth further investigations.

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