The Effect of Volatile Oils on De-Bonding of Polycarbonate Bracket Reinforced with Ceramic Filler: A Quantitative Study (Part 1)

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Abstract

Introduction: The introduction of esthetic brackets is a new clinical approach in the field of orthodontics. Two main types of esthetic brackets have been developed and became widely available: the polycarbonate brackets, and the ceramic brackets. Both types have faced two significant de-bonding problems: (1) enamel and bracket fracture. The two problems have been well documented and continue to be of concern to clinicians.

Aim: The aim of this in vitro study was to evaluate quantitatively the effect of three volatile oils and their combinations on the de-bonding of ceramic brackets.

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 the following groups according to the type of volatile oil and period of application: clove oil (5 & 30 minutes), peppermint oil (5 & 30 minutes), Black seed volatile oil (5 & 30 minutes), and mixture of peppermint + Black seed volatile oils (5 & 30 minutes) in addition to control group. The brackets were then de-bonded using the Instron Universal Testing Machine and the de-bonding force was recorded for each type of the volatile oils in different times of application.

Result: The result revealed that the Black seed volatile oil gave the lowest de-bonding force in 5 minutes and 30 minutes of application whereas the mixture of the Black seed oil + peppermint oils gave the highest force in both time of application. Further, the peppermint oil application for 5-minute was found to be in the second rank whereas the Clove oil was in the third rank. Furthermore, in the 30 minutes of application the Clove oil was in the second rank whereas the peppermint was found to be in the third rank and the combination of Black seed oil + peppermint oil shows the highest debonding force.

Conclusion: Despite the limitation of the present study result, an assumption has been drawn from the results obtained that (1) the 5-minutes of black seed or peppermint volatile oils application can be considered the best debonding agent compared to the other volatile oils; (2) the clove oil can be used after de-bonding to facilitate the final cleansing-up of enamel surface. To confirm the latter assumption, further investigations are needed.

Keywords: Debonding, polycarbonate bracket reinforced with ceramic filler, quantitative study, tensile force, volatile Oils.

I. INTRODUCTION

The introduction of esthetic brackets has generated a new clinical approach in the field of orthodontics. Two main types of esthetic brackets have been developed and became widely available: the polycarbonate brackets, and the ceramic brackets. However, ceramic brackets are produced from aluminum oxide (alumina) particles, and these brackets are available in polycrystalline and monocrystalline forms. However, these brackets are expensive due to the difficulty of milling, i.e., the cutting process1. Further, both types of brackets suffered two significant de-bonding problems: [1] enamel fracture and, [2] bracket fracture. These two problems have been well documented in the literature and continue to be of concern to clinicians. Besides, the color stability of ceramic brackets throughout orthodontic treatment remains to be an important characteristic.

Several in vitro studies reported that ceramic brackets, both monocrystalline and polycrystalline, undergo a color...
change when subjected to coffee, black tea, coke, and red wine [2]-[4].

Three mechanical bracket removal techniques were used [5]. The first technique uses a lift-off de-bracketing Instrument (LODI) where the pistol-grip plier is placed over the bracket. The force will be applied to the bracket tie-wings. However, it was reported that the LODI cannot be used when removing ceramic brackets due to their brittleness [6]. Recently, different types of lasers have been used for the removal of ceramic brackets but it has been reported that the laser application leads to softening of the adhesive material [7].

The ultimate tensile strength is defined as the maximum stress that a material can withstand while being stretched or pulled before failing or breaking [8].

Graber stated “the elongation of ceramics at failure (brittle fracture) is less than 1%, yet the elongation of stainless steel at failure (ductile fracture) is approximately 20%. Hence, ceramic brackets do not flex, and this implies that ceramic brackets are much more likely to fracture than metal brackets under identical conditions [9].

Several attempts were made focusing on reducing the force applied during the de-bonding of esthetic brackets. Both bonding and de-bonding procedures were found to affect these forces. Some of these attempts investigated the factors related to the bonding procedure such as:

1. Reducing the length of acid-etching [10]-[12].
2. Altering the type of etch including poly-acrylic acid crystal growth [13]-[15].

Despite these extensive investigations these attempts failed in reducing the de-bonding force to the range recorded with metal brackets. Besides, enamel and bracket fracture on de-bonding remained a problem.

Equally important, other attempts investigated the factors related to the de-bonding procedure. Several new techniques of de-bonding esthetic brackets have been designed to reduce the clinical incidence rate of irreversible enamel surface damage during de-bonding, but with limited success. These methods included:

1. The ultrasonic method that used special tips [16],
2. The electro thermal method that involved an apparatus which transmitted heat to the adhesive through the bracket [17] and
3. Laser de-bonding [18]. Although these new techniques have been advocated, mechanical de- bonding of esthetic brackets with sharp-edged pliers remained the technique of choice [1], [19], [20].

Based on that, no wonder, therefore that several researchers directed their efforts towards techniques that might aid mechanical de-bonding by weakening the resin/enamel bond prior to the application of the de-bonding pliers. To achieve that, the following have been attempted:

(a) addition of plasticizers to orthodontic bonding resin [21]
(b) notchting of orthodontic bonding resin [22] and
(c) chemical softening of orthodontic bonding resin [23]-[25].

Under the latter approach, two chemical agents based on peppermint oil were marketed for use as a de-bonding agent. It has been reported that a 60-second application of peppermint oil facilitated the removal of ceramic brackets and also helped in removing the residual resin from the enamel surface [1], [26]. However, [25] found that a significant softening effect was found only when peppermint oil was applied to orthodontic (Conico) bonding resin for a longer period (180 seconds). Other chemical agents such as ethanol or acetone have also been assessed for their effect on the de-bonding of ceramic brackets [26].

In addition, clove oil was also found to be incompatible with self-curing composite resins. Additionally, the bases and liners containing eugenol were found to be affected significantly the hardness of a conventional, chemically cured resin, chemically cured micro-filled resin, and light-cured resins [26].

Reference [27] stated “Within the limitations of this study, even though ceramic brackets required significantly higher debonding force compared to metal brackets, debonding stress was limited to the bonding site and did not affect the surrounding cracks on enamel”. However, for ceramic brackets, the value was significantly higher than for metal brackets and is comparatively higher than the value range of previous studies (from 10.4 ± 4.1 MPa to 21.67 ± 5.19 MPa14-17). The explanation for such high shear strength in this study may be the fact that flattened enamel may expose more enamel rods and thus, improve the bond quality [28], [29].

Various methods to de-bond metallic and ceramic brackets have been described in the literature, including the use of special debonding pliers, ultrasound or laser application, electro-thermic debonding, special instruments, and the use of bonding materials presenting thermo-expandable microcapsules to facilitate debonding [19]-[21], [25].

However, relatively very few published studies on this topic when compared to other areas of orthodontic research. Thus, the aim of the present investigation was to study the effect of several volatile oils on de-bonding of polycarbonate bracket reinforced with ceramic fillers that may be of clinical significance to the orthodontist.

II. MATERIAL

A. 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:

1) 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 according to [30] protocol. Each tooth was stored in a separate container, and the containers were then randomly

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numbered from 1 to 180.

2) 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-methacrylate resin) (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).

3) Debonding Materials

a. Debonding solvents: Clove oil (eugenol), Peppermint oil (menthol), Black seed volatile oil (Thymoquinone) and a mixture of Peppermint and Black seed volatile oil were used as de-bonding agents.

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

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

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-occlusal-gingival) 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.

B. 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.

C. 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. However, few studies have stated the amount of load applied during bonding, but all with a much higher load. Among those [21] who used 250 gm of load measured by a Correx force indicator. 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, according to [30] protocol.

D. Method of Debonding

The teeth were divided into the following groups (Fig. 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.
In the group of mixture of the peppermint and black seed volatile oil, one drop of each solvent was applied to the buccal surface of each tooth.

![Image](image-url)

Fig. 3. Teeth distribution and solvents application.

**E. 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 (Fig. 4).

![Image](image-url)

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

This de-bonding technique represents the clinical situation and was recommended by [2], [19]. 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 (Fig. 5).

The whole apparatus was then covered by a plastic sheet in a way that does not interfere with the movement of the Instron (Fig. 6). The purpose of this plastic cover is to avoid any bracket loss after de-bonding for further assessment. 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 de-bonded brackets, to assure blade sharpness (4 pliers were used). This was recommended by [23]. The samples were randomly distributed among the pliers to reduce the effect of any difference in pliers mounting. The four pliers used, de-bonded equal number of samples (5 teeth) from each subgroup.

![Image](image-url)

Fig. 6. Plastic cover to prevent bracket loss.

**F. Quantitative Assessment**

The aim of the quantitative assessment was to measure the force needed to de-bond the bracket. The force load was automatically recorded from the Instron machine to the computer and presented in Newtons (N).

**G. Statistical Analysis**

The descriptive analysis for the actual de-bonding forces were calculated and recorded in Newton (N).

One-way analysis of variance (ANOVA) and Tukey’s tests were performed to test the effect of solvent type on the de-bonding force. Further the two-way analysis of variance and the independent Student t-test were performed to test the effect of different solvents at different times of application. Further, the two-way analysis of variance and Scheffe’s test were performed to test the effect of the time of application on the de-bonding force among the different volatile oils.

Survival analysis was also carried out and this relates the probability of bond failure (de-bond) to the load applied. The use of this probability function analysis in bond strength testing has been advocated previously by [20]. It gives a prediction of the performance of the material at any level of stress which can be related to the clinical situation [24]. Furthermore, the Wilcoxon test was applied for statistical infer.

**IV. RESULTS**

**A. Descriptive Analysis of the De-Bonding Forces**

The de-bonding forces (in Newton, N) for all experimental groups were measured. The black seed volatile oil showed the lowest de-bonding force at five and thirty minutes whereas the mixture of peppermint and black seed volatile oils showed the highest force at both times. (Table I).
The effect of solvents and their application times on the de-bonding force:

The two-way analysis of variance and Scheffe’s test were performed to test the effect of the time of application on the de-bonding force among the different volatile oils.

The results presented in Fig. 7, revealed that 30 minutes application of peppermint volatile oil needs higher de-bonding force compared to the five minutes. On the other hand, the black seed volatile oil and the mixture of the peppermint and black seed volatile oils showed that the amount of force required to de-bond was almost the same at both time of application. The Scheffe’s test revealed that the black seed volatile oil applications at 5 minutes of application gave a significantly lower mean de-bonding force compared to the other volatile oils. Also, at 30 minutes application, the black seed volatile oil application exhibited the least mean de-bonding force compared to the other volatile oils.

Using the ANOVA test, it was found that there is a significant difference in the de-bonding force between the different volatile oils.

The Tukey’s test indicated that the black seed volatile oil recorded the lowest significant de-bonding force (67.45 N), compared the other volatile oils. The clove oil and peppermint oil were in the second rank with no significant difference in the mean de-bonding forces of 83.52 N and 89.13 N respectively. The highest de-bonding forces (with no significant difference) were found in both the mixture of peppermint and black seed volatile oils and the control with Mean de-bonding forces of 106.68 N and 106.72 N respectively (Fig. 8).

The survival analysis relates the probability of bond failure (de-bond) to the load applied. It is characterized by the Weibull modulus and the characteristic level (in this case the characteristic de-bonding force (N)). The Weibull modulus expresses the spread of the data, whereas the characteristic de-bonding force is a normalizing parameter that corresponds with the mean or median de-bonding force for a Gaussian distribution. Since the survival analysis is applied mostly to skewed data, the median is a more appropriate indicator than the mean. However, both medians and means were close to each other in the present study.

The probability of failure at 105 N was determined for each group as this approximated to the median de-bonding force required to de-bond the control group (106.72 N). It is also presented graphically in Fig. 9 and consists of the cumulative probability of bond failure.

Further, Wilkoxon test was also applied to test the significant difference between different volatile oils. The result indicated that there was a significant difference in the median de-bonding force among the volatile oils (P-value = 0.0001). Furthermore, Wilkoxon test was also applied to test the significant difference between the two times of application for each volatile oil. The result showed that, only the clove oil and peppermint oil were significantly affected by the time of application (Table II).
TABLE II: WILCOXON TEST FOR THE SIGNIFICANT DIFFERENCE BETWEEN THE TWO TIMES OF APPLICATION FOR EACH VOLATILE OIL (at p<0.05)

| Solvent*Time | Chi-Square | DF | P Value | Sig. |
|--------------|------------|----|---------|------|
| Clove 5 minutes | 4.3185 | 1 | 0.0377 | S |
| Clove 30 minutes | 5.1305 | 1 | 0.0235 | S |
| Peppermint 5 minutes | 0.2111 | 1 | 0.6459 | NS |
| Peppermint 30 minutes | 0.0409 | 1 | 0.8397 | NS |

S: significant difference at p<0.05* NS: no significant difference.

V. DISCUSSION

Several attempts started to focus on reducing the force applied during the de-bonding of esthetic brackets. Both bonding and de-bonding procedures were found to affect these forces.

An effective de-bonding agent should be capable of reducing not only the de-bonding force, but also the amount of adhesive remaining after de-bonding with no enamel or bracket fracture. Thus, a comparison between the quantitative and qualitative results for each volatile oil is instrumental in determining the best de-bonding agent. Hence, each variable should be considered separately when determining the best volatile oil for de-bonding.

In the present study, it was found that the black seed volatile oil had the least mean de-bonding force. However, no significant difference was observed between 5 and 30 minutes of application. On the other hand, the control and the mixture of peppermint and black seed volatile oil showed the highest mean de-bonding force with no significant difference between the two times of application.

Further, the same feature was observed when comparing the clove oil and the peppermint oil. There was no significant difference in the mean de-bonding force in 5 and 30 minutes of application. But interestingly, both were significantly affected by the time of application. The clove oil revealed a higher mean de-bonding force at 5 minutes than at 30 minutes. The opposite was true for the peppermint oil (Table I).

On the other hand, the survival analysis gave similar results when comparing the characteristic de-bonding force and the probability of failure at 105 N. This indicates that the application of clove, peppermint, or black seed volatile oils is of benefit quantitatively. It also might give a clue that the latter volatile oil, if used frequently by a patient in his diet, medication, or as a mouthwash may give rise to a problem of frequent loose brackets that need to be re-bonded during orthodontic treatment.

It is worth considering that any applied chemical agent should not be assessed on its effect on the adhesive only in giving the final de-bonding force results; but also, on its effect to the whole conditioner-adhesive-bracket combination system. However, the chemical reaction between the volatile oil and the combination system was not investigated in the present study. Even though, it was observed that the latter finding indicated that the clove oil weakens the system more with time, whereas the peppermint oil strengthens the system more with time.

The mean de-bonding forces for the three volatile oils (black seed, peppermint and clove oils) were all within the acceptable range (significantly lower than the control) (Fig. 9). Thus, the decision to choose the preferable volatile oil will depend on the difference of the debonding force results. Thus, the 5 minutes of black seed and peppermint oil application gave lower mean de-bonding force than the 30 minutes application. Accordingly, the 5 minutes black seed and peppermint oils application seems to give the best preferable results. This may encourage the application of black seed or the peppermint oil in the clinic 5 minutes before de-bonding the case.

In their recent study [31] used eugenol, Iso eugenol, Methyl-eugenol. They divided their study sample into three groups (n= 15 each) control, eugenol 10 minutes application and eugenol 24 hours application. The eugenol 24 hours application showed significantly lower mean shear bond strength (2.29 +/− 0.69 MPa) than the other two groups control (6.68+/− 1.73 MPa) and eugenol 10 minutes group (4.72 +/− 2.48 MPa) at P< 0.05. The result of the present study is only in agreement with [31] result in the time of application, the longer the time of application (24 hours) the lower the force of debonding compared to 10 minutes when applying clove oil and in disagreement when applying black seed as well as the peppermint volatile oil. However, in the present study the 5 minutes application showed lower force compared to 30 minutes (Table I).

Among the volatile oils investigated in the present study, the peppermint oil was the only volatile oil that was studied previously by [18] specifically its effect on de-bonding forces of ceramic brackets. However, the two studies differ in many variables, the most being

1) the conditioner – adhesive-bracket combination.
2) the type of loading (shear, using de-bonding plier).
3) the configuration of specimen testing jig.
4) the crosshead speed of mechanical testing machine.
5) the significance level (Table III).

Thus, a valid comparison between the two studies is very limited, if not impossible. Hence, we are left with comparison within each study, such as the effect of peppermint oil at different times of application, on the de-bonding of ceramic brackets.

In the current study; the application of peppermint oil gave significantly lower de-bonding force compared to the control group (at 5 minutes than at 30 minutes) which disagrees with [25] findings (Table III) who did find any significant difference between peppermint oil application and the control. However, it is Worth to mention that; the level of significance in [25] study was at P <0.001 whereas in our study was at P < 0.05. This could be one of the possible justifications of disagreement between the two studies. Previous work by [24] showed that the micro-hardness of the Transbond adhesive was not affected by the application of peppermint derivative de-bonding agent (P-de-A) for 30, 60, 90, 120 or 180 seconds, whereas the micro-hardness of the Concise adhesive was lowered only significantly after 180 seconds of application. This was supported by the findings reported by [23], who suggested that peppermint oil functioned as crazing facilitating crack propagation through the composite bond layer during the removal of the ceramic brackets.
We can draw from the above discussion that the criteria for the selection of the best volatile oil as a de-bonding agent should include, acceptable de-bonding force, + minimal or no adhesive remnant, + no enamel fracture + and no or minimal bracket fracture. Fulfilling all these criteria simultaneously remains a challenge. However, based on the findings of the present study, one can also suggest that the 5- minutes of black seed and peppermint oil application could be considered as the best de-bonding agent compared to the other volatile oils, and that the clove oil can be used after de-bonding to facilitate the final cleansing-up of enamel surface. To confirm this latter assumption, further investigations are needed.

The present investigation and the few previous and recent published reports [24], [25], [32]-[35] on chemical solvents opened the door for a new challenging area of research that requires detailed instructions for bonding and procedures has also been a concern in contemporary dentistry. To confirm this latter assumption, further investigations are needed.

VI. CONCLUSION

Black seed volatile oil gave the lowest de-bonding force whereas the peppermint volatile oil was in the second rank and the mixture of black seed and peppermint volatile oils demonstrated the highest debonding force.

Clove oil reduces the de-bonding force (5 minutes 89.37 N) and this effect increased with time (30 minutes 77.68 N).

Finally, an assumption has been drawn from the present investigation that the 5-minutes of black seed and peppermint volatile oils application were considered the best debonding agent compared to the other volatile oils.

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CONFLICT OF INTEREST

Authors declare that they do not have any conflict of interest.

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