vol.3, No. 2, pp. 285-292 ISSN 1805-3602

Effect of different conditioning methods on the surface roughness of dental amalgam: SEM analysis

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Received for publication: 24 January 2014.
Accepted for publication: 24 April 2014

Abstract

The purpose of this study was incorporation of SEM micrographs for evaluating the effect of different mechanical and chemical treatments on surface roughness of dental amalgam. Amalgam was condensed in 18 plastic molds and the following modification methods were accomplished on samples surface:

1) Chemical solutions (5 groups) on fresh amalgam 2) Chemical solutions (5 groups) on aged amalgam 3) Sandblast on aged amalgam 4) Sandblast on fresh amalgam (2 groups) before & after primary setting. 5) Use of diamond bur (0.10 coarse) on aged amalgam. 6) Acid etch (37% phosphoric acid) on fresh amalgam. 7) Metal etch on fresh amalgam. 8) Varnish solvent in amalgam capsule, trituration &condensation (fresh). 9) Standard sample without any treatment.

All groups were analyzed by SEM (×l00, ×500 magnifications were used for roughness quantification and chemical analysis respectively). The numbers of surface peaks were counted in 5 area of each micrograph and consequently the data were assessed by K-S & student T- tests.

All groups have statistically significant difference with control except three ones (incorporation of phosphoric acid, EDTA+NH_3, and NaOH+I_2 on fresh amalgam). Sandblast, diamond bur & metal etch reduced surface roughness and use of chemical solutions showed different reactions.

Keywords: Dental Amalgam, Scanning Electron Microscopy, Surface roughness

Introduction

Bonding of resin composite to set amalgam is considerably beneficial in clinical dentistry (Garcia-Barbero *et al*, 1994). Actually, the possibility of such complex restorations leads to a kind of morpho-functional treatment; the amalgam guarantees the mechanical behavior while composites reinforce the residual dental tissues and ensures aesthetics (Plasmans & Reukers, 1993). In this manner, the composite can mask the extension of amalgam on the buccal surface or any other visible part of the tooth (Bedini *et al*, 1994).

Another most desirable advantage of these complex restorations could be repair of a fractured tooth structure adjacent to an old amalgam filling (Zcan *et al*, 2006); because repair of a restoration is more cost-effective (Randall *et al*, 2002) and notably more conservative to tooth tissues comparing to total replacement (Blum *et al*, 2012). Since bonding a composite resin to the remaining tooth structure reduces the risk of any consequent fracture (Denehy & Torney, 1976; Fissore *et al*, 1991), composites are preferred against amalgam material in the mentioned situations for reconstruction (Franchia *et al*, 1999).

Moreover, combined amalgam-composite restorations would be helpful in deep CL II preparation when a gingival margin lies close to or apical to the cementoenamel junction. It has been documented that the microleakage at the resin/cementum interface is significantly higher than amalgam/cementum and amalgam/composite resin interfaces

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(Hadavi *et al*, 1991; Cardash *et al*, 1990). Therefore, in these conditions a technique has been described to fill the proximal portion of class II cavities up to the contact area with amalgam and the remainder by resin composite (Cardash *et al*, 1990).

Furthermore, extensive buccal amalgam restorations are common problems in orthodontic propositions. These teeth are frequently managed by a surrounding band that could resist debonding forces. However, one of the most important problems rendering the bands is that they increase the possibility of periodontal disease (Boyd & Baumrind, 1992; Huser *et al*, 1990). On this ground, a reliable bonding of orthodontic attachments to dental amalgam is vastly desirable (Sperber *et al*, 1999).

Additionally, enhancement the bonding strength of resin cements to amalgam cores would be quite advantageous for retention of extra coronal restorations (Watts *et al*, 1992).

Bonding of another dental material to amalgam has been extensively investigated (Mojon *et al*, 1989). Although a growing number of commercial repair systems for the direct veneering of amalgam have been introduced to the market, the best protocol for performing an amalgam repair is still a controversial issue (Blum *et al*, 2012).

Essentially, the suggested techniques for bonding a resin to amalgam could be categorized as either mechanical or chemical adhesion (Sperber *et al*, 1999). The chemical bonding was introduced by using compounds such as 4-methacryloxyethyl trimellitate anhydride (4-META) or 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) (Sperber *et al*, 1999). However, a true chemical bond between amalgam and resin composite is not reliable and reported as a questionable phenomenon in literatures (Bichacho *et al*, 1995; Miller *et al*, 1992). Hence, the primary basis for bonding a dental composite to amalgam includes the mechanical bonding (Winkler and Moore, 1994).

Any mechanical adhesion is based on three principles including surface topography of the solid state, the viscosity of the liquid and the surface free energies (Eick *et al*, 1972). Although many studies evaluated the bond strength or microleakage of resin/amalgam interface (Garcia-Barbero *et al*, 1994; Zcan *et al*, 2006; Blum *et al*, 2012; Sperber *et al*, 1999; Watts *et al*, 1992; Bedini *et al*, 1994; Franchia *et al*, 1999), surface topography of amalgam is the least understood issue among the discussed three factors (Winkler and Moore, 1994). However, it has been shown that increasing the surface roughness result in a greater bonding of resins to solid states (Jung *et al*, 1999); but few investigations quantified the altered surfaces of amalgam for bonding.

The aim of this paper was evaluating the effect of different treatments on the surface roughness of amalgam samples using scanning electron microscopy (SEM).

Materials and methods

Specimen preparation

Eighteen rectangular 3mm 4mm 4 mm plastic molds were filled by the triturated amalgam (non-gamma 2, Spherical, high-copper alloy with 49% Ag,31% Sn,20% Cu) (Cinalux, Owzan,Iran) according to the manufacturer's instruction using a hand instrument condenser by a single operator.

Surface treatment

Among eighteen prepared amalgam samples, one of them was immediately carved by a triangular hand instrument and then sand blasted by 50 μm Al_2O_3 particles (Korox R, Bego, Bremen, Germany) using an intraoral sandblaster (Dento-PrepTM, R NVIG A/S, Daugaard, Denmark) from a distance of 10 mm at a pressure of 2.5 bar for 4 s (This sample is referred as Group No.16 in Table.1). In the remainder seventeen specimens, the carving procedure was accomplished after initial setting (3.5 min recommended by the manufacturer). Consequently, different surface conditioning methods were applied to various groups which are summarized in Table 1. As it is demonstrated, after carving, nine samples received treatment protocols, while seven samples were allowed to set for 24h at 23°C prior to surface treatment (noted as fresh and aged specimen in the Table.1 respectively). One sample which serves as control group did not receive any surface modification.

SEM analysis

After 30s washing and air drying, all samples were gold coated. Subsequently, they were examined under SEM (Tubney Woods, Abingdon, Oxfordshire, UK) to investigate the surface morphology. Each specimen was analyzed by two magnifications including ×100 and ×500. At the magnification of 100, each picture was meshed into 500 μm squares using Meazure software (version 2.0.158). Five different squares were randomly selected on every pictures and the number of surface peaks was quantified in each square. In this manner we had five quantitative records in each group considered as five samples in either group (n = 5/group).

Table 1. Description of different surface conditioning methods for various experimental groups

Group No.	Surface conditioning Protocol	duration	The age of sample
1	Al_2O_3 sandblasting	3 sec	Aged
2	Incorporation of copal varnish into the amalgam capsule prior to trituration	___	Fresh
3	EDTA + NH_3	5 min	Fresh
4	EDTA + H_2O_2	5 min	Fresh
5	NaOH + I_2	5 min	Fresh
6	$FeCl_3$ + I_2	5 min	Fresh
7	HNO3 + NaOH + NaCl	5 min	Fresh
8	HNO3 + NaOH + NaCl	5 min	Aged
9	$FeCl_3$ + I_2	5 min	Aged
10	NaOH + I_2	5 min	Aged
11	EDTA + H_2O_2	5 min	Aged
12	EDTA + NH_3	5 min	Aged
13	0.1 coarse diamond bur using a high speed handpiece	3 sec	Aged
14	Phosphoric acid gel 37%	5 min	Fresh
15	Al_2O_3 sandblasting	3 sec	Fresh
16	Al_2O_3 sandblasting	3 sec	*Fresh
17	Metal etch solution	5 min	Fresh
18	Control group without any conditionin	___	Fresh

*Less than 4 min after trituration

On the other hand, at the magnification of ×500, the surface chemical analysis of each sample was accomplished by the attached SEM sensor.

Statistical analysis

In order to determine if the data have a normal distribution, the obtained numbers related to the experimental groups were analyzed by Kolmogroph Smirnove test. Consequently, each group was separately compared to the control using independent T-test (α= 0.05).

Results

As it is illustrated in Table 2, among seventeen modified group, most of them have significant difference with unmodified group. Meanwhile, some of them have more surface roughness comparing to the control while the others have less. It means that sandblasting of fresh or aged amalgam, using of diamond bur on aged specimens, and also incorporation of metal etch solution on fresh amalgam decrease its surface irregularities.

Conversely, application of neither EDTA+NH_3, NaOH+I_2 solutions, nor phosphoric acid gel would not significantly modify the surface topography of amalgam.

Furthermore, the surface chemical analyses of different groups are demonstrated in Fig.1 to Fig.5. As can be seen, surface modification leads to reduction of some ingredients on amalgam surface. Moreover, surface micrograph of three samples is demonstrated in Fig.6.

Table 2. Mean surface roughness values among the experimental groups

Group No.	Mean ± SE
1	7.8±0.84*
2	26.8±1.3*
3	22.2±1.64
4	13.4±1.52*
5	22.4±1.4
6	22.8±0.84*
7	16.4±0.55*
8	17.2±0.84*
9	3.6±0.55*
10	26.8±1.3*
11	12.6±1.14*
12	16.6±1.67*
13	10.4±1.14*
14	20.4±1.14
15	4.2±0.84*
16	2.2±0.84*
17	19.2±0.84*
18	21.2±0.84

*Statistical significant difference comparing to the control group (P<0.05)

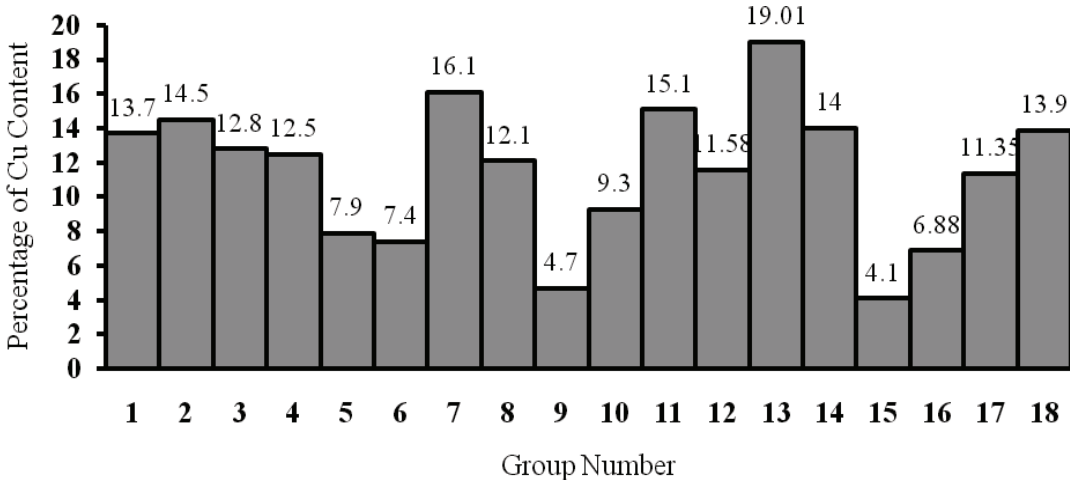

Figure 1. The percentage of copper (Cu) content on the surface of eighteen experimental groups analyzed by Scanning Electron Microscopy

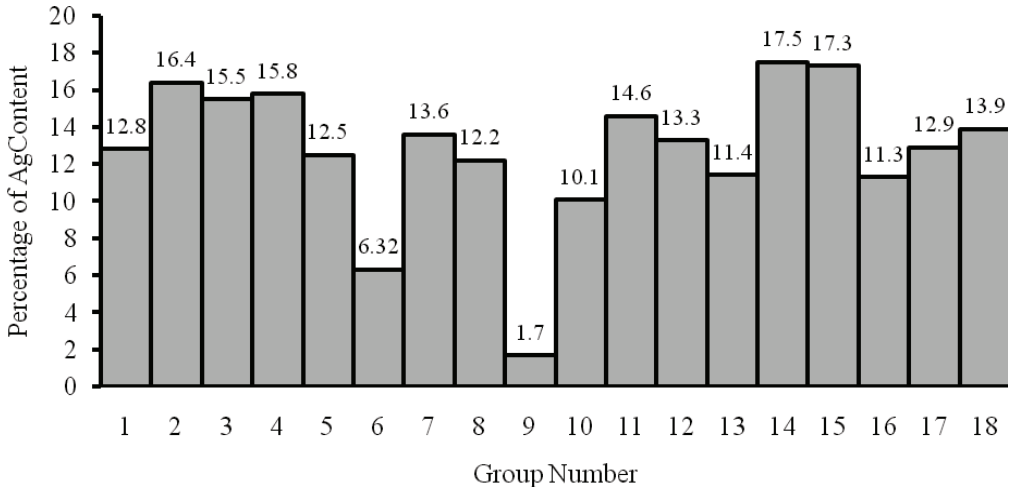

Figure 2. The percentage of silver (Ag) content on the surface of eighteen experimental groups analyzed by Scanning Electron Microscopy

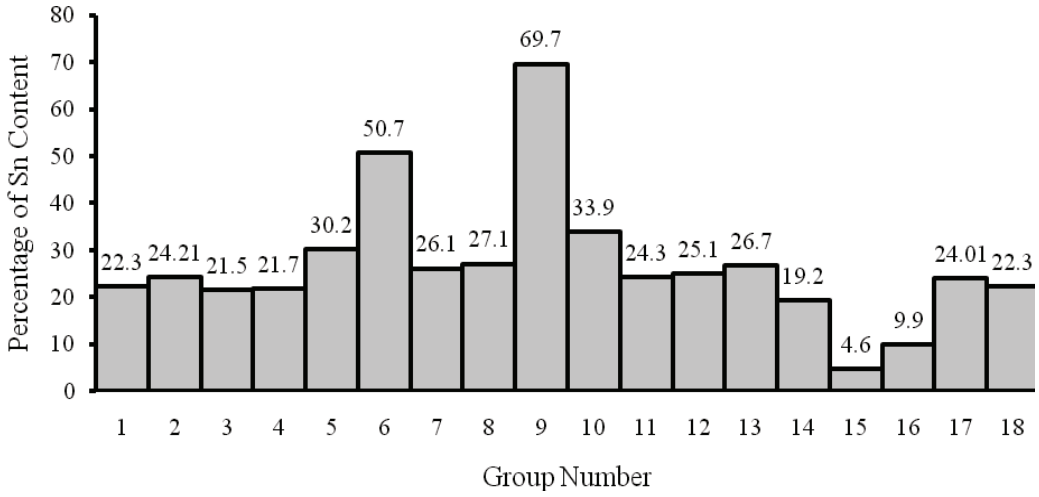

Figure 3. The percentage of tin (Sn) content on the surface of eighteen experimental groups analyzed by Scanning Electron Microscopy

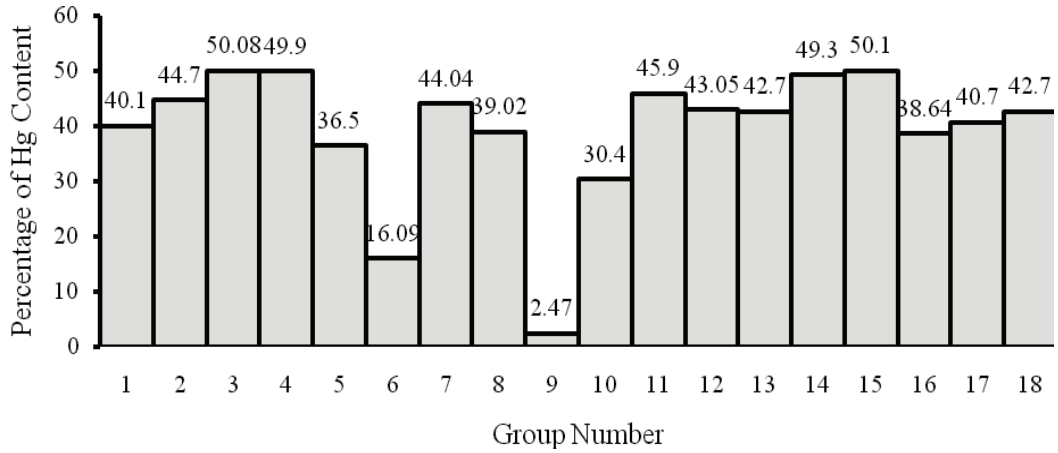

Figure 4. The percentage of mercury (Hg) content on the surface of eighteen experimental groups analyzed by Scanning Electron Microscopy

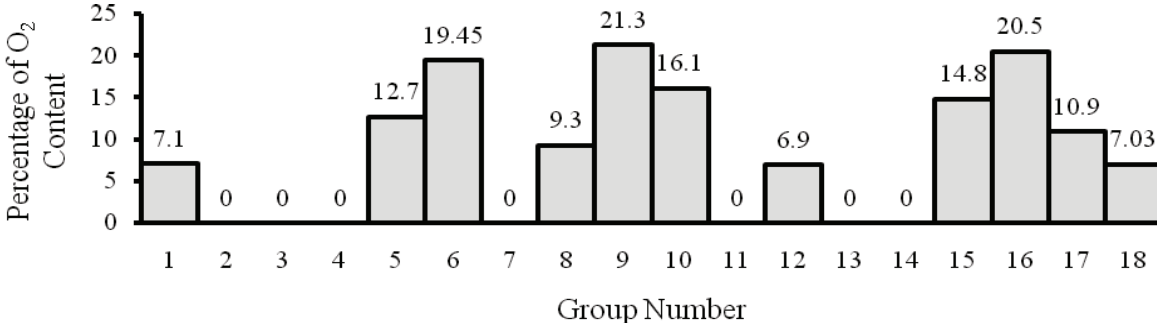

Figure 5. The percentage of oxygen (O_2) content on the surface of eighteen experimental groups analyzed by Scanning Electron Microscopy

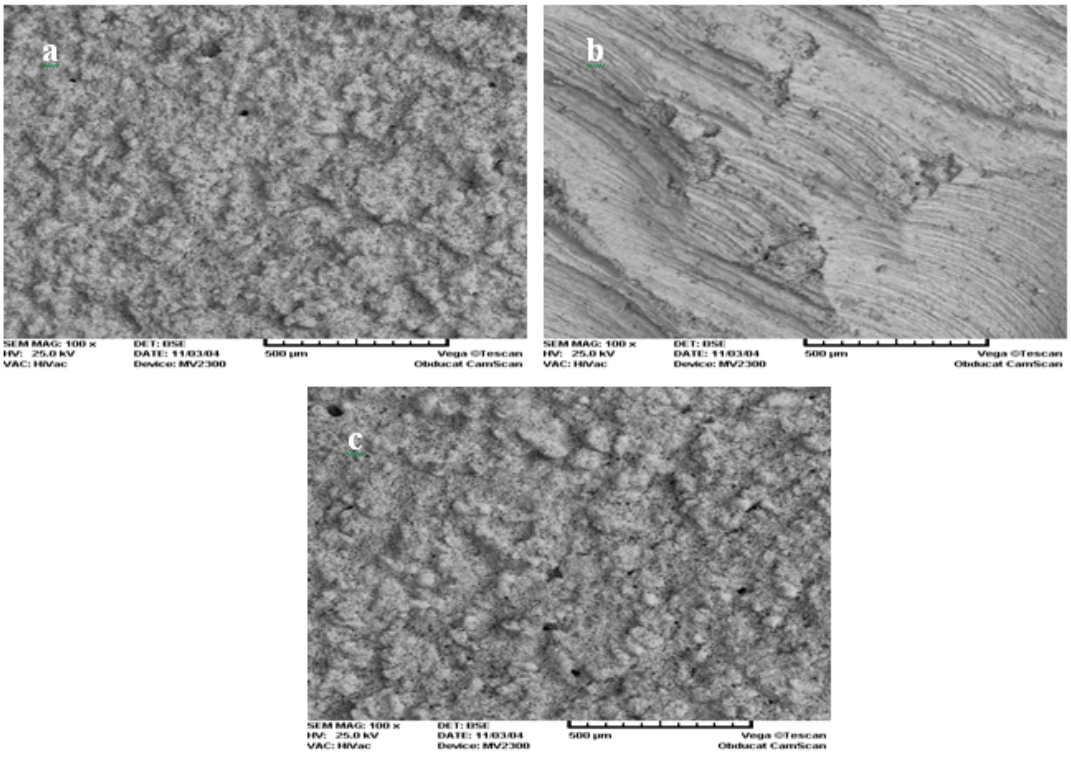

Figure 6. SEM micrographs of three samples including: control unmodified group (a), surface treatment by 0.10 coarse diamond bur (b), and treated by metal etch solution (c).

Discussion

Our results showed that except three groups (3rd, 5th and 14th groups), all the other treated samples had statistical significant difference with the control group. The highest surface roughness is obtained in the 2nd and 10th groups those are more than the control. Whereas, eleven numbers of experimental samples had mean surface roughness less than the unmodified group.

Direct clinical relevance of the present study is related to the situations of bonding any resinous material to an existing amalgam restoration (Winkler and Moore, 1994). Since a true chemical bond between amalgam and resin composite is controversial yet (Bichacho *et al*, 1995; Miller *et al*, 1992). this bonding phenomenon is based on the mechanical tag formation (Watts *et al*, 1992), which is considered as either micro- or macro-mechanical retention. Numerous studied focused on the bond strength between amalgam and various materials by different surface treatments; most of them argued that for achieving acceptable bond strength to amalgam, surface preparation method plays a more significant role than the type of bonding resin (Zcan *et al*, 2006; Blum *et al*, 2012; Sperber *et al*, 1999; Atta *et al*, 1990; Takeya *et al*, 1988). Although overall they suggest establishing a rough surface on the existing amalgam restorations for additional possibilities of retention (Cooley *et al*, 1989; Rawlinson, 1987), just few of them investigated the surface parameters of amalgam.

Frequently in dental material science the surface irregularities are evaluated by surface profilometers. Although this method is quite valuable, it has some disadvantages including a planar measurement over line distance (Konishi *et al*, 1985). Accordingly, its results are reported as directional depended (Winkler and Moore, 1994). For example, Winkler.M etal evaluated the effect of amalgam surface roughness on the bond strength of amalgam/resin interface using profilometer. They concluded that if the measurement accomplished in the direction parallel to the finishing scratches, the surface roughness is correlated to the shear bond strength, while measuring in the direction perpendicular to the finishing scratches did not showed any correlation between these two variables (Winkler and Moore, 1994).

The laser scattering method has overcome the mentioned disadvantage of profilometers, but it can discriminate the roughness changes in dental amalgams (Konishi *et al*, 1985).

The SEM micrograph has been used in numerous studies on surface topography, but almost all of these researches used the SEM micrograph just for qualitatively describing their surface morphology (Sperber *et al*, 1999; Jung *et al*, 1999; Janus *et al*, 2010; Sadaghiani *et al*, 2007). However, in very few articles it has been reported that with image analysis systems, the SEM could yield quantitative information (Luo *et al*, 2001). Since SEM micrograph provides us a very good representation of surface changes (Luo *et al*, 2001), in the current research we obtained numerical data from the SEM micrographs that could be an advantage in surface analysis.

Regarding to our result, the bur prepared group had more surface irregularities comparing to the sand blasted groups. This finding is in accordance with Blum I.R etal who examined their samples by three-dimensional profilometry (Blum *et al*, 2012). However, they reported that the tensile bond strength of amalgam/resin interface were significantly higher in the alumina sandblasting group compared with diamond bur (Blum *et al*, 2012). They argued that sandblasting would improve surface homogeneity by removal of large surface defects and leads to formation of an improved adhesive bond, while using bur can produce large surface asperities (Blum *et al*, 2012).

Furthermore, our air abraded samples had smoother surface comparing to the unmodified group that is in agreement with Blum I.R etal who reported a low Ra-value in their sandblasted group (Blum *et al*, 2012). It has been highlighted in some articles that during condensation, many mechanical irregularities form on the amalgam surface, but since amalgam is a relatively ductile material, it would smear as it is abraded, and thus the surface irregularities may be smoothed out (Winkler and Moore, 1994).

Therefore, although we used a new method for measuring the surface roughness, our results are approximately similar to the investigators who used profilometer.

The NaOH, I_2, HNO_3, $H2O_2$, $FeCl_3$, NH_3 and EDTA solutions are documented as chemicals that react with some ingredients of amalgam alloy including Hg^{2+} or Ag^{+} ions, reducing them to Hg or Ag elements and extract them from the surface of the material (Akaiwa *et al*, 1977; Pereira *et al*, 2010; Rotstein *et al*, 2001). Hence, we incorporated these solutions as etching agent for amalgam surfaces; meanwhile our chemical analysis confirmed this application consequently. As can be seen in Fig.1 and Fig.2, in both fresh and aged amalgam surfaces which were exposed to these solutions, the Cu and Ag components were obviously decreased comparing to the control group.

All together, although in vitro investigations are just a potential predictor of clinical performance and they are not a direct translation of in-vivo situations (Blum *et al*, 2012), our findings revealed that most

of our conditioning methods significantly modified the topography of amalgam surface comparing to the control group. Nevertheless, these results are applicable to the spherical alloy which is used in this study while it is reasonable to test whether they are efficient as well for the admixed or lathe-cut alloys.

Conclusions

In summary, the most important finding of this research was that incorporating SEM micrographs for quantifying surface roughness were in accordance with the previous profilometry studies.

Moreover, application of diamond bur, Al2O3 sandblasting particles, and metal etch solution reduced the surface roughness of amalgam samples comparing to the control group. Whereas, chemical solutions lead to different surface modifications on fresh amalgam samples comparing to aged ones.

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