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Nanomechanical properties and wear resistance of dental restorative materials

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Abstract. The effects of thermocycling procedure and material shade on the mechanical properties and wear resistance of resin-based dental restorative materials are investigated. The modulus of elasticity, hardness, plasticity index and wear resistance are determined for the conventional composite, the nanohybrid composite and the nanofilled dental composites. Disc-shape samples are prepared from each material to investigate the effects of thermocycling procedure on the mechanical properties and wear resistance of different types of dental restorative materials. In this respect, a group of samples is thermocycled and the other group is stored in ambient conditions. Then nano-indentation and nano-scratch tests are performed on the samples to measure their mechanical properties and wear resistance. Results show that the A1E shade of the dental nanocomposite possesses higher modulus of elasticity and hardness values compared to the two other shades. According to the experimental results, the mean values for the modulus of elasticity and hardness of the A1E shade of the nanocomposite are 13.71 GPa and 1.08 GPa, respectively. The modulus of elasticity and hardness of the conventional dental composite increase around 30 percent in the oral environment due to the moisture and temperature changes. The wear resistance of the dental composites is also significantly affected by moisture and temperature changes in the oral conditions. It is observed that thermocycling has no significant effect on the hardness, plasticity index and wear resistance of the nanohybrid composite and the nanocomposite dental materials.

Keywords: dental restorative polymers; nano-indentation experiment; nano-scratch experiment; surface analysis; thermocycling effect

1. Introduction

Resin-based dental restorative composites are a class of polymeric materials which are widely used in dentistry. The ultimate purpose of resin-based dental restorative materials is to act as a material with biological, functional and esthetic properties similar to those of the healthy tooth structure. There is an increasing interest to replace amalgam by resin-based dental restorative composites due to their beauty and strength in comparison with amalgam. Resin-based dental restorative composites were previously used for the restoration of anterior teeth, but by increasing the load bearing capacity of these materials, they are now used in restoration of posterior teeth too. Since their introduction to dentistry, resin-based dental restorative composites have undergone considerable changes in order to achieve mechanical properties comparable to dental amalgams (Demarco *et al.* 2012, Maserejian *et al.* 2012, Sowmya *et al.* 2013).

Dental composites are generally made of fillers, silane coupling agent and a resin consisting of a polymer matrix which usually involves bisphenol-a-glycidyl methacrylate (Bis-GMA) mixed with triethyleneglycoldimethacrylate (TEGDMA) and reinforced with dispersed particles including barium or zinc glasses, quartz, zirconia, silica, etc. In almost all commercial restorative resins, Bis- GMA is used as the primary organic ingredient (Oliveira *et al.* 2012, Fu *et al.* 2014).

Numerous researchers have attempted to improve the mechanical properties of dental composite resins (Moszner and Salz 2001, Park *et al.* 2010, Leprince *et al.* 2013). Studies on the resin matrix are often based on the development of new monomers (Zhou *et al.* 2013, Makvandi *et al.* 2016), while researches on the filler content are focused on the filler loading, particle size and development of new particles (Tian *et al.* 2007, Bindu *et al.* 2013). The mechanical properties of composites depend strongly on the size and amount of the filler particles.

The addition of nano-size particles to resin-based dental composites is one of the most important advances in the recent years (Melo *et al.* 2013, Ozak and Ozkan 2013). In dentistry, resin-based composites used for posterior restorations must possess high mechanical properties while those used for anterior restorations require superior esthetics. By adding nanoparticles to the dental composite resin, researchers could develop dental nanocomposite restoratives which have almost all requirements for both posterior and anterior restorations (Beun *et al.* 2007).

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Table 1 Dental restorative materials used in this study

Material Type	Type of resins	Filler particles	Filler loading (By volume)	Shade
Composite (Filtek Z250)	BIS-GMA, UDMA, BIS-EMA	Zirconia/silica cluster	60%	B1
Nanohybrid composite (Filtek Z250 XT)	BIS-GMA, UDMA, PEGDMA, TEGDMA	Silica, Zirconia/silica cluster	67.8%	B1
Nanocomposite (Filtek Z350 XT)	BIS-GMA, UDMA, TEGDMA, PEGDMA, BIS-EMA	Zirconia, silica, zirconia/silica cluster	63.3%	WE & WD & A1E

On the other hand, various shades of dental restorative materials have been developed to create desired physical properties similar to those of the natural tooth. The polymerization process of light-curing dental restoratives and also their mechanical properties are affected by the light absorption of different shades (Öztürk *et al.* 2013, Passos *et al.* 2013, Ilie and Stawarczyk 2014). However, very few researches have been performed to study the variation of mechanical properties based on different shades of dental restorative nanocomposites.

The mechanical properties of common dental restorative composites are often evaluated at ambient conditions, while the effects of temperature changes and humidity on the mechanical properties of different types of restorative composites have not been widely considered in the literature (Nakamura *et al.* 2010, Ferracane 2011, Sideridou *et al.* 2011). Therefore, it is important to study the variations in the properties of resin-based dental restorative composites induced by temperature changes and humidity (Carsí *et al.* 2013, Eftekhari and Fatemi 2016, Zhang *et al.* 2016). Moreover, little information is available in the literature about the effects of oral conditions on the wear resistance of dental restorative materials, while the abrasion of dental restoratives caused by direct contact between the tooth and the restoratives during operational loads may reduce the quality and esthetics of the restoration. The oral conditions, including temperature changes and chemical solutions intensify the abrasion of dental restoratives. Such temperature changes are normally simulated by an in-vitro process called thermocycling (Stewardson *et al.* 2010, Machado *et al.* 2012).

The nano-indentation as well as the nano-scratch experiments are known as two favorite methods for obtaining the materials properties including the modulus of elasticity and hardness values and also the wear resistance for small scale specimens (Shokrieh *et al.* 2013, Karimzadeh *et al.* 2014, Karimzadeh *et al.* 2014, Choi *et al.* 2016). The applications of these two methods are increasing in various types of materials including the dental materials. For example, these techniques have been used for determining the modulus and hardness of polyamide-12/layered-silicate nanocomposites (Aldousiri *et al.* 2011), for evaluating the mechanical and morphological properties of clay nanocomposites (Shen *et al.* 2005, Shen *et al.* 2006) and also for measuring the mechanical properties of bone cement (Ayatollahi *et al.* 2012, Karimzadeh and Ayatollahi 2012). Since nano-indentation and nano-scratch tests require very small sample, they are appropriate for costly materials like dental restorative composites. Desired characteristics including the load control precision, the scan size and the absence of chemical environment and electrical fields make the nano-scratch experiment a suitable alternative to conventional wear testing methods (Ayatollahi *et al.* 2012).

The present study investigates the effects of three common dental restorative shades on the modulus of elasticity and hardness of a dental restorative nanocomposite measured by nano-indentation test. Moreover, the thermocycling procedure in distilled water is used to study the influences of oral conditions on the mechanical properties and wear resistance of three different types of dental restorative materials.

2. Materials and methods

The purpose of this study is to investigate the effects of thermocycling procedure and material shade on the mechanical properties and wear resistance of commercial resin-based dental restorative materials. In this respect, the variables are thermocycling procedure, dental material type and shade which can be considered as input parameters. The levels of thermocycling parameter are thermocycled and non-thermocycled, while the levels of dental material parameters include the conventional composite, the nanohybrid composite and the nanofilled dental composites and the levels of shade parameter are dentine (WD), white enamel (WE) and A1 enamel (A1E). The output parameters are modulus of elasticity, hardness, plasticity index and wear resistance, which are measured through nano-indentation and nano-scratch experiments.

2.1 Sample preparation

Filtek Z350 XT (3M ESPE, USA) nanocomposite, Filtek Z250 XT (3M ESPE, USA) nanohybrid composite and Filtek Z250 (3M ESPE, USA) conventional composite were used for preparing the samples. Three different shades of the dental nanocomposite, including white dentine (WD), white enamel (WE) and A1 enamel (A1E) were used to study the effect of shade on the mechanical properties. The basic characteristics of these dental materials are presented in Table 1 according to the manufacturer's data sheet. A mold for disc-shaped specimens of 10 mm diameter and 4 mm thickness was prepared. Each dental restorative material was injected into the mold and then light cured. For light curing an LED light (Sony, Japon) with intensity of 400 mW/cm^2 was used as close as possible to the restorative surface and each side of the specimen was light cured for 20 seconds according to the manufacturer's instructions. Then the specimen was removed from the mold. In order to create a smooth surface required for nano-

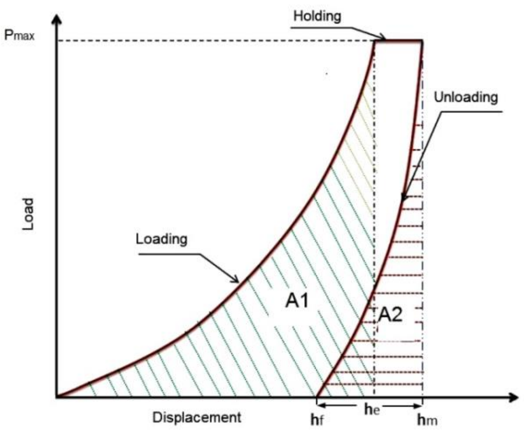

Fig. 1 Important parameters of nano-indentation test in a schematic load-displacement curve

indentation and nano-scratch tests, the specimens were grounded using 400 to 2500 grits sandpapers and then smoothed by diamond pastes (Lapex, Eu) with mesh sizes of 1, 0.5 and 0.25 microns, respectively. The roughness values of samples were inspected using atomic force microscopy (AFM) images.

2.2 Thermocycling

The thermocycling procedure was performed by placing one specimen of each material type into the thermocycling machine in stilled water bath and then thermocycling it for 2000 cycles, between temperatures of +5 and +55℃ with dwell time of 30 seconds (Stewardson *et al.* 2010). For each type of material, one sample was maintained as control specimen, which was kept in the incubator at 37℃ and the room humidity.

2.3 Nano-indentation test

The nanomechanical properties of each test specimen, such as hardness and modulus of elasticity were extracted by Triboscope system (Hysitron Inc., USA) based on ISO 14577. Since bulk specimens were used, a Berkovich indenter tip (Hysitron Inc., USA) was applied in all experiments. The test instrument was calibrated according to the approach proposed by Oliver and Pharr (Oliver and Pharr 2004).

During the loading segment of the nano-indentation experiment, the maximum indentation displacement was set equal to 300 nm in which the indentation load was applied monotonically within 30 seconds. To consider the time dependent behavior of the material, the indenter was held on the specimen for 10 seconds at the maximum indentation load. Eventually, the unloading segment of the experiment was done by raising the indenter tip from the specimen at the same rate of loading. At least five nano-indentation experiments were performed on different spots of each specimen, and the curves of normal indentation load versus normal displacement of the indenter were obtained from the test device. Each indentation spot was scanned by using an atomic force microscopy (AFM) before and after the test to analyze the nano-indentation hole. The AFM inspection was performed by the same instrument and the same tip of the nano-indentation experiment. The important parameters that are required to be measured from the force-displacement curve and to be used in analyzing the experimental data are shown in Fig. 1 in a typical force-displacement curve.

In this research, the hardness was calculated by using the data obtained from the force-displacement curves and the effective modulus of elasticity was determined from the Oliver and Pharr’s method (Briscoe and Sinha 2003, Oliver and Pharr 2004) as presented in Eqs. (1)-(2)

$$H = \frac{P_{max}}{A} \tag{1}$$

where H is the material hardness, P_{max} is the maximum normal indentation load and A is the projected contact surface between the indenter and the specimen. For the effective modulus of elasticity (E_{eff})

$$E_{eff} = \frac{\sqrt{\pi}}{2} \frac{dP}{dh} \frac{1}{\sqrt{A}} \tag{2}$$

$$\frac{1}{E_{eff}} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \tag{3}$$

where, E_i and ν_i are the modulus of elasticity and Poisson’s ratio of the indenter tip, and ν is the Poisson’s ratio of the test sample. In Eq. (2), h is the indentation depth and dP/dh represents the slope of the unloading part of the load-displacement curve obtained from the nano-indentation test at maximum indentation depth. Considering the values of ν=0.31 (Chung *et al.* 2004), E_i=1140 GPa and ν_i=0.07 (from the technical data available for the related Triboscope system), the modulus of elasticity of samples could be calculated from Eq. (3).

The plasticity index is a parameter for specifying the elastic-plastic behavior of the material ranging from 0 for perfecte elastic behavior, to 1 for full plastic behavior. In the nano-indentation test, the plasticity index (ψ) is calculated using Eq. (4) (Ayatollahi *et al.* 2012)

$$\psi = \frac{A_1 - A_2}{A_1} \tag{4}$$

where A_1 is the area under the loading segment of force-displacement curve and A_2 is the area under the unloading segment. These areas are shown in Fig. 1.

As described in [14], the plasticity index can be determined from Eq. (5)

$$\psi = \frac{h_m - h_e}{h_m} \tag{5}$$

where h_m is the maximum indentation depth at the end of loading segment and h_e is the elastically reversible depth. The difference between h_m and h_e demonstrates the residual indentation depth which can be determined from the load-displacement curve.

2.4 Nano-scratch test

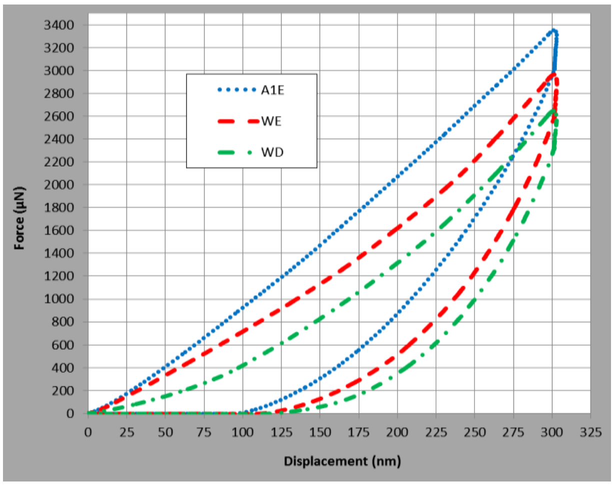

Fig. 2 Force-displacement curves obtained for different shades of the dental nanocomposite

In the nano-scratch experiment, the indenter penetrated in to the surface of specimen by applying a normal load of 1950 μN and was drawn with a constant scratching speed of 0.13 μm.s^{-1} through the preset scratch length of 4 μm. At least three nano-scratch tests were performed on randomly selected sites of each specimen. The sample surface at the scratch site was scanned before and after the nano-scratch experiment. The pre-scratch scan was performed to evaluate the smoothness and the tilt angle of the specimen surface. The material behavior beneath the indenter at the scratch site was investigated using the post-scratch scan. Comparing the pre-scratch scans with the post-scratch ones determines the wear resistance and elastic-plastic behavior of the sample material. The same instrument and the same indenter tip as the nano-indentation experiment were used for the nano-scratch experiment and the AFM evaluation.

3. Results and discussion

3.1 Shade effect

Samples of force-displacement curves obtained from nano-indentation experiments on different shades of the dental nanocomposite are illustrated in Fig. 2.

The values of the hardness and modulus of elasticity of the specimens were calculated based on the descriptions given in section 2.3 by using the Force-displacement curves. Fig. 3 shows the mean values and the standard deviations of modulus of elasticity and hardness obtained from the nano-indentation experiment for each shade of the dental nanocomposite.

One way ANOVA test reveals significant differences existing in the modulus of elasticity values obtained for different shades (p-value<0.05). Subsequently, the Tukey HSD test is applied to evaluate pairwise differences between the mean values of modulus of elasticity for the shades of the dental nanocomposite. The results indicate a statistically significant difference between the mean modulus of elasticity values of WD and A1E shades (p-value=0.025). While, no significant differences are

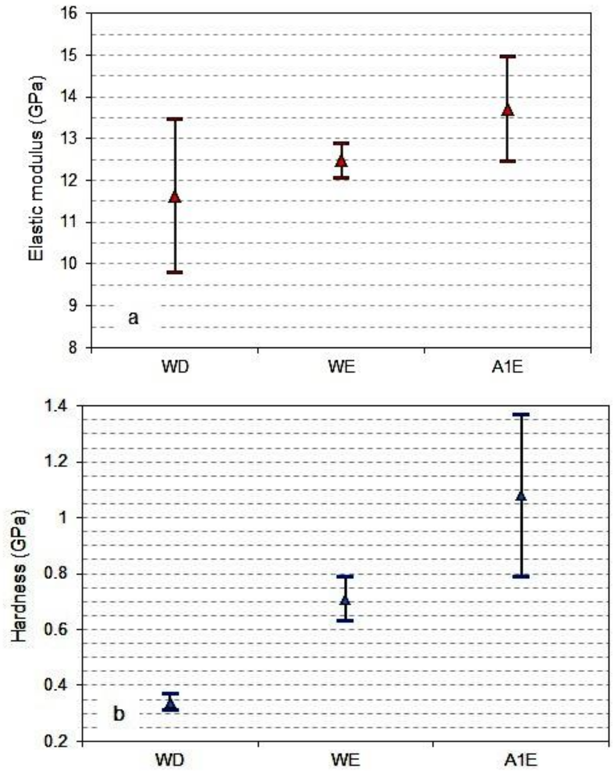

Fig. 3 (a) modulus of elasticity and (b) harness of different shades of the nano-composite samples obtained from the nano-indentation test

measured between the mean values of modulus of elasticity of the other shades. The same procedure was applied to the values of hardness measured for different shades. It was found that the hardness values vary significantly between all pairs of the nanocomposite shades as indicated by the Tukey HSD test.

As shown in Fig. 3, the highest values of hardness and modulus of elasticity belong to the A1E shade which can be related to the color of the shades. According to the dental nanocomposite data sheet (Askikfgajer *et al.* 2010), the A1E shade is darker than the other two shades. Since the capacity of light absorption is higher in the darker shades, they are expected to have better polymerization which leads to stiffer material with higher modulus of elasticity and hardness values (Aguiar *et al.* 2005), as observed for the A1E shade. The modulus of elasticity and hardness of WE shade are also larger than the corresponding values for the WD shade, which again can be due to its darker color.

Previous studies on the shade effects on the micro-hardness of dental composites and dental nanohybrid composites indicated that darker shades could have increasing or decreasing effects on the hardness value of the restorative composite (Aguiar *et al.* 2005, Price *et al.* 2005, Lodhi 2006). On the other hand, the size, percent content and distribution of particles affect the light scattering and its absorption, hence they have an influence on the polymerization behavior of restoratives (Aguiar *et al.* 2005). Since these factors vary from composite to nanocomposite restoratives, the mechanical properties of different material shades should be investigated

Table 2 Modulus of elasticity and harndess of thermocycled and non-thermocycled specimens obtained from the nano-indentation test

Sample preparation condition	Composite		Nanohybrid composite		Nanocomposite	
	Modulus of elasticity (GPa)	Hardness (GPa)	Modulus of elasticity (GPa)	Hardness (GPa)	Modulus of elasticity (GPa)	Hardness (GPa)
Non-thermocycled	11.46±0.46	0.46±0.05	13.77±0.46	0.72±0.07	12.48±0.42	0.71±0.08
Thermocycled	14.45±0.46	0.63±0.06	14.79±0.58	0.83±0.08	13.52±0.48	0.81±0.09

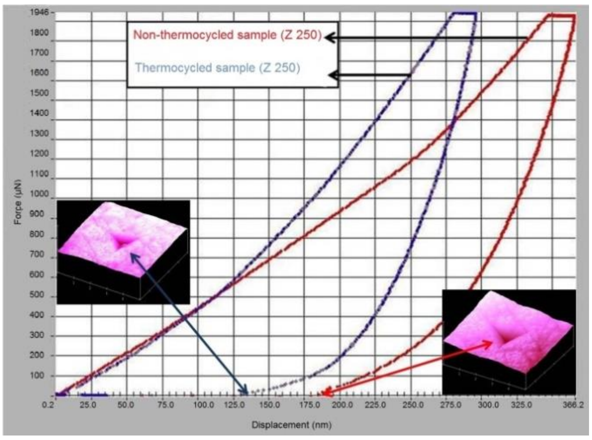

Fig. 4 Force-displacement curves of non-thermocycled and thermocycled samples of the conventional dental composite

independently for different types of dental restorative materials.

3.2 Thermocycling effect

3.2.1 Modulus of elasticity and hardness

The values of modulus of elasticity and hardness determined from the nano-indentation test for thermocycled and non-thermocycled samples are presented in Table 2. Each value is the average of data determined from five nano-indentation tests on a sample. For the non-thermocycled samples, it is observed that the modulus of elasticity and hardness of nanohybrid composite are more than those of the other restorative materials.

According to Table 2, the modulus of elasticity and hardness for all the samples thermocycled with 2000 cycles have increased compared to the non-thermocycled samples for each of the three dental materials. However, the thermocycling affects the conventional composite restorative sample more than the other two. The use of independent sample t-test for statistical analysis shows that a significant difference exists between the elastic moduli of non-thermocycled and thermocycled samples for all three restorative materials (p-value<0.05). However, based on statistical analysis, various degrees of significance are observed for thermocycling effects on the hardness of the dental restoratives. In the dental composite, thermocycling has significant effect on its hardness (p-value=0.002), while in the nanohybrid composite and in the nanocomposite no significant difference is seen between the hardness of non-thermocycled and thermocycled samples (p-value>0.05). Fig. 4 shows the force-displacement curves of a non-thermocycled specimen with a thermocycled specimen of the dental composite obtained from the nano-indentation test. The curves show that by increasing hardness, the maximum depth is reduced and the curves are shifted to the left.

To investigate the thermocycling effect on the mechanical properties of resin-based dental restoratives, two factors can be studied:

- The effect of moisture on dental restorative structure.
- Thermal stresses between the constituents of the materials caused by temperature changes during the thermocycling procedure.

Moisture can have influence on the mechanical properties of composites through two mechanisms. During the first mechanism, the water molecules penetrate in the matrix of composites expands its volume (Eftekhari and Fatemi 2016). This procedure results in a reduction in the stiffness of material.

The second mechanism is the dissolution of the composite components in water, which causes weakening of the surface properties. Previous research studies have indicated that storing the dental composites and dental nanocomposites in a humid environment has different effects on their mechanical properties depending on the temperature and the storage time. In some researches (Gladys *et al.* 1997, Sabbagh *et al.* 2002), keeping the specimens in water increased the modulus of elasticity and in some others (e.g., Papadogiannis *et al.* 2008), reduce the modulus of elasticity. Meanwhile, some of the researchers have also reported (Chung *et al.* 2005, Janda *et al.* 2006) that the storage of dental composites in water had no effect on the material modulus of elasticity. Thus, there is no common rule for the effects of water on the mechanical properties of the samples and the effects may depend on the material properties and the conditions of storage.

The presence of thermal stresses is another effective factor in thermocycling, which can strengthen the connections between the components of material and increase the modulus of elasticity and hardness.

The results presented in Tables 1-2 reveal that the modulus of elasticity and hardness of material have direct relationship with its filler loading. The nanohybrid composite with the highest filler loading has the highest modulus of elasticity and hardness, and vice versa for the conventional dental composite with the lowest filler loading. The modulus of elasticity of dental composite improves about 26% after thermocycling, while for the nanohybrid and nanocomposite the improvements are around 7% and 8%, respectively. The difference observed between the effects of thermocycling on the modulus of elasticity of composite in comparison with those of the nanohybrid composite and nanocomposite seems to be due to different types of resins used in these restorative materials.

3.2.2 Plasticity index

As mentioned earlier, the plasticity index (ψ) was used to study the elastic-plastic behavior of the samples. The plasticity index indicates the amount of irreversible work with respect to the total work done during the nano-

Table 3 The values of plasticity index of samples

Material	Plasticity index ψ	
	Non-thermocycled	Thermocycled
Conventional Composite	0.51±0.05	0.46±0.04
Nanohybrid composite	0.47±0.03	0.43±0.02
Nanocomposite	0.45±0.03	0.42±0.02

Table 4 Ratios of lateral force to normal force (LF/NF)

Material	L_F/N_F	
	Non-thermocycled	Thermocycled
Conventional Composite	0.25±0.01	0.28±0.01
Nanohybrid composite	0.28±0.01	0.30±0.02
Nanocomposite	0.27±0.02	0.29±0.01

indentation procedure. A lower plasticity index exhibits less residual penetration on the sample. Table 3 demonstrates the values of plasticity index determined for the dental restoratives.

For each restorative material, the plasticity index of the thermocycled sample is lower than that of the non-thermocycled sample.

The results presented in Tables 2-3 reveal that the plasticity index has an inverse relation with the modulus of elasticity. For example, the lowest value of plasticity index was observed for the non-thermocycled conventional composite which has the highest values of modulus of elasticity and hardness. The AFM images show that the residual penetration in the thermocycled sample is less than that of the non-thermocycled sample (see Fig. 2).

The reduction in the values of plasticity index due to thermocycling can be clearly seen in Table 3 for all the studied dental materials. This reduction indicates that thermocycling increases the amount of energy recovered during the unloading and actually, improves the elastic recovery of the surface. Thus, the residual penetrations on the surfaces of thermocycled samples after nano-indentation are less than those of the non-thermocycled ones and this can be a reason for the enhancement in the hardness value. Therefore, the thermocycling procedure changes the material behavior from ductile towards brittle.

3.2.3 Wear resistance

Fig. 5 illustrates the AFM images and the longitudinal scratch sections of the non-thermocycled and thermocycled specimens of the dental nanocomposite. As depicted in the AFM images of the scratch site, the residual depth of the scratch in the thermocycled sample is less than that of the non-thermocycled one. The AFM image of thermocycled sample shows that the material resisted against the sliding of indenter and pushed the tip upward during the scratch test. Thus, after thermocycling, a higher force is required to push the tip of indenter through the material if the penetration depth remains the same as that of before thermocycling. This indicates that thermocycling made the material more resistant against wear.

The scratchability or wear resistance of materials can be studied also by the ratio of lateral force to the normal force (L_F/N_F) obtained from the nano-scratch experiment. A higher L_F/N_F indicates lower scratchability and higher wear resistance of the sample. The values of this ratio for all samples are presented in Table 4. The results show that the load ratio (L_F/N_F) increases for all the studied dental materials after thermocycling. However, this increase is statistically significant only for the conventional dental composite (p-value=0.007), while no statistically significant difference is observed between the non-thermocycled and thermocycled specimens for the nanohybrid composite and for the nanocomposite (p-value>0.05).

The results presented in Tables 2-4 also show that the ratio of lateral force to normal force (L_F/N_F) has a direct relationship with the modulus of elasticity (and also with hardness). For example, among all the samples, the thermocycled nanohybrid composite has the highest values

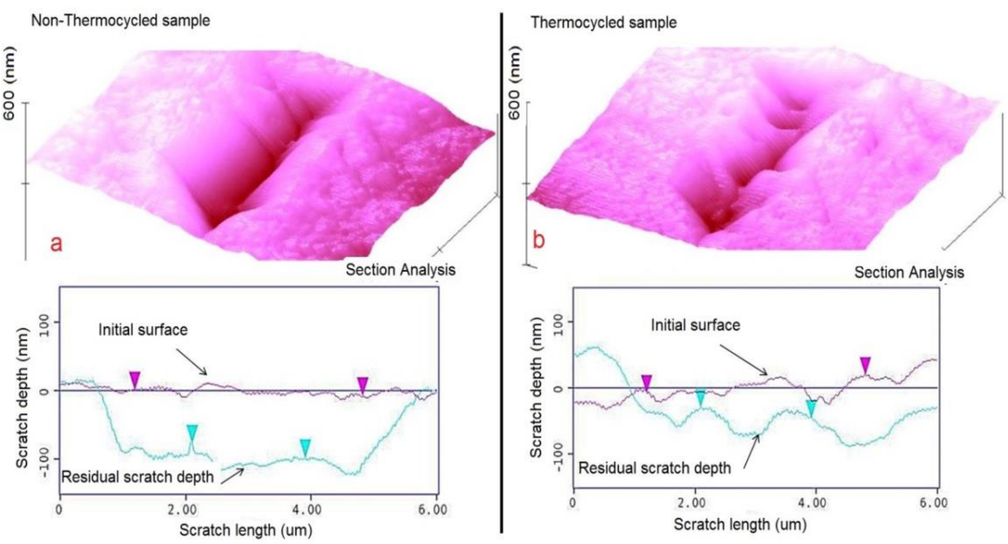

Fig. 5 AFM image and section of scratch in the dental nanocomposites: a) the non-thermocycled sample and b) the thermocycled sample

of modulus of elasticity and hardness and also the highest value of L_F/N_F. The experimental results obtained from the nano-indentation show an increase in both the modulus of elasticity and hardness, and a reduction in the plasticity index, which seems to be effective in the improvement of the elastic recovery properties.

The results of this study provide some useful information to clinicians for selecting appropriate types and shades of dental restorative materials. Considering the effect of temperature change on the mechanical properties and wear resistance of the resin-based dental restoratives, one may propose to apply the nanohybrid composite or the nanocomposite for dental restorations or orthodontic applications. Among the different shades of the nanocomposite, the A1E shade possesses higher modulus of elasticity and hardness. Thus, this shade may be selected for the cases wherein shade is not a critical issue, such as treatment of the posterior teeth.

4. Conclusions

The experimental results obtained in this research revealed that the highest values of hardness and modulus of elasticity were related to A1E shade of the dental nanocomposite, which were equal to 1.08 GPa and 13.71 GPa, respectively. For all types of the studied resin-based dental materials, the elastic moduli of samples subjected to 2000 cycles of thermocycling increased at least 8% compared to those of the non-thermocycled samples. Moreover, it was observed that thermocycling had no significant effect on the hardness, plasticity index and wear resistance of the nanohybrid composite and nanocomposite. The improvements in the modulus of elasticity and hardness of the restorative composite due to thermocycling were around 30 percent, which were more than the other two dental restorative materials. The differences observed in the level of change in mechanical properties can be interpreted by the difference in the volumes of fillers and the types of resins used in the resin-based dental composite materials.

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