

## Nanohydroxyapatite and Novamin on Roughness and Discoloration Following Orthodontic Adhesive Removal

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### Abstract

**Objective:** The purpose of the study was to assess the impact of two types of toothpaste containing Nanohydroxyapatite and Novamin on teeth surface roughness and teeth discoloration following orthodontic brackets debonding and adhesive removal. **Material and Methods:** Eighty human premolars were extracted before starting orthodontic treatment and used as the samples for the current study. All samples were attached with metal brackets and then divided at random into two groups (n=40) by using toothpaste containing either Nanohydroxyapatite (Lacalut white and repair toothpaste) (LSH) and toothpaste containing Novamin (Sensodyne repair and protect) (LSN). The adhesive removal was done using a stain-buster bur system and wearing dental Loupes at a low-speed handpiece. All the colors of the teeth were first evaluated with a VITA easyshade® spectrophotometer. In addition, enamel surface roughness measurements were done using Stylus Profilometry (Surface roughness tester Time®3200). The records were done as first measurements (E1) and (R1). The second records for the teeth (E2) and (R2) were done after adhesive removal. A third record was done after the staining procedure by immersion of teeth in black tea as (E3) and (R3). Finally, the fourth measurements were carried out after brushing the teeth with Novamin toothpaste and Nanohydroxyapatite toothpaste as (E4) and (R4). **Results:** The outcomes were analyzed statistically via T-test ( $P \leq 0.05$ ). The results displayed a statistically considerable effect in reducing the enamel surface roughness for both groups (LSH) and (LSN), with no difference between the two groups. In addition, both groups had a statistical impact on changing the

color of the teeth, with a higher effect of (LSN) group than the (LSH) group. **Conclusion:** Novamin and Nanohydroxyapatite toothpaste had a considerable impact in reducing enamel surface roughness. However, Novamin toothpaste showed better results than Nanohydroxyapatite in reducing enamel discoloration.

### Open Access

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### Introduction

The protective outer surface of enamel consists of biomineral, tiny quantities of proteins and water. It is a barrier to the occlusal region, allowing the teeth to resist the constant impact and shear stresses they receive [1]. Enamel is a mineralized human oral cavity tissue (around 85% hydroxyapatite (HAP) crystals as an inorganic phase, 3% biomacromolecule as organic, and an essential 12% water) [2].

People's assessment of their malocclusions and tooth abnormalities has risen as they have become more aware of facial and dental aesthetics, which leads to growing numbers of young and older individuals seeking orthodontic treatment for "aesthetic purposes [3,4]. A bracket is a part of the fixed orthodontic appliance. Attaching a bracket involves multiple stages, including cleaning and etching the tooth enamel, conditioning the surface, and bonding [5].

Acid etching is essential for connecting brackets to the tooth surface. It will result in micropores, allowing resin material to penetrate the enamel and micromechanical retention, enhancing bonding strength. However, these steps result in roughness and the degradation of hydroxyapatite minerals [5]. After the end of fixed orthodontic therapy, brackets are mechanically debonded, and the remaining adhesive resin must be physically removed to prevent biofilm

accumulation and discoloration [6]. The roughening of enamel caused by adhesive clearance may result in discoloration. Adhesive residuals may alter enamel color due to internal changes generated by the adhesive resin's physicochemical interaction and outward changes caused by the superficial absorption of food coloring agents [7].

Unfortunately, orthodontic therapy may have negative consequences on the teeth' surfaces. These include enamel damage due to etching and enamel surface modifications resulting in demineralization [8].

Studies revealed that the rates of plaque in the mouth of orthodontic patients are two to three times greater than in average persons with high biofilm production rates [9]. Continuous cycles of demineralization and remineralization occur in dental tissue. Decreased oral pH leads to demineralization, which may cause mineral loss from the enamel composition. If the pH increases, calcium, phosphate, fluoride, and other agents will accumulate, causing a rebound [10]. Tooth brushing with a polyvalent paste seems to be the most effective strategy for plaque reduction, salivary mineral resource acquisition, and enamel remineralization [9]. Novamin is a fabricated mineral with a near structure to enamel minerals that may aid in remineralizing enamel structures that have been destroyed. In the form of calcium sodium phosphosilicate, Novamin is composed of calcium, sodium, phosphorus, and silica. The silica in Novamin may release crystalline hydroxyl-carbonate apatite (HCA), which has a composition similar to that of a mineral in teeth; in addition, Novamin Particles are less than 20 microns in size, all of these enhance enamel remineralization [11]. Novamin might aid in remineralizing demineralized enamel on teeth after orthodontic therapy due to the presence of CPP-ACP (casein phosphopeptide-amorphous calcium phosphate), nano complexes such as carbonate-hydroxyapatite nanocrystals and calcium sodium phosphosilicate [12]. Novamin permits calcium and phosphate to be liberated from the surface. These crystals repair minute surface flaws, resulting in firmer, smoother, less sensitive teeth. Additionally, this substance reduces surface protein precipitation, reducing pigment's capacity to attach to teeth over time [13].

On the other hand, enamel cannot regenerate, unlike other natural bone and dentin [14]. Enamel comprises 20–40 nm-sized hydroxyapatite particles (HAP) [15]. Due to its near resemblance to the fundamental structures of enamel, it has been proposed that 20-nm-sized HAP, also known as nanohydroxyapatite (nanoHAP), is

the most biocompatible and bioactive of the manufactured apatites [16]. NanoHAP has a significant attachment to the tooth surface. Compared to the broad and amorphous varieties, these properties allow adhesion to the tooth surface. Fascinatingly, a layer of nanoHAP produced on the surface of the tooth is very rigid to the acid solution, thus protecting the tooth from upcoming decalcification [17].

## Material and Methods

### Sample preparation

Two hundred fifteen extracted human maxillary first premolars that had been removed from individuals undergoing orthodontic treatment were gathered.

After extraction, the teeth were washed with water to remove any traces of blood. Each tooth was thoroughly rinsed to remove soft tissue remnants and debris [18].

The samples were picked after checking by 10X magnifying lens and visible light cure machine [19], and those that had the following criteria from the presence of internal stains, enamel decalcifications, decay, fluorosis, enamel defects, restorations and cracks were excluded.

As a result, eighty premolar teeth have been utilized in this research. The samples were prepared and set as every three teeth were placed vertically on a stone block to the level that only the buccal surface of the crowns could be seen. The samples were then coded 1 to 80. The buccal area of the samples was brushed using non-fluoridated pumice and a nylon brush. After that, the samples were kept in closed containers with deionized water. Up to the day of bonding, the deionized water was changed every day to reduce dehydration and bacterial development.

Over the buccal surface of the crown, each sample had a square piece of paste tape positioned on it, and a circular aperture was cut out of the tape, which allowed a working area for bonding, adhesive removal, color analysis using a spectrophotometer, and surface roughness measurements.

### Bonding, debonding the brackets and adhesive cleaning

The samples were bonded via Transbond XT and the standard acid etching technique. Each tooth was etched with 37% phosphoric acid for 15 seconds, sprayed with water and air for 20 seconds, then dried in the air for 10 [7]. Through using a disposable brush, a light coating of Transbond XT primer was applied to the etched tooth surface.

Each sample was supplied a maxillary first premolar bracket Equilibrium® 2 type (Dentaurum company, Ispringen, Germany) with the base of the bracket coated with a smear layer of isolation medium (vaseline) and then lined with adhesive composite. The

brackets were then positioned in the working area and pressed on the tooth surface using bracket holder tweezers.

For 20 seconds, the resin on the bracket was exposed to the curing light (10 seconds from the occlusal side and 10 seconds from the gingival side) [20].

Following bracket bonding, the specimens were kept in artificial saliva within sealed containers in an incubator at 37°C for one week to reach equilibrium in their water sorption and to resemble the oral cavity environment. The saliva changed every three days [5].

Then, each bracket was removed using bracket holder tweezers, leaving a uniform rectangle of bonded composite resin approximating the mesh of the maxillary first premolar bracket base.

Then, the samples were separated randomly into two groups (LSN) and (LSH), consisting of 40 teeth according to the toothpaste type that was used (either Nanohydroxyapatite or Novamin toothpaste) to brush the teeth. Following that, each group was cleaned from the adhesive material using a method of wearing dental loupes (Ergo custom-made dental loupes 6X with led light (Admetic, United States)) and using (Broca em zircônia multilamina) stainbuster zirconia multi-blade bur (Morelli Ltda, Brazil) in low-speed contra-angle handpiece from 10000 to 20000 RPM, then the teeth polished with (Ponta descartável) disposable burs for finishing (Morelli Ltda, Brazil).

The adhesive removal was accomplished when the enamel surface was smooth to the touch, and dental loupes and chair light showed no evidence of resin.

Then, a staining method was prepared by collecting a cup of 250 ml of freshly boiled water and one black teabag. Then, all the samples were immersed in the prepared black tea 55°C solution every day for 10 minutes and kept 24 hours in 37 degrees Celsius distilled water. The entire process took 30 days [21].

### Teeth brushing

After the staining procedure was completed, the samples in the collection (LSN) were exposed to Novamin (Sensodyne repair and protect toothpaste), while the (LSH) group was subjected to Nanohydroxyapatite toothpaste (Lacalut white and repair toothpaste) as an enamel remineralization material. The brushing protocol for all groups was carried out as a Pea-sized (0.25g) amount of toothpaste on a toothbrush used to brush five teeth. Each tooth was brushed for 15 seconds with a soft-bristled Rotary toothbrush (Oral-B Electric rechargeable toothbrush), fixed at a constant distance by the holder. After brushing, the tooth was maintained in contact with the toothpaste slurry for an

additional 165 seconds, so the total time was 3 minutes for each tooth. Then, the tooth was rinsed with distilled water by triple syringe, and the samples were kept in artificial saliva within sealed containers in an incubator at 37°C. These procedures were carried out every 12 hours for 30 days, and the artificial saliva was changed every three days [5,22].

#### Color measurements

The spectrophotometer used was VITA easyshade®. The device utilizes the CIE  $L^*a^*b^*$  value. The measurements were initially done for the samples after the adhesive removal protocol; these were verified as the first gauge value (E1) and followed by a second gauge value for the samples (E2) after the staining procedure. Then, a third measurement after thirty days of brushing was recorded as (E3). Before every measurement, the calibration was repeated. However, to minimize the potential for inaccuracy, three separate values were measured and averaged. The level of measurement differences in tooth color was evaluated using the device's  $L^*a^*b^*$  and  $\Delta E$  values. The  $L^*$  value, representing the degree of whitening, ranges from 0 (black) to 100 (white), whereas the  $a^*$  value indicates the red (+) and green (-), and the  $b^*$  value indicates yellow (+) and blue (-) axes. The following formulation was used to calculate the difference between the two colors [23]

$$\Delta E = \{(L2-L1)^2 + (a2-a1)^2 + (b2-b1)^2\}^{1/2}$$

$\Delta E$  1: The difference between the recorded results attained after adhesive removal and the final recorded results after thirty days of tea submersion. Clinically describes the discoloration that develops after therapy.

$\Delta E$  2: The change among the results attained next thirty days of tea soaking and after thirty days of teeth brushing. Represents the clinical capacity of toothpaste and brushing to restore natural enamel color.

In addition, the color change was evaluated for all the samples by a single blind evaluator at four-time points for  $L^*$ ,  $a^*$  and  $b^*$  values:

- After adhesive removal ( $L^*1$ ,  $a^*1$  and  $b^*1$ ).
- After the Staining procedure through tea submersion ( $L^*2$ ,  $a^*2$  and  $b^*2$ ).
- After Teeth brushing ( $L^*3$ ,  $a^*3$  and  $b^*3$ ).

#### Surface roughness measurements

A surface profilometer (Time®3200 Surface Roughness Tester; TIME Group Inc, Beijing, China) was used in this research with a microneedle to scan the surface. The outcomes were indicated by the measure "roughness average" (Ra). Average roughness value (Ra): the arithmetic means of the peak height and valley depth from a mean line. This parameter specifies the total surface roughness in micrometers ( $\mu m$ ). Typical Enamel surface roughness (ESR) ranges between 0.59 to 0.66  $\mu m$  [24]. The device was set with a 0.25 mm cutoff and 0.1 mm/s speed. Calibration

was checked with a standard ( $Ra = 1.57 \mu m$ ) after every ten samples. The surface roughness of each specimen was calculated in three points; the mean value of the three points was considered as the mean surface roughness (Ra) (Figure 1).

Surface roughness was evaluated for all the samples by a single blind evaluator at three-time points:

- After adhesive removal (R1).
- After the Staining procedure through tea submersion (R2).
- After Teeth brushing (R3).



Figure 1. Stylus surface roughness tester time 3200 for measuring the surface roughness (Ra) for the samples.

#### Statistical analysis

The statistical analysis was done using IBM SPSS V26 and Minitab V.17. The mean and standard deviations (S.D.), Standard error (S.E.), Minimum (Min.), and maximum (Max.) values were recorded. Independent t-test was used to analyze the data. The level of significance was fixed at 5%, and  $P \leq 0.05$  was considered statistically significant.

### Results

#### Analysis of the enamel surface roughness (ESR)

There was no significant difference (SD) in the ESR level for all the samples between R1 and R2 time points. However, there was a SD in the ESR plane for all the samples between the (R1) and after-teeth brushing (R3) time points by two types of toothpaste. The enamel surface roughness level reduced after teeth brushing from (0.590 to 0.478  $\mu m$ ). Moreover, there was no SD among the (LSN) group and (LSH) after the adhesive removal time point (R1).

Furthermore, there was no SD among the (LSN) group and (LSH) after the teeth brushing time point (R3).

These results showed that both toothpastes (Novamin and Nanohydroxyapatite) reduced the ESR at the same level (no one was better than the other) (Table 1).

Time points	Groups	No	Mean	S.D	t-test	P-value
R1 versus R2 for all the teeth	R1 for LSN+LSH	80	0.590	0.212	1.01	0.312
	R2 for LSN+LSH	80	0.560	0.158		
R1 versus R3 for all the teeth	R1 for LSN+LSH	80	0.590	0.212	3.85	0.001
	R3 for LSN+LSH	80	0.478	0.151		
R1 for LSN versus R1 for LSH	R1 for LSN	40	0.587	0.180	0.43	0.669
	R1 for LSH	40	0.570	0.190		
R3 for LSN versus R3 for LSH	R3 for LSN	40	0.506	0.178	-0.43	0.672
	R3 for LSH	40	0.488	0.200		

Table 1. T-test to compare the Ra for the groups (LSN) and (LSH) at different time points.

#### Descriptive statistics of the enamel discoloration

The color variation depends on ( $\Delta E$ ) in addition to ( $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ ).

The results of the color assessment in this study depend on  $\Delta E$  values,  $L^*$  records,  $a^*$  records and  $b^*$  records. 3.7  $\Delta E$  units were normally assigned as the clinical diagnostic value of color changes. [26].

Evaluation of teeth color differences at  $\Delta E1$  and  $\Delta E2$ :

After immersion of all the samples in black tea (staining procedure), the discoloration was considered highly clinically distinguished compared to after the adhesive removal period because the  $\Delta E1$  was (31.713) units (Table 2). Moreover, after teeth brushing for all the samples with two kinds of toothpaste, the mean of ( $\Delta E2$ ) was considered to have very high clinical significance (31.37 units) concerning (3.7) units clinical threshold value (Table 2) and (Figure 2).

Table 2.  $\Delta E1$  and  $\Delta E2$  measurements for all the samples.

Color change	No	Min.	Max.	Mean	SD	SE
$\Delta E1$	80	16.266	61.035	31.713	11.545	0.75
$\Delta E2$	80	16.266	61.035	31.713	11.545	0.75

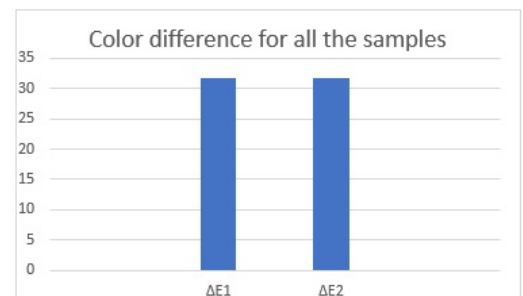


Figure 2. Discoloration means levels of the teeth ( $\Delta E1$ ,  $\Delta E2$ ).



### Assess which toothpaste had a better effect on teeth re-coloring after the staining procedure

There was a highly significant effect between the (LSH) group with  $\Delta E2$  mean of 28.281 units and the (LSN) group with  $\Delta E2$  mean of 34.472 units. This result meant Novamin toothpaste greatly influenced re-coloring enamel after tea submersion than Nanohydroxyapatite toothpaste. The difference between the two  $\Delta E2$  was 6.192 units, which exceeded the clinical threshold value of 3.7 units (Table 3).

Table 3. Descriptive statistics of the enamel discoloration ( $\Delta E2$ ) between (LSH) and (LSN) groups.

Discoloration Groups	No	Min.	Max.	Mean	S.D	S.E
$\Delta E2$						
LSH	40	12.60	55.34	28.281	9.73	1.53
LSN	40	16.11	58.86	34.472	10.90	3.44
T-Test				3.79		
P-value				0.000		

However, according to the T-test, the comparison occurred horizontally between two groups to measure the  $L^*$  value. There was a SD at the  $L^*2$  time point between the group (LSH), which had 60.27 units and the group (LSN), which had 53.867 units. This result meant the group (LSH) had a significantly whiter teeth color than the group (LSN) by (6.403) units. In addition, at the time point  $L^*3$ , there was no SD among the two groups, and both groups were at the same luminosity standard.

However, according to the T-test, the comparison occurred vertically for the same group to measure the  $L^*$  value at two different time points. For the (LSH) and (LSN) groups, there were significant differences between the  $L^*2$  and  $L^*3$  time points, but the Novamin toothpaste moved the whitening level of the teeth 5.795 units greater than the Nanohydroxyapatite toothpaste (Table 4).

Table 4. T-test for the statistical analysis of ( $L^*$ ) values in the (LSH) and (LSN) groups at two different time points ( $L^*2$  and  $L^*3$ ).

Time points	Groups				T-test	P-value
	N	LSH	LSN			
		Mean S.D.	Mean S.D.			
After tea submersion ( $L^*2$ )	40	60.27 9.51	53.86 11.27	3.88	0.00	
After teeth brushing ( $L^*3$ )	40	85.66 3.68	85.05 3.06	1.14	0.25	
T-test		22.26	23.87			
P-value		0.000	0.000			

In addition, according to the T-test, the comparison occurred horizontally between two groups to measure the ( $a^*$ ) value. There was a significant difference at the  $a^*2$  time point between the group (LSH), which had 7.73 units and the group (LSN), which had 9.687 units. This result meant the group (LSN) had a significantly redder teeth color than the group (LSH). In addition, at the time point

$a^*3$ , there was no SD among the two groups, and both groups were at the same red-green standard.

However, according to the T-test, the comparison occurred vertically for the same group to measure the ( $a^*$ ) value. For the (LSH) and (LSN) groups, there were significant differences between the ( $a^*2$ ) and ( $a^*3$ ) time points, but the Novamin toothpaste reduced the red color level of the teeth by (2.087) units greater than the Nanohydroxyapatite toothpaste (Table 5).

Table 5. T-test for the descriptive statistics of ( $a^*$ ) values in the (LSH) group and (LSN) group at two different time points ( $a^*2$  and  $a^*3$ ).

Time points	Groups				T-test	P-value
	N	LSH	LSN			
		Mean S.D.	Mean S.D.			
After tea submersion ( $a^*2$ )	40	7.73 3.30	9.68 3.63	2.52	0.014	
After teeth brushing ( $a^*3$ )	40	-0.7 1.245	-0.83 0.93	0.53	0.599	
T-test		15.12	17.73			
P-value		0.000	0.000			

Moreover, according to the T-test, the comparison occurred horizontally between two groups to measure the ( $b^*$ ) value. There was no SD at the ( $b^*2$ ) and ( $b^*3$ ) time points between the group (LSH) and the group (LSN) (both groups were at the same yellow-blue standard). However, according to the T-test, the comparison occurred vertically for the same group to measure the ( $b^*$ ) value. For the (LSH) and (LSN) groups, there were significant differences between the ( $b^*2$ ) and ( $b^*3$ ) time points, but the Novamin toothpaste reduced the yellow color level of the teeth by 1.57 units greater than the Nanohydroxyapatite toothpaste (Table 6).

Time points	Groups				T-test	P-value
	N	LSH	LSN			
		Mean S.D.	Mean S.D.			
After tea submersion ( $b^*2$ )	40	35.33 3.52	35.91 2.33	0.87	0.38	
After teeth brushing ( $b^*3$ )	40	27.29 5.23	26.30 3.33	1.00	0.31	
T-test		8.06	14.92			
P-value		0.000	0.000			

Table 6. T-test for the descriptive statistics of ( $b^*$ ) values in the (LSH) group and (LSN) group at two different time points ( $b^*2$  and  $b^*3$ ).

## Discussion

### Evaluation of Tea Impact on ESR and Color

The present result shows that the tea had no considerable impact on ESR, which agrees with previous studies [25]. This is because sugar-free black tea has an anti-erosion effect because of its high pH values, respectively 5.7 and 6.3, which are higher than the

enamel threshold P.H. (5.5). While the longer the enamel was exposed to solutions with a low pH (pH 2.0–4.0), the greater the danger of erosion.

Moreover, the discoloration was considered highly clinically distinguished compared to after the adhesive removal period, which may refer to the long period (30 days) for the samples immersed in black tea. Moreover, the adhesive cleaning procedure leads to irregularities in the enamel surface, increasing the chances of retaining beverage pigments [26].

### Evaluation of toothpaste effect on ESR

In orthodontics, bonding of brackets to the tooth surface requires an acid etching procedure. Consequently, microporosity will allow the resin substance to penetrate the enamel structure and cause micromechanical attachment, increasing the adhesion strength. However, this procedure damages hydroxyapatite and raises enamel surface roughness [5].

This research showed that there was a SD in reducing enamel surface roughness after thirty days of brushing the teeth with Nanohydroxyapatite toothpaste and Novamin toothpaste. Moreover, there was no SD among the two types in reducing the ESR level (both kinds of toothpaste had the same effect level).

This result aligns with Utari et al. (2021), which concluded that remineralization of the tooth surface with Novamin-containing toothpaste reduces enamel surface roughness [5]. In addition, Jumanca et al. (2019) concluded that utilizing toothpaste with Nanohydroxyapatite, such as Lacalut white and repair toothpaste and Sensodyne repair and protect toothpaste efficiently treats demineralized enamel surfaces and fixes minor tooth surface defects and made a preservative coat on the etched enamel surface [27].

### Evaluation of toothpaste effect on enamel color change

By comparing the two types of toothpaste used in this study, the Novamin toothpaste greatly affected enamel re-coloring after tea submersion than Nanohydroxyapatite toothpaste. The difference between the  $\Delta E3$  for the two types of toothpaste was (6.192) units, which exceeded the clinical threshold value. The teeth sample for the Novamin toothpaste had more lightness at about (5.79) units, less red at about (2.08) units and less yellow color level by (1.57) units than the teeth samples of Nanohydroxyapatite toothpaste.

Instead of the amount of their whitening ability, It seems that the mechanical action of removing surface tooth colors is mostly attributable to the physical properties of the minerals present in the composition of toothpaste. Mechanical friction, such as silica

and other cleaners, combined with remineralization chemicals, primarily eliminates extrinsic coloring, giving an impression of whitening [10]. In their study, Jumanca et al. (2019) aimed to show the value of employing Nanohydroxyapatite-containing toothpaste on human teeth and in what way it works at the microscopic seen by capping the etched enamel. The result was that Sensodyne toothpaste was the maximum active in precipitating an intense coat on the tooth surface, a coat that shields and improves the tooth defects. At the same time, the Lacalut toothpaste precipitated a thin coat, and the enamel prisms could be noticed as not completely capped.

The present result aligns with other research that used Several types of sensitive-relief toothpaste and found toothpaste with Novamin increased the L\* value after simulating brushing with an electric toothbrush (Braun Oral-B Advance Power) and immersion in coffee, making natural teeth and composites seem brighter [28].

## Conclusion

1- Black tea (without sugar) did not affect ESR but highly affected teeth discoloration.

2- Both Novamin and Nanohydroxyapatite-containing toothpaste had a considerable impact in reducing ESR. However, Sensodyne repair and protect toothpaste showed better results than Lacalut white and repair in reducing enamel discoloration.

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