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INFLUENCE OF FOOD SIMULATING FLUIDS AND THERMOCYCLING ON SURFACE ROUGHNESS AND WEAR RESISTANCE OF DENTAL COMPOSITE RESTORATIVES

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ABSTRACT

Objective: The aim of the investigation was to evaluate the effect of food simulating fluids and thermocycling on surface roughness and wear resistance of CAD/CAM (Lava™ Ultimate, 3M ESPE), (SR Nexco® Paste, Ivoclar Vivadent) and (Filtek™ Z350 XT, 3M ESPE) resin composites.

Methods: A total of 360 disks were fabricated and divided into three groups. Each group was divided into 12 subgroups (n=10); 6 subgroups for measuring surface roughness and the other 6 subgroups for measuring wear resistance. Four subgroups are immersed for 1 month in different food simulating fluids (distilled water, ethanol, heptane and citric acid). The fifth subgroup was thermocycled for 5000 cycle, while the control subgroups were examined after 24 hours post-polymerization. Surface roughness was assessed using environmental scanning electron microscope. Wear resistance testing was carried out by custom made toothbrush machine. The results were tabulated and statistically analyzed.

Results: The results revealed that the food simulating fluids and thermocycling had a deteriorating effect on the three tested materials. Lava™ Ultimate showed the least significant deterioration when compared to SR Nexco® Paste and Filtek™ Z350 XT.

Conclusion: Lava™ Ultimate had better resistance to both food simulating fluids and thermocycling, which may be due to its higher nanofillers percentage and its highly cross linked matrix.

KEYWORDS: Direct and indirect resin composites, CAD/CAM, food simulating fluids, surface roughness, wear resistance.

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INTRODUCTION

Esthetic dentistry is a global phenomenon that continues growing over the past years denoting enormous changes. In today's world, looking good is a prime concern. The emphasis of dentistry in the present times is not only on prevention and treatment of disease, but on meeting the demands for better esthetics. The evolution of esthetic restorative materials, such as dental resin composites and ceramic materials, in collaboration with the adhesive dentistry allowed a sort of coping with such increasing demands. Many modifications have been introduced to the resin composite restorations such as in fillers size, organic matrix composition (1).

In principle, the growing interest in nanotechnology and its application in resin composites was presented in using nanosized particles to alter the structure of such restorative materials. Hence improving the mechanical, chemical, and optical properties of those substances (2)**.**

Accordingly, Mitra and others introduced novel nanofillers and then employed various methacrylate resins and curing technologies to develop nanocomposites. Two classes of resin composites that include nanoscale filler particles in their composition have been introduced namely, nanofilled and nanohybrid resin composites. While nanofilled composites use nanosized particles throughout the resin matrix, nanohybrids include a mixture of nanosized and conventional filler particles (3).

Generally, dental restorative composite materials are classified into direct and indirect composites. Due to the limitations of the direct resin composites, indirect resin composites were developed. The latter are the restorations fabricated outside the oral cavity. Although the indirect method requires more tooth structure removal, extra time and cost that may all be beyond patients' desire, but still the indirect resin restorations can overcome some limitations of the direct ones. Such as polymerization shrinkage and its destructive sequelae, since the polymerization shrinkage is limited to the luting cement. Moreover, the survival rate of indirect resin composite restorations was higher than the direct restorations. They also would offer an esthetic substitute to ceramic materials for the posterior fillings (1).

Furthermore, the indirect resin composite restorations can be classified according on fabrication techniques into direct-indirect/semi-direct method, indirect fabrication technique and CAD/CAM technique. What's more, in order to increase the survival rate of indirect composite restoration, many products were introduced with modifications in both the matrix and the fillers. Among those is resin nano ceramic restorations which are supplied the form of blocks to be milled via CAD/CAM milling machine (1). It was claimed that such material contains clusters of silanated nano ceramic particles imbedded into a highly cross linked resin matrix, combining the properties of the two basic aesthetic restorations; resin composites and ceramic materials (1,2)**.**

Worth to mention that, a desirable property for a restorative material is the high wear resistance. Clinically, the wear of a restoration may result from the functional contacts, the interproximal contact areas, the attrition of food bolus, as well as toothbrushing. Wear is a complex process, since it involves abrasion, adhesion, fatigue, erosion and friction, interacting collectively. The resistance to wear of a material can be evaluated through its mass loss and superficial smoothness, after a certain period of toothbrushing. One of the dramatic problems of rough surfaces is that, it can lead to the increased plaque retention and staining. The surface texture can also influence the esthetical properties of composite resins, since it affects the light reflectance and the appearance of such materials (4).

The undeniable question for every modification is its long term longevity in the complex oral environment. Many aging methods were suggested to simulate the degrading nature of oral cavity. FDA recommended aging of polymeric materials by immersion into four types of fluids that simulate the oral beverages and food; distilled water, ethanol, heptane and citric acid.

Consequently, the aim of this study was to evaluate the surface roughness and wear resistance after immersion in food simulating fluids and thermocycling over three composite resin materials; nanofilled resin nano ceramic, indirect microhybid resin composite and direct nanofilled resin composite.

MATERIAL AND METHODS

Grouping of the specimens

A total of 360 samples were fabricated from three types of dental resin composite restorative materials and divided into three groups (120 samples in each group). Group 1 samples were machined from a resin nano-ceramic CAD/CAM blocks (Lava™ Ultimate, 3M ESPE, USA), group 2 samples were fabricated from an indirect micro-hybrid resin composite (SR Nexco® Paste, Ivoclar Vivadent, Liechtenstein) and group 3 samples were fabricated from a direct nanofilled resin composite (Filtek™ Z350 XT, 3M ESPE, USA).

Samples of each group were randomly divided into 12 subgroups (n=10); 6 subgroups were tested for surface roughness, while the other 6 subgroups were tested for wear resistance. For each test, four subgroups were immersed in four food simulating fluids (S_1: distilled water, S_2: 75% ethanol, S_3: heptane and S_4: 0.02N citric acid), S_5 samples were thermocycled, while S_0 subgroup samples used as control samples. Samples immersed in food simulating fluids were kept in tightly sealed glass test tube for 1 month. Thermocycled samples were subjected to 5000 cycle (temperature between 55 and 5 ° C) for 30 second in each path with 5 seconds delay time. The control samples were left in the air at room temperature (25-30°C) for 24 hours to maximize polymerization of the samples then subjected to testing.

TABLE (1): The materials, compositions and manufacturers of the materials employed in this study.

Group	Material (commercial name)	Composition (wt %)	Manufacturer (Lot No.)
Gp 1	CAD-CAM resin nano ceramic blocks (Lava™ Ultimate)	Highly cross linked polymeric matrix of UDMA[1] and TEGDMA[2] • Fillers: (80% wt) - Non-agglomerated, non-aggregated 20 nm silica fillers - Non-agglomerated, non-aggregated 4:11 nm zirconia fillers - Aggregated zirconia/silica cluster fillers	3M ESPE, USA (N842943) Shade: A3.5
Gp 2	Indirect Micro-hybrid resin composite (Nexco®Paste)	• Aromatic aliphatic UDMA +Aliphatic dimethacrylates (17% wt) • Highly dispersed silicon dioxide (19.7% wt) • Prepolymer/Copolymer (62.8% wt) • Catalysts and stabilizers (0.3% wt) • Pigments (0.1% wt)	Ivoclar Vivadent, Liechtenstein (R32664) Shade: A3.5
Gp 3	Direct nano-filled resin composite (Filtek™ Z350 XT)	Matrix of Bis-GMA[3], UDMA, TEGDMA, PEGDMA[4] and bis-EMA[5] • Fillers: (72.5% wt) - Non-agglomerated, non-aggregated 20 nm silica fillers - Non-agglomerated, non-aggregated 4:11 nm zirconia fillers - Aggregated zirconia/silica cluster fillers 0.6: 10 microns.	3M ESPE, USA (N436589) Shade: A3.5

[1] UDMA: Urethane dimethacrylate, [2] Triethylene glycol dimethacrylate, [3] bisphenol A glycidyl methacrylate, [4] polyethylene glycol dimethacrylate [5] Bisphenol A polyethethylene glycol diether dimethacrylate

Samples preparation

Disks with dimensions of (8mm X 2mm) were fabricated. In group 1, the blocks were cut into cylinders using lathe machine, with a low speed diamond disk (BesQual, NY 11373, USA) under profuse cooling. The disks were polished using fine and extra-fine Sof-Lex™ disks (3M ESPE, USA). In group 2 and 3, the resin composite was packed into a Teflon mold (8mm X 2mm), pressed between tMylar strips and glass slaps from both sides to minimize the oxygen inhibited surface layer. Samples were pre-cured using LED light curing unit for 20 seconds with light intensity 1500 mW/cm^2 (Radii Plus, SDI Limited, Australia). Light intensity was checked before curing the specimens. Samples of group 2 were post-cured using manufacturer supplied light curing furnace (Lumamat® 100) for 25 minutes.

Surface roughness measuring

Surface roughness was measured using Environmental Scanning Electron Microscope (ESEM) (Quanta 200, FEI, multinational gathered at Netherlands). Roughness value is represented as (Ra) that describes the arithmetic mean of all values of the roughness profile over the evaluated length using the software XT document (**5**).

Weight loss due to wear

Wear test was made using a custom made wear machine that delivered a wearing action to the samples with a soft bristles toothbrush head (Fuchs®, Fuchs Oral Care Gmbh, Egypt). Each wear cycle in done with a new toothbrush head loaded with 200 gm weight with rate: 280 cycles/minute for 100 minutes under immersion of toothpaste slurry (diluted with water with 1:1 ratio) toothpaste (Signal, Unilever Mashreq, Egypt) that is changed with each cycle. After completion of wear cycle, each sample was rinsed thoroughly to remove all traces of the slurry then dried.

The samples were weighted before and after wear cycles in a digital analytical balance with 0.0001 gm accuracy (AND, HR-200 Maz.210g, Japan) The loss of weight (ΔW) for each sample was calculated by the difference between weight before (W_1) and after wear (W_2) testing from the following equation (**4**): $\Delta W = W_1 - W_2$

Statistical analysis

Data was presented as mean and standard deviation (SD) values. Data was explored for normality using Kolmogorov-Smirnov tests for Normal distribution, data showed normal distribution so; One Way ANOVA was used to study the effect of different resin composites within each storage variable and the effect of different storage variable within each composite resin material. Duncan's post-hoc test was used for pair-wise comparison between the mean when ANOVA test was significant.

Pearson's correlation used to correlate between different tested parameters. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM® SPSS® (SPSS Inc., IBM Corporation, NY, USA) Statistics Version 23 for Windows.

RESULTS

Surface roughness measurements (Ra):

The mean and standard deviation of the surface roughness values (Ra) were presented in (Table 2). Generally, samples immersed into ethanol showed the statistically significant highest mean surface roughness values for the three tested materials; Filtek™ Z350 XT, Nexco® Paste and Lava™ Ultimate (195.64 μm ± 2.39, 188.99 μm ± 2.15 and 185.42 μm ± 4.08) respectively compared to the other subgroups. On the other hand, the statistically significant lowest mean surface roughness value was recorded for the control group samples of the three tested materials Filtek™ Z350 XT (80.81 μm

± 2.02), Lava™ Ultimate and Nexco® Paste (84.78 μm ± 2.34 and 90.0 μm ± 2.19) respectively.

ESE micrographs for the tested materials Lava™ Ultimate, Nexco® Paste and Filtek™ Z350 XT respectively were signified throughout (figures A1, A2 & A3 - F1, F2 & F3) according to the food simulating fluids, thermocycling and the control (citric acid, control, ethanol, heptane, thermocycling and distilled water) correspondingly.

Weight loss due to wear:

The mean and standard deviation values of the wear resistance test are presented in (table 3). Generally, samples immersed into ethanol showed the highest mean weight loss -due to wear- value was shown for the three tested materials; Filtek™ Z350 XT (1.5371 mg ± 0.044), Lava™ Ultimate (1.5338 mg ± 0.037) and Nexco® Paste (1.5326 mg ± 0.039) with no significant difference between the three mean values. On the other hand, the control samples

TABLE (2): Descriptive statistics and test of significance of the effect of food simulating liquids and thermocycling on surface roughness of the tested materials.

Groups	Restorative Materials					
	Lava™ Ultimate M_1		Nexco®Paste M_2		Filtek™ Z350 XT M_3	
Subgroups	Mean [μm]	SD	Mean [μm]	SD	Mean [μm]	SD
$S_{0(control)}$	84.78 f B	2.34	90.00 f A	2.19	80.81 f C	2.02
$S_{1(water)}$	97.21 e C	2.77	110.80 e A	2.12	100.43 e B	1.56
$S_{2(ethanol)}$	185.42 a B	4.08	188.99 a B	2.15	195.64 a A	2.39
$S_{3(heptane)}$	120.93 d C	2.24	132.46 d A	2.52	128.81 d B	2.27
$S_{4(citric\ acid)}$	139.91 c B	2.66	148.57 c A	3.24	140.63 c B	1.93
$S_{5(thermocycled)}$	157.85 b B	2.07	169.53 b A	3.55	171.44 b A	2.63

Results with the same letter are not significantly different within the same subgroup, 2 ways ANOVA ($p\leq0.05$)

Small Letters indicate the difference within the same column ***Capital letters indicate the difference within the same row***

TABLE (3): Descriptive statistics and test of significance of the effect of food simulating liquids and thermocycling on wear resistance for the tested materials.

Groups	Restorative Materials					
	Lava™ Ultimate M_1		Nexco®Paste M_2		Filtek™ Z350 XT M_3	
Subgroups	Mean [gm]	SD	Mean [gm]	SD	Mean[gm]	SD
$S_{0(control)}$	0.6226 e C	0.032	0.7259 e B	0.02	0.7694 e A	0.033
$S_{1(water)}$	1.0597 d	0.016	1.0639 d	0.012	1.0796 d	0.008
$S_{2(ethanol)}$	1.5338 a	0.037	1.5326 a	0.039	1.5371 a	0.044
$S_{3(heptane)}$	1.1244 c B	0.005	1.1197 c B	0.008	1.1368 c A	0.009
$S_{4(citric\ acid)}$	1.1341 c B	0.011	1.1254 c B	0.006	1.1489 c A	0.005
$S_{5(thermocycled)}$	1.3257 b B	0.021	1.3365 b B	0.042	1.4138 b A	0.025

Results with the same letter are not significantly different within the same subgroup, 2 ways ANOVA ($p\leq0.05$)

Small Letters indicate the difference within the same column *Capital letters indicate the difference within the same row*

Cells with no letter indicate that there is no significant difference between results

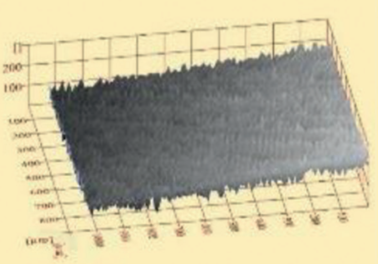

Fig. A1. ESE Micrograph Lava™ Ultimate- citric acid

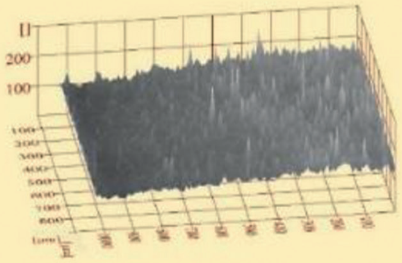

Fig. B1. ESE Micrograph Lava™ Ultimate- control

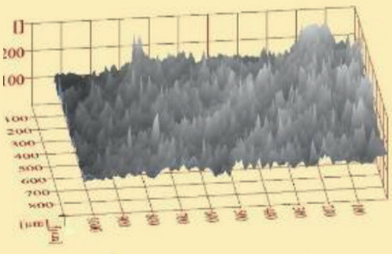

Fig. C1. ESE Micrograph Lava™ Ultimate- ethanol

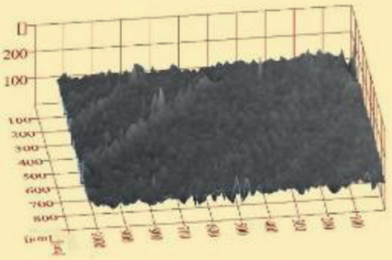

Fig. D1. ESE Micrograph Lava™ Ultimate- heptane

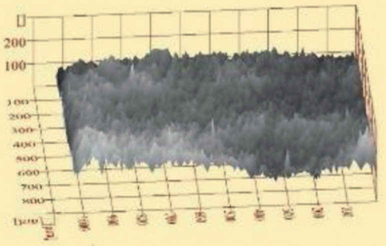

Fig. E1. ESE Micrograph Lava™ Ultimate- thermocycling

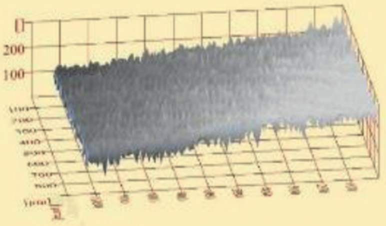

Fig. F1. ESE Micrograph Lava™ Ultimate-distilled water

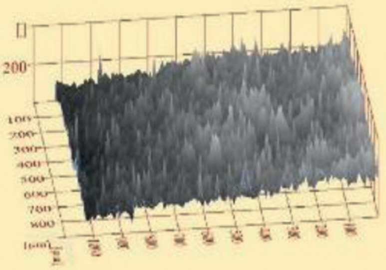

Fig. A2. ESE Micrograph Nexco® Paste- citric acid

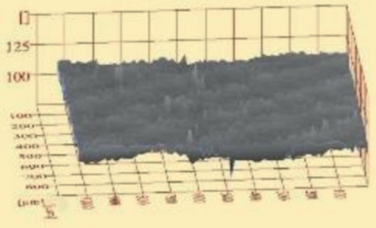

Fig. B2. ESE Micrograph Nexco® Paste- control

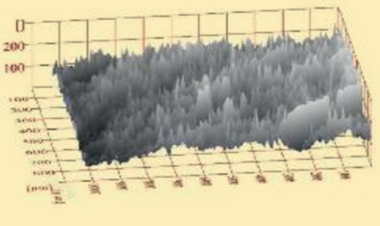

Fig. C2. ESE Micrograph Nexco® Paste- ethanol

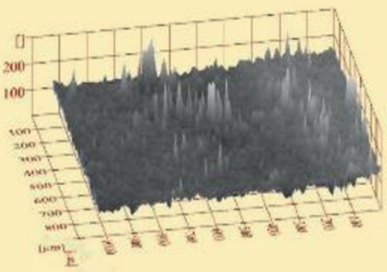

Fig. D2. ESE Micrograph Nexco® Paste -heptane

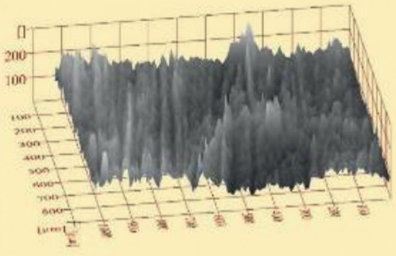

Fig. E2. ESE Micrograph Nexco® Paste thermocycling

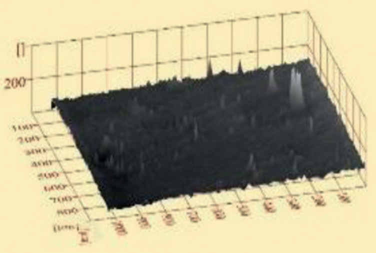

Fig. F2. ESE Micrograph Nexco® Paste- distilled water

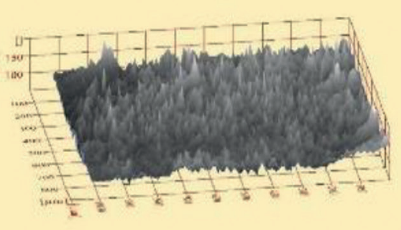

Fig. A3. ESE Micrograph Filtek™ Z350 XT- citric acid

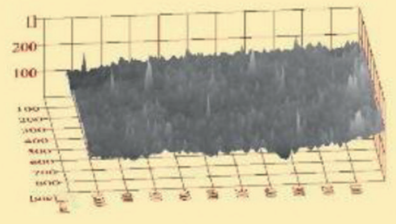

Fig. B3. ESE Micrograph Filtek™ Z350 XT -control

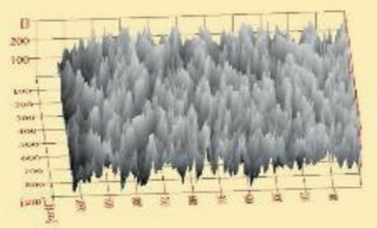

Fig. C3. ESE Micrograph Filtek™ Z350 XT- ethanol

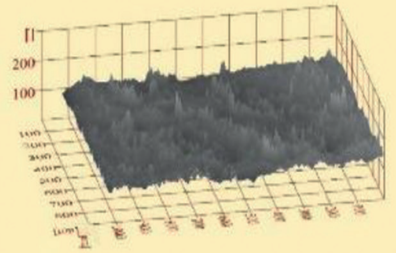

Fig. D3. ESE Micrograph Filtek™ Z350 XT -heptane

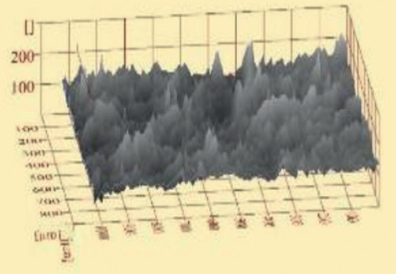

Fig. E3. ESE Micrograph Filtek™ Z350 XT- thermocycling

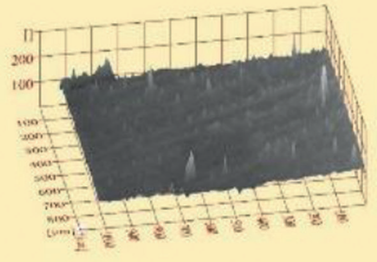

Fig. F3. ESE Micrograph Filtek™ Z350 XT- distilled water

showed the lowest mean weight loss values for the three tested materials Lava™ Ultimate (0.6226 mg ± 0.032), Nexco® Paste (0.7259 mg ±0.02) and t Filtek™ Z350 XT (0.7694 mg ± 0.033), with a significant difference between the three subgroups.

Correlations between different tests

Pearson's correlation showed a strong positive relation between surface roughness and weight loss due to wear (P value ≤0.001 and Person correlation coefficient = 0.934).

DISCUSSION

In modern dentistry, resin composite materials and ceramics are increasingly used due to several desirable qualities, such as esthetic appearance and good physical and mechanical properties (2). Ceramics showed some drawbacks such as wear to opposing enamel (6) and brittleness (7).

The resin composite restorative material is a good choice when seeking highly esthetic restoration with minimal removal of tooth structure. Nowadays, the composite resin restoration showed reliable longevity (8).Due to the sorption phenomenon of the resinous part of the resin composite, the food simulating fluids are recommended by Food and Drug Administration (FDA, 1976, USA) as an accelerating environment. Food simulating fluids are four liquids; distilled water simulates the wet oral environment provided by saliva and water, heptane simulates butter, fatty meats, and vegetable oils, while citric acid and ethanol solution simulate certain beverages including alcohol, vegetables, fruits, candies, and syrups (9).

In the current study, surface roughness and surface loss due to wear were measured as the surface characteristics of esthetic restorations and are one of determining factors of the success of such restorations. Surface smoothness and wear resistance of the dental restorations will reduce the risk of biofilm adhesion, recurrence of caries, staining and gingival irritation (10).

Many tests were suggested to assess the wear of dental restoration in-vitro such as Pin on disc tribometer, toothbrush simulator, jaws simulator, nanoindentation and scratch test (11). In the current study toothbrush wear test was used to simulate the clinical conditions. Toothbrush wearing action is the main cause of material loss of restorations in non-stress areas (12). The abrasion process produced by oral hygiene methods can adversely affect the surface characteristics of restoratives (13).

The results of the current study revealed a destructive effect after immersion into food simulating liquid as a result of the sorption nature of the resin composite material. In sorption process, portion of the liquid locates into the micro-voids, another part located among polymer chains without noticeable volume changes and the last portion of the liquid separates the polymer chains leading to an increase of the volume (swelling) (14). The sorption and diffusion of solvents between the polymeric chains will result in separation of polymeric chains and interruption of their arrangement. Moreover, the leaching out of the unreacted components from the resinous matrix. All that will negatively affect the physical properties of the resin composite material (15).

The results of the current study showed that immersion into ethanol showed the most destructive effect over the three composite resin materials. Such finding was in accordance with other studies (16-19). Organic solvents like ethanol have the potential for damaging the polymeric matrix. Ethanol can penetrate the resin matrix fully and promote the release of unreacted monomers. The partial dissolving of the resin matrix may result in the degradation of the filler-matrix interface (20,21).

Thermocycling showed a deteriorating effect over the three tested materials that may be due to water absorption as the water molecules inside the polymeric structure have a plasticizing effect (22). Furthermore, the change in the temperature would be associated with the reduction in the intermolecular bonds between the matrix chains.

The results of the present study showed that Filtek™ Z350 XT showed the least resistance to food simulating fluids and thermocycling compared to the other two tested materials. This may be explained by the lower degree of conversion of the direct composite resin restorative material; compared to the other two indirect composite resin restorative materials; Lava™ Ultimate and Nexco® Paste (23). The higher the degree of conversion decreased the sorption process made the composite resin more resistant to the effect of thermocycling which may lead to water absorption by a diffusion-controlled process, and it causes leaching of unreacted monomers and swelling of the matrix (24).

Surface roughness

The surface roughness of the composites is influenced by several material factors, such as the type, shape, size and distribution of the inorganic fillers (10).

According to the present study results, the control group of Filtek™ Z350 XT showed a lowest surface roughness values (Ra). This is due to its packing against Mylar strips inside the mold. Many authors reported that the use of Mylar strips showed smoother surface compared with the use of Sof-Lex™ disks to finish and polish nanofilled, nanohybrid, microhybrid, mcirofilled, hybrid and packable resin composites (10,25,26).

The control samples of microhybrid Nexco® Paste showed the highest mean surface roughness

(Ra) value compared to the both nanofilled resin composites Lava™ Ultimate and Filtek™ Z350 XT. One explanation of this result could be referred to the filler size difference between the tested materials. Nexco® Paste contains microhybrid fillers which are larger than the nanofillers of Lava™ Ultimate and Filtek™ Z350 XT, where it was stated that the surface roughness has been reduced by decreasing the filler size and increasing the filler content. Use of a finer filler size results in less inter-particle spacing, more protection of the softer resin matrix and less filler plucking (10).

Wear resistance

After curing of the composite resin, the surface of the composite resin has a resin-rich layer which has a higher wear tendency (10). This fact could explain the lower wear resistance of the Filtek™ Z350 XT samples compared to Lava™ Ultimate and Nexco® Paste samples because these indirect composite resin materials are subjected to additional curing so contain a matrix with more degree of polymerization, that is more wear resistant.

The control samples of Lava™ Ultimate showed more wear resistance than the control samples of Nexco® Paste. This could be explicated by the higher filler content of the former samples. Further investigations should be done to compare the degree of polymerization of the resin matrix of the both materials. Based on the aforementioned findings, this study praises the use of Lava™ Ultimate because of its higher fillers content in addition to its highly cross linked resin matrix. Stating that Lava™ Ultimate is a CAD/CAM material that needs a CEREC® CAD/CAM machine which is costly equipment, in conjunction with the need for well-trained operators. All that may limit its everyday application.

CONCLUSION

1. The food simulating fluids and thermocycling negatively affected the surface properties of the all tested materials.
2. The ethanol showed the most deteriorating effect on the surface properties of all tested materials.
3. Lava™ Ultimate was the most stable material after immersion in food simulating fluids and thermocycling.

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