

The influence of dietary simulating solvents on a recent composite topography (in vitro study)

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Abstract:

Objective: study the influence of dietary simulating liquids on the micro hardness, surface roughness, and color stability of polished and non-polished organically modified ceramic, at different storage time.

Methods: 240 samples were prepared and then stored in a dark phial containing distilled water (D.W) at 37C for 7 days before conditioning then the samples divided into 3 groups, 80 samples in each, and stored in the conditioning liquids at 40 C for 1, 7 and 14 days At the end of each conditioning time the specimens were subjected to color comparison test, surface roughness test (RA) and microhardness test Vickers hardness number (VHN).

Results & conclusion Increase aging time will increase the VHN value and decrease the Ra value of ormocer specially for the control group (aged in D.W). Aging in (food stimulated liquids) FSLs produced an increase in VHN value but to a degree less than that of D.W. which in turn produced significant effect of these liquids on Ormocer hardness with time in comparison to the control group Most effect will be seen with 50% Ethanol VHN and color stability. The effect of FSLs on Ormocer VHN were on long term duration (11 days) while for color change ;(7 days) is the critical time and after the first week no additional color will take place. Surface roughness of polished and non-polished Ormocer was not affected by any FSLs and at any aging time. Polishing of Ormocer will increase the VHN value and Ra value for all FSLs and for all time intervals.

Change in organic base matrix of composite resins is of great value for improving composite properties and durability.

Keywords:

Micro hardness, surface roughness, and color stability of polished and non-polished organically modified ceramic, (D.W), FSLs.

Introduction:

Composite restoratives are not stable after polymerization and constantly interact with their environment.

Since the beginning of the early 1800s intermittent controversy has

surrounded the use of mercury in dental amalgam restoration ⁽¹⁾. More patients today are well informed about dental care and are seeking tooth-colored restorative alternatives. The development of a wear-resistance composite resin for use in posterior teeth has been an elusive goal for the dental

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profession recent advances in resin-based composite systems have led to expect a high degree of success when restoring posterior teeth ⁽²⁾. With packable composite, which have been introduced as an amalgam alternative, the time that posterior composite is a total replacement for amalgam is quickly coming ⁽³⁾.

Ormocer, the newly developed packable organically modified ceramics are interesting and promising materials. And may become an interesting filling material for stress bearing class II restorations.

Composite restoratives are not stable after polymerization and constantly interact with their environment ⁽⁴⁾.

Durable composite must have the ability to resist chemical degradation by oral environment; in addition to mechanical stress. The chemical environment is one aspect of the oral environment that could have an appreciable influence on the *in vivo* degradation of composite resins. Oral solvents change the properties of dental composite, e.g. food-simulation solvents ⁽⁵⁻⁷⁾.

Intra-oral conditions can be expected to be more complex than those achieved by employing 37°C distilled water in laboratory. So the role of dietary factors as aetiological agents have been investigated in this research according to the FDA latest version of Guidance for chemistry recommendations ^(6,7).

Studies investigating the effects of different polishing methods on newer packable composite restorative materials is limited, and the effect of food-simulating liquids on dental composite properties were largely varied with

different results that is may be due to the type of material or testing procedure.

Materials and methods:

Samples Preparation

A piece of dental strip was fixed on a glass slab using adhesive tape, then a Teflon mold which has 4.5 mm diameter & 2 mm height was putting on the strip to be ready for composite insertion ^(8,9).

The ormocer was injected into the mold, by using dental gun supplied from manufacturer; till slightly overfilled. Again a piece of dental strip & a cover micro slide was putting onto the mold, with 2 × 50 gm weight on each side of the slide to provide a standardized pressure & let the excess material extruded outside the mold. When the outline of the mold was clearly seen the light source was applied with the nozzle tip directly contact the glass slide over the mold (distance about 1.3 mm ± 0.1 which is the thickness of the micro slide & strip).

Curing time was 40 seconds as recommended by manufacturer. After the mold has been removed we get a disc of ormocer 4.5 mm in diameter and 2 mm height. Each mold was used for construction of 5 disks of ormocer. Any disk having an air bubble or pitted surface were discarded & changed by another one.

Half of the prepared disks were polished immediately after light polymerization to simulate clinical situation. Using gray Politip- finisher silicon rubber & green Politip-polisher silicon rubber sequentially in a low-speed handpiece mounted on dental surveyor with water coolant. Rotary instrumentation was done for 30 seconds per specimen. The other half of samples

remained as-Set surface & both strips were discarded after each use.

Food simulating liquids

For Ethanol the concentration of pure Ethanol is 100%, it is diluted to the concentration 10% & 50% according to the formula:

$$M_{dil} V_{dil} = M_{conc} V_{conc}$$

Where the M and V terms are the molarity and volume of the *dilute* and *concentrated* solution. Using graduated cylinder and pipette.

10% Ethanol: Simulate aqueous & acidic foods.

50% Ethanol: Simulate low & high alcoholic foods.

Corn oil: Simulate fatty foods.

Highlights of the 1995 recommendations: The agency's commitment to international harmonization under the general agreement on Tariffs & Trade (GATT) has been considered. As a result: 40°C for 10 days is now acceptable to model long-term & storage at ambient temperature.

The 240 disks were divided and stored in 20ml of D.W as a control groups or 20ml of the FSLs as a conditioning groups (Figure 1).

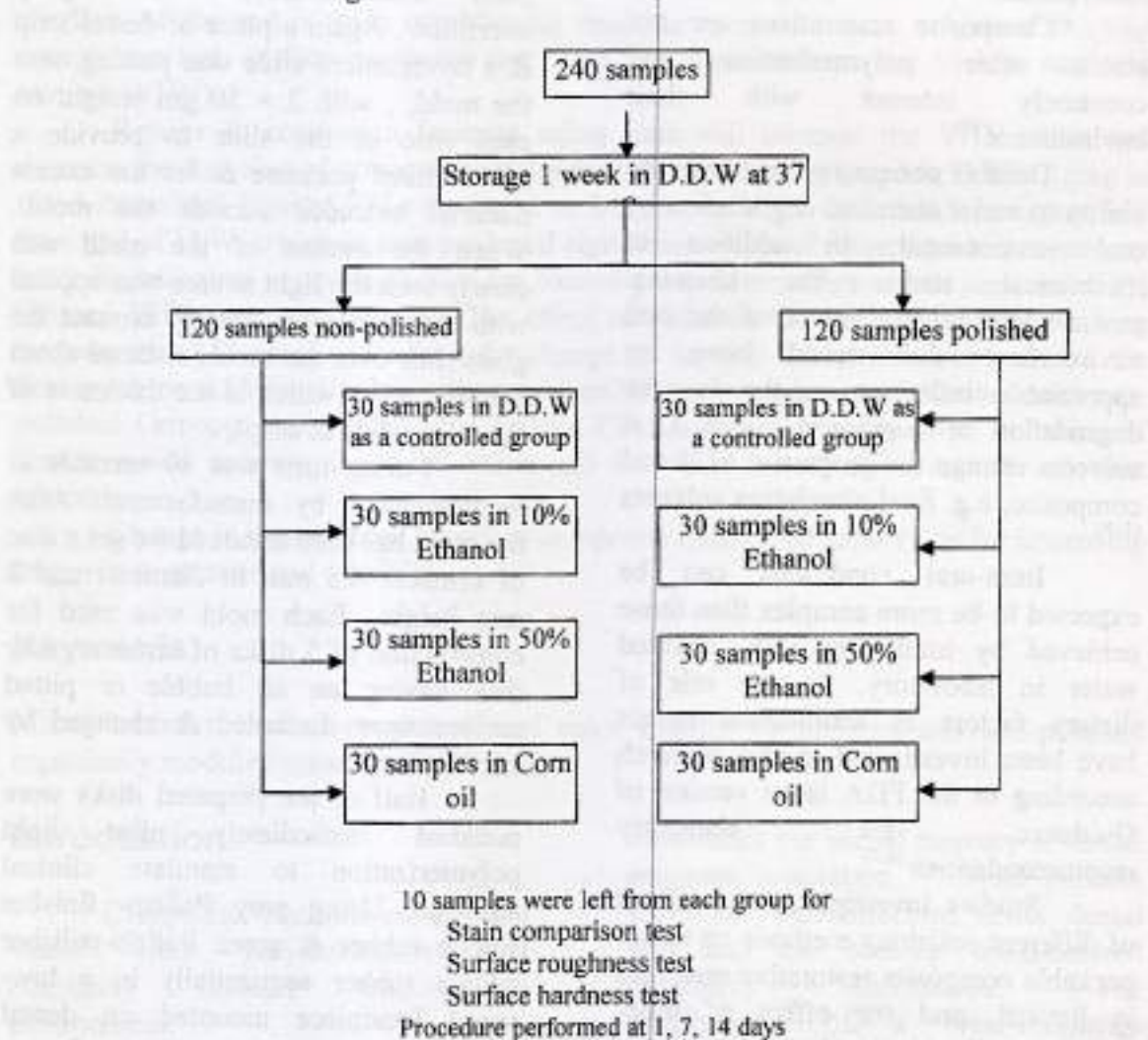


Figure (1): Schematic Diagram for Samples Grouping

At the end of each conditioning period, the samples were lightly rinsed with deionized distilled water D.D.W & gently dabbed dry with filter paper before each test measurement.

Staining comparison test: Evaluation of the stain & color comparison was done according to ISO: 7491 and American Dental Association (ADA) specification number 27 (ADA 1978). The stain comparison was conducted by placing each sample & its control on a white background surrounded by black background, 4 people with normal color vision view the specimens for a period of not longer than 2 seconds under daylight (ISO 7491 permit only 3 observers).

Each sample was ranked from 1 to 4 according to the intensity of its stain.

Score 1 means no change

Score 2 means questionable change

Score 3 means definite change but less than score 4

Score 4 means greater amount of change

Surface Roughness Test: The profilometer (perthen GmbH, Germany) was used for the average surface roughness (Ra) measuring.

Ra : is the arithmetic mean of all values of the roughness profile within the measuring length.

Stylus radius used was 2.5 μm , each sample was measured three times using traveling length of 1.5 mm & Ra value of each sample was the average of these measurements.

Surface Hardness Test: The 136 degree diamond pyramid or Vickers hardness test was used to determine the surface hardness^[12].

The 136 degree diamond pyramid-shaped indenter is forced into the material with 100g load (the load that produces a well-scribed indentation in pilot study). After indentation was made, the surface hardness of composite disks was measured by the average of the two diagonals resulting from the indentation then the VHN were taken from a table with the microhardness machine or it could be obtained from the formula

$$VHN = 1.85 P/d^2$$

P = load in kg, d = average length of the measured diagonals in mm.

Results:

The mean RA value of surface roughness polished and non-polished ORMOCER when stored in different conditioning liquids for different aging periods are reflected.

1. Surface roughness of polished and non-polished ORMOCER was not significantly affected by storage in the various food simulating liquids for different storage time. While there is a very highly significant increase in surface roughness of ORMOCER by polishing for all conditioning ESLs (10% Ethanol, 50% Ethanol, corn-oil), control group and at each time intervals, 1, 7, 14 days. Increased aging periods will decrease the RA value specially for the control group aged in D.W fig (2), table (1).

2- Surface hardness

the mean VHN of the polished and non-polished ORMOCER when stored in different FSLs for different aging periods are reflected in fig.(3).

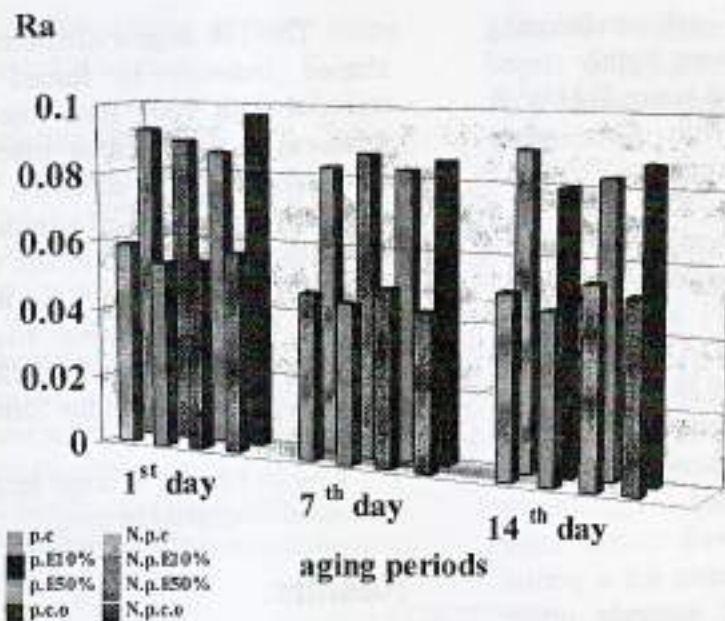


Fig (2): Means of Ra values of polished and non-polished ORMOCER aged in different FSL's at various times

Table (1): Solubility Parameter of food-simulating liquids (FSL)

FSL	Solubility Parameter $\times 10^{-4} \text{ J}^{1/2} \text{ m}^{-1/2}$
Heptane	1.51
Ethanol in water	
100%	2.60
75%	3.15
50%	3.70
25%	4.24
0%	4.79

There was no significant change in surface hardness was noted when aging and non-polished ORMOCER in different FSLs 1 and 7 days.

14 days storage in different FSLs revealed significant decrease in VHN for polished and non-polished ORMOCER in comparison to the control group.

There was significant increase in VHN by polishing ORMOCER for each time of storage (1,7,14 days) and in different FSLs.

Polishing ORMOCER produce significant increase in VHN for all storage time (1,7, 14 days) and in different FSLs.

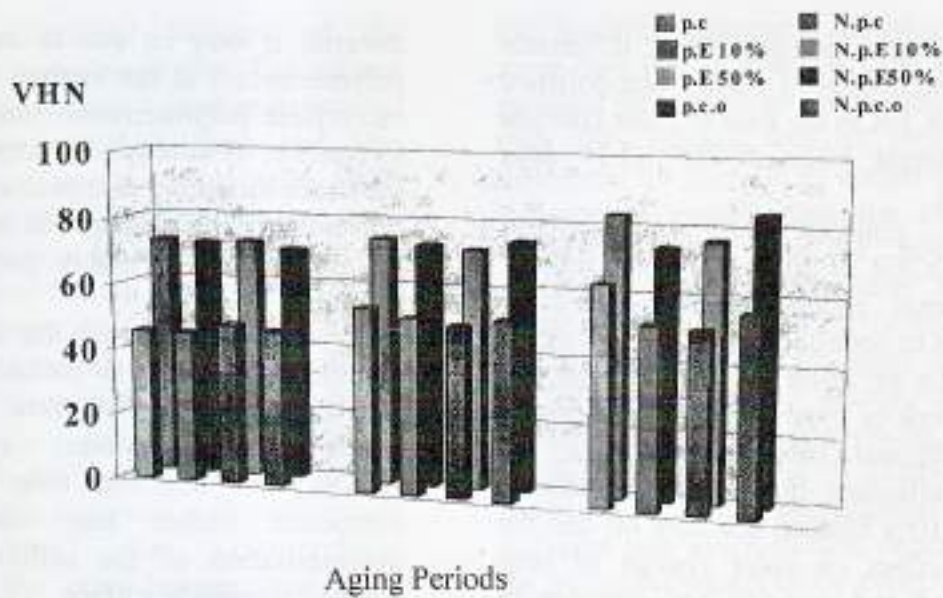


Fig.(3): Means of VHN values of polished and non-polished ORMOCER aged in different FSL's at various times.

3- color

the mean of color changes of the polished and non-polished ORMOCER when stored in different FSLs in

comparison to control group for different aging periods are reflected in fig 4.

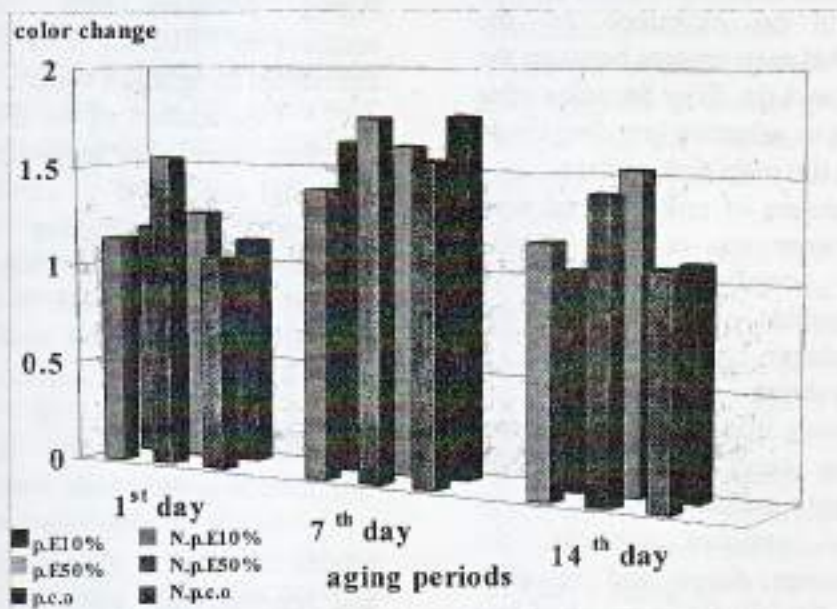


Fig.(4) Means of color values of polished and non-polished ORMOCER aged in different FSL's at various times

There was a non-significant difference between polished and non-polished ORMOCER in the base of color changes in different FSLs & for 1,7,14 days storage.

Aging polished and non-polished ORMOCER in different FSLs produced significant change in color in comparison to control group. The most effect of FSLs on color stability is after the first week of conditioning and after that no additional color will take place. The most affecting liquid is 50% ethanol while 10% Ethanol and core oil had the same effect on color change of both polished and non-polished ormocer in comparison to the control group.

Discussion:

1. surface roughness:

the results from this study revealed that surface roughness of ormocer polymerized against matrix strip was lower than that polished group this finding could be explained as: the irregularity that may present between the resin matrix and the filler particles after polishing due to selective grinding could be hidden by the resin rich surface.

Surface roughness of polished and non-polished ormocer was not significantly affected by conditioning in food-simulating liquids at different storage time (1,7,14 days)

2.surface hardness

A worthy result from this study is that there was a very highly significant increase ease surface hardness by polishing of ormocer surfaces, for different storage times and in each conditioning liquids. So removal of less than 1 mm from the surface of ormocer will exposed harder substrate.

The possible explanation for this softer non-polished surface than the bulk

material is may be due to incomplete polymerization at the surface⁽¹³⁾. This incomplete polymerization may be due to reaction of atmospheric oxygen with the chain-formation free radicals and so preventing chain propagation and cross-linkage to give a highly polymerized structure⁽¹⁴⁾.

Other explanation for the lower microhardness of the celluloid strip-finished composite surface is that restriction in monomer movement between the celluloid strip and the composite surface may hinder the polymerization of the celluloid strip-covered composite surface

Another possible explanation is the percentage of unreacted double bonds is up to twice as high at the interface to the matrix band as compared to the bulk because in bulk of material, a free radical is surrounded 3-dimensionally by possible reaction partners, while reaction partners only on one side.

Aging polished and non-polished ormocer in FSLs for 1 & 7 days was equivalent to aging in water.

Degradation of the filler / matrix interface occur after long-term exposure of dental composite to certain solvents used as food-simulating liquids⁽⁶⁾. Ethanol cause polymer degradation & softening of the resin matrix cause crack formation in the resin and resin/filler interface with the reduction of hardness conditioning results in layers of subsurface damage as opposed to degradation of the whole specimen.

Many studies demonstrate the effects of ethanol on dental composite and reported that aging in Ethanol or food ingredient produced damage in Bis-GMA based composite and shown to be a good solvent for Bis-GMA resins^(5,6,7). Bis-GMA matrix is hydrophilic and

absorbs substantial amounts of the ethanol & water molecules. The solvent penetrates the cross-linked resin matrix, which becomes less hard and less fracture resistant. Composite with lowest cross-linked resin were most easily penetrate by the solvent, and the highly cross-linked matrix was simply more resistant to the effect of solvent even after saturation was attained. So all these factors explained the resistance of ormocer to dissolution by FSL's, because it don't contain Bis-GMA and base on new inorganic - organic matrix (polysiloxane), this incorporation increase the cross-linkage, and as a result, decrease water sorption and solubility.

Long term aging period (14 days) at elevated temperature (40 C) produced significant decrease in VHN for polished and non-polished ormocer in comparison to the control group.

Such a long term aging in a very good solvent liquids that present in FSL's produced swelling of the polymer matrix and considerable surface damage.

This degradation reduced the hardness and enhanced the wear of composite. FSL's have solubility parameter from 1.5 to $4.8 \times 10^{-4} \text{ J}^{1/2} \text{ m}^{-3/2}$.

With all restoratives, the decline in hardness during pre-conditioning maximized at about $\psi = 3 \times 10^{-4}$, which corresponds to a 75% ethanol, see table (1) ^(1,3f).

The effect of aging in FSL's on non-polished ORMOCER is more than on the polished one. The possible explanation is that the soft surface layer, which results from, unpolymerized and poorly polymerized resin, may result in greater water absorption and greater decrease in hardness. Also it is clear that the effect of Alcohol is more than corn oil. These results could be explained as

the solubility parameter of alcohol is more than that of corn oil. And for the polished groups, the effect of 10% is less than 50% Ethanol with no significant difference for corn oil in comparison to the control group. Also the possible explanation is that the solubility parameter differs among these liquids. Other possible explanation to this non significant difference for aging in corn oil is that it may reduce oxygen inhibition during post-curing and eliminates leaching out of fillers. Also the extent of damage may depend on the diffusion rate which in turn, depends on the molecular weight of the penetrant.

The period of pre-conditioning in the FSL's before the test measurements was two weeks without interruption. This length of pre-conditioning time is rather extensive in light of the fact that composite restorations come into contact with foods only briefly in a sporadic way. Accordingly, the test results to be reported may exaggerate the softening effects on the chemical resistance of the dental composites tested ⁽⁷⁾.

There was overall increase in VHN with time specially in control group that aging D.D.W that may be due to incomplete polymerization surface, lead to increase in hardness with time by the continued cross linkage reaction that occur within polymers. As a whole, the storage time from 1 to 14 days cause increase in VHN of polished and non-polished ormocer surface in each FSL's except for polished group aged in 50% Ethanol which produced increase in the mean of VHN but to a non significant difference.

So we can conclude that the effect of FSL's aging on VHN of ORMOCER surface is minimize the increase in surface hardness with time storage in comparison to the control group

(D.D.W), and not a decrease in VHN value .

Possible explanation is that the highly cross-linked matrix (like ormocer) was more resistant to the effect of solvent, even after saturation was attained. But this saturation with a powerful solvent such as 50% Ethanol-water solution may prevent the continued of cross-linkage reaction that occurs within the polymers.

3 Color

As all the observers who viewed the specimens gave scores range from 1 to 3 only. So the score 4 were excluded from the scale and statistical analysis.

This result indicated, without any statistical analysis that polished and non-polished ORMOCER in all FSL's conditioning are in agreement with ISO 4049 and ISO 7491. " When the material is assessed in accordance to ISO: 7.10 none of the three observers shall observe more than a slight change in color ". Increased filler levels of composite results in increased color stability. So the incorporation of the inorganic-organic matrix in ormocer technology may explained its color stability the greatest amount of color change occurred on 7day interval for both polished and non-polished ORMOCER in all FSL's. This may be reflected to the increase of polymerization and cross-linkage reaction, which related to the composition of the material and storage condition, or it's may be due to the amount of water sorption and saturation with the conditioning liquids. Some studies reported that polymerization reached a termination point after one day⁽¹¹⁾, or few days⁽¹⁵⁾, after polymerization. But in this study with ORMOCER the microhardness of the surface were continue to increase as the whole storage time specially for the

control group which may indicate continue of polymerization during this period. And so the color is more stable in the day 14 where the critical time to stain is the first week.

50% Ethanol-water solution had more effect on color changes in comparison to the control (D.W.) group for both polished and non-polished ormocer and along the whole study time, Tables (36) & (40). This may be due to its high solubility parameter, which in turn may influence the surface chemistry of the composite.

10% Ethanol and corn oil had the same effect on color change of both polished and non-polished ORMOCER in 1, 7, 14 days aging. This also may be due to the solubility parameter.

It is worth noticing that the possibility of color change could be occurred for composite stored in water. So whether ORMOCER had a very good color stability or the FSL's produced color change just like the water is not checked in this study and need more research.

Conclusions:

1. hardness and roughness of polished surface of ormocer was so much greater than that of non-polished surface
2. polishing will increase hardness and roughness of ormocer in comparison to non-polished surface but has no effect on its color stability
- 3-ORMOCER aged in distilled water will continue to increase VHN with time.
- 4-Only long-term aging in FSL's (14 days) will decrease the VHN of ORMOCER in comparison to D.W. This effect is actually inhibit the large amount of hardness raise that occur with D.W. and it is not a really decrease in

hardness. The major effect is achieved by 50% Ethanol then 10% Ethanol followed by corn oil.

5-Aging in FSL's will not affect the surface roughness of both polished and non-polished ORMOCER at any testing time.

6-The most critical time from aging ORMOCER in FSL's in the base of color stability are the first 7 days, and on the day 14 there were no more color change took place. The most affecting liquid is 50% Ethanol.

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