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Effect of Different Storage Conditions on the Mechanical Properties of Silorane Based Composite versus Microhybride and Nanohybrid Methacrylate Based Composite

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Abstract

Original Research Article

Purpose of study to investigate mechanical properties at different storage conditions for three different brands of Silorane, Microhybrid and Nanohybrid composite. ***Materials and methods:*** A total of 189 specimens were prepared, equally divided into three main groups of 63 specimens each group. The first group was rectangular bar used to study the flexural strength and modulus of elasticity, second group was Disc Shaped used to study the Vickers micro-hardness test whereas; the third group was cylindrical shaped used to study the creep specimens were stored for periods of 24 hours (saline) or one month (artificial saliva or Ethanol 70%) in an incubator. **RESULTS** the highest flexure strength mean value saline (137.4 ± 12.68 MPa), Saliva (109.3 ± 4.262 MPa) Ethanol lowest (72.73 ± 6.725 MPa). The difference was significant ($P<0.05$). The highest Young's Modulus value saline (29.57 ± 4.2 GPa) followed by Saliva (29.13 ± 0.39 GPa) Ethanol lowest (19.64 ± 2.26 GPa). The difference was significant ($P<0.05$). No significant difference between saline and saliva aging ($P<0.05$). The highest micro-hardness mean value saline (98.74 ± 1.387 HV) followed by Saliva aging group (92.17 ± 2.109 HV) while Ethanol aging group recorded the lowest mean value (83.19 ± 1.053 HV). The difference was significant ($P<0.05$).Saline (24hr) aging group recorded the highest creep mean value saline (1.281 ± 0.021 %) followed by Saliva aging group (1.266 ± 0.198 %) while Ethanol lowest value (1.123 ± 0.051 %). The difference was statistically non-significant (0.05). ***Conclusion:*** The flexural strength and hardness of the silorane and methacrylate composite were significantly influenced by saliva or ethanol. The Young's Modulus of the silorane and methacrylate composite were not significantly influenced by saliva and significantly influenced by ethanol. The creep of silorane and methacrylate composite were not significantly influenced by saliva or ethanol.

Keywords: mechanical properties Silorane, Microhybrid, Nanohybrid, composite.

INTRODUCTION

Since their introduction into the dental market 40 years ago, dental resin composites have proven to be successful [1].Tooth colored composite materials gained a wide popularity during the last decades. Apart from the development of a minimal invasive preparation technique as well as improved adhesion to tooth structures these materials exhibit predictable long-term stability [2, 3].

Composite restorative materials represent one of the many successes of modern biomaterials research, since they replace biological tissue in both appearance and function. At least half of posterior direct restoration placements now rely on composite materials [4].Unfortunately, technique demands on these restorations with regard to mechanical properties, placement, and need for in situ curing leave significant room for advancements, particularly with respect to their polymerization shrinkage and polymerization-induced stress, coefficient of thermal expansion, fracture, abrasion and wear resistance, marginal leakage, and toxicity [5].

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The aesthetic success of tooth colored restorative dental materials such as resin-based composite (RBC) is influenced by several factors including translucency and opacity which are viewed as being vital components indicating the quality and quantity of light reflection on curing [6].

Properties of the dental composites are greatly influenced not only by the properties of their fillers but also by the chemical structure of the monomers used in the matrix phase [7, 8].

RBCs are three-dimensional combinations of at least two chemically different materials with a distinct interface, The majority of methacrylate-based restorative systems consist of a blend of micro- or nano-sized fillers with larger silicate- or zirconia-filler particles that are classified as a macro-hybrid, micro-hybrid, or nano-hybrid composite resin [9-12].

In spite of the undeniable technological advances introduced during the last four decades, the volumetric shrinkage that accompanies the chain growth polymerization of dimethacrylate monomers remains a major concern for the clinical performance of composite restorations [13]. To overcome this situation silorane-based composite resins, such as the resin Filtek Silorane were introduced in the market. The silorane matrix is formed by the cationic ring-opening polymerization of the silorane monomers [14]. The “silorane” molecule represents a hybrid that is made of both siloxane and oxirane structural moieties. The novel resin is considered to have combined the two key advantages of the individual components: low polymerization shrinkage due to the ring-opening oxirane monomer and increased hydrophobicity due to the presence of the siloxane species. The mechanism of compensating stress in this new system is achieved by the opening of the oxirane ring during polymerization [15].

The matrixes of RBCs are susceptible to softening by organic acids and various food and liquid constituents [16]. Under oral conditions, RBCs may be exposed either intermittently or continuously to chemical agents found in saliva, food, and beverages [17]. Consequently, the leaching of compositefillers and the disintegration of filler-resin interface (silane coupling agent) can also occur under oral conditions Therefore, in the case of RBCs, degradation typically occurs because of these two reasons: (1) hydrolytic breakdown of the bond between the silane and filler particles and the fillerresin matrix, resulting in debonding ultimately; and (2) the softening of dental resins through the plasticizing action of water9). As for the effects of solvents on dental composites, many factors come into play — such as the hydrophilicity of polymers and the crosslinking density of the network [18].

Synthesis of a new monomer system named “silorane”, which is obtained from the reaction of oxirane and siloxane molecules. The silorane-based composite (SBC) exhibited low polymerization shrinkage due to the ring-opening oxirane monomer and increased hydrophobicity due to the presence of the siloxane species [15]. It was also claimed that SBC was stable and insoluble in biological fluids simulated using aqueous solutions containing epoxide hydrolase, porcine liver esterase, or diluted HCl [19]. In light of these favorable properties, this new monomer system may be a promising solution to overcoming the negative effects of oral fluids on the mechanical properties of RBCs.

So the main target in the development of the silorane-based composite (P90) is to decrease shrinkage stress, however this should not be on the expanse of the mechanical properties [20].

Since low shrinkage and high mechanical properties are generally opposite properties, our study aimed to analyze the the effects of different storage conditions on mechanical behavior at micro- and macro-scale of a silorane-based composite in comparison with tow methacrylate-based micro-hybrid and nano-hybrid materials.

LITERATURE REVIEW

I. Historical Perspectives

J. Redtenbacher (a German chemist) in 1843 sparked the ongoing studies of current resin monomers when he discovered a new acid, to which he gave the name of acrylic acid [21].

By 1900, methacrylic acid, as well as several of its esters including methyl methacrylate, had been synthesized and polymerized Methyl methacrylate polymerizes by an addition mechanism through the carbon-carbon double bonds to form poly (methyl methacrylate) [22].

In the late 1930s, poly (methyl methacrylate) was introduced for indirect filling resin. The discovery of the benzoyl peroxide-tertiaryamine redox initiator-accelerator system, allowing methyl methacrylate to polymerize at ambient temperature, laid the basis for direct filling resins, which were developed in Germany during World War II [23].

In the 1940s the acrylic resins that replaced silicate cements were the only aesthetic materials available in the field of conservative dentistry, but they have many drawbacks [24].

In 1951, Epoxy resins appeared to be worth investigation, as they harden at room temperature with little shrinkage to produce an insoluble polymer with adhesiveness to most solid surfaces. In the epoxide group, the ring is in a somewhat unstable condition, epoxy resins were given up due to slow hardening, preventing their use as a direct filling material [25].

In 1955, Buonocore used orthophosphoric acid to improve the adhesion of acrylic resins to the surface of the enamel [24].

Bowen (1956) [26], synthesized a new monomer, initiating the era of dental resin composites. The monomer 2, 2-bis [4-(2-hydroxy-3-methacrylyloxypropoxy) phenyl] propane, resembles an epoxy resin, except that the epoxy groups are replaced by methacrylate groups. Polymerization of the monomer, which was given the acronym Bis GMA, occurs through the carbon-carbon double bonds of the two methacrylate groups [27].

Bowen (1970) [28] devised a unique monomer system based on the premise that certain isomeric crystalline dimethacrylates are capable of forming a eutectic that is liquid at room temperature ,this was all in an effort to solve the problems associated with the use of BisGMA. Three aromatic diesters, were used along with alternative accelerators, e.g. ascorbyl palmitate, and so the problem of color instability was avoided allowing the formulation of excellent resin materials [29].

Baily *et al.,* (1975) [30], reported that a variety of bicyclic monomers, including spiro orthoesters, spiro orthocarbonates, bicyclo ketal lactones, trioxabicyclooctanes, and unsaturated diketals of benzoquinone, would undergo double ring opening with either no change in volume or an actual expansion [31]. The use of spiro orthocarbonates (SOC'S) as a component in dental resin composites resulted in a nearly volume neutral polymerization and in a doubling of the adhesive strength of the resin to etched enamel [32].

In 1983 the most significant changes in commercial composites have been made through altering the filler component. These changes have prompted the periodic development of classification systems for dental composites based upon filler size and volume fraction. By 1983 Lutz and Phillips published a system for characterizing dental composites based on particle size. The microfills were divided into subclasses which included a characterization of the type of pre-polymerized resin fillers incorporated, i.e., splintered, agglomerated, or spherical [33].

Mathias *et al.,* (1987) [34], Synthesized a class of difunctional monomers, termed oxybismethacrylates, this product exhibits cyclopolymerization, this type of reaction generally involves 1,6-dienes which cyclize to introduce 5-and/or 6-membered rings into the polymer back-bone.

Munksgaard *et al.,* (1987) [35], prepared various amounts of microfiller, condensed microfiller, prepolymer plus microfiller and macrofiller then added each one to an unfilled light-curable resin, they measured the wall to wall polymerization contraction of each of them in dentine cavities. They concluded that increasing amounts of microfiller did not affect the wall to wall contraction but mixtures made by the three other fillers showed a decreasing in wall to wall contraction with increasing filler concentration.

Ferracane (1989) [36], reported many studies that have a correlation between mechanical properties and filler volume.

Phillips (1991) [37], initially fused a crystalline quartz and various borosilicate or lithium alumino silicate glasses were used as fillers for dental composites to make a paste that could be hardened into a dental restorative material with strength and stiffness far surpassing those of the unfilled polymer itself. However, quartz has drawbacks in that it is not radiopaque and can be very abrasive to enamel. Another drawback to the original quartz and glass fillers were that the particles were large and very hard in relation to the surrounding polymer matrix.

Khan *et al.,* (1992) [38], introduced current composites filled with radiopaque silicate particles based on oxides of barium, strontium, zinc, aluminum, or zirconium.

Willems *et al.,* (1992) [39], published a similar classification system of Lutz and Phillips. The basic difference between the two reflects the fact that the most popular non-microfill composites have smaller mean particle sizes and fewer large particles than composites of a decade ago.

Li T *et al.,* (1996) [40], Synthesized a UDMA analogues containing either a phenoxymethyl group on the periphery or a bulky aliphatic group to replace the core segment of the UDMA. The objective is to reduce the possibility for water to attack the urethane linkages. These resins showed 10 %to 30 % reduction in water sorption compared to conventional UDMA, but also had lower flexure strength because of their low viscosity.

Peutzfeldt A (1997) [41], proposed the first type of urethane dimethacrylate which was synthesized from hydroxyalkyl methacrylates and diisocyanates, these monomers have molecular weights nearly equal to that of BisGMA, but are less viscous. The advantages of UEDMA have been reported to be the lower viscosity and a greater flexibihty of the urethane linkage, which may improve toughness.

Stansbury and Antonucci (1999) [42], Synthesized Fluorinated based on bis-GMA and UDMA with lower water sorption than bis-GMA resins.

Weinmann *et al.*, (2005) [43], described the synthesis of a new monomer system named silorane obtained from the reaction of oxirane and siloxane molecules. The novel resin claimed to have combined the two key advantages of the individual components: low polymerization shrinkage due to the ring-opening oxirane monomer and increased hydrophobicity due to the presence of the siloxane species.

Chen *et al.*, (2006) [44], developed a nano-composite resin by using an epoxy resin matrix with nano-silica fillers through ring-opening polymerization. The results showed that the nano-composite resin was exhibited low polymerization shrinkage strain which is only a quarter of the currently used methacrylate-based composite resins.

Lohbauer *et al.*, (2006) [45], believed that currently methacrylate resin formulations dominate both the commercial market and research evaluation. The resin phase is composed primarily of dimethacrylate monomers typically selected from BisGMA, BisEMA, and/or UDMA. These base monomers result in restorative materials with excellent mechanical properties, rapid polymerization, and low shrinkage.

Pereira *et al.*, (2007) [46], Incorporated methylated and fluorinated derivatives of BisGMA into BisGMA/TEGDMA resins, but it resulted in greater hydrophobicity and reduced water sorption with no significant improvements in mechanical properties.

Chen *et al.*, (2008) [47], modified an oligomeric bisphenol-A monomers with pendant urethane acrylates and exhibited reduced volume shrinkage and improved mechanical properties.

Sahin *et al.*, (2009) [48], produced Bisphenol-A derivatives both with carboxylic acid and phosphonic acid functional groups without degradable esters and with carboxylic, amide, and hydroxyl functional groups to improve adhesion. Acidic monomers have been synthesized from o-hydroxyaryl phosphonates that exhibited rapid polymerization [49].

Jingwei H *et al.*, (2011) [50], proposed a series of polymeric iodine quaternary ammoniums salts with different alkyl chain lengths synthesized by the reaction of dimethyl amino ethyl methacrylate (DMAEMA) with different kinds of alkyl iodides, and their structures were characterized by FTIR, H-NMR, and C-NMR analysis. In minimum inhibitory concentration determination, the antibacterial activity increased with increasing alkyl side chain length of these monomers (5 to 16), then decreased when the alkyl chain length increased to 18.

II. The Effect of Storage Conditions

De Moraes *et al.*, (2008) [51], assessed the effect of 6 months of ageing in water on surface/subsurface hardness of two micro hybrid resin composites (Filtek Z250 and Charisma) .indentations were made on surface and subsurface layers, at 24 hours and repeated after 6 months of storage. The results showed, the Filtek Z250 composites presented significantly harder surface and subsurface layers in comparison with Charisma at 24 hours and after 6 months of storage.

Furuse *et al.*, (2008) [52], showed that Filtek Silorane has good polishing characteristics. The material showed little colour change after artificial ageing and the surface gloss was retained.

Ilie & Hickel (2009) [53], Siloranes have been shown to have good storage stability in various media (alcohol, water or saliva) and compared to conventional composites they are less susceptible to changes if stored in ethanol.

Yesilyurt *et al.*, (2009) [54], investigated the effects of food-simulating liquids (FSL) on the hardness of a silorane-based composite (Filtek Silorane) and to compared it with three methacrylate-based composites (MBCs), one of them was FiltekZ250. The results revealed that compared to control group the hardness of Filtek Silorane were not significantly affected by food stimulating liquid. Conversely, the hardness of MBCs significantly decreased after conditioning in water and ethanol.

Ilday *et al.*, (2010) [55], compared the effects of different beverages upon the surface hardness of microhybrid, nanohybrid and Filtek Silorane composite. After 7-day storage, samples were taken from the beverages. Specimens were subjected to post-immersion microhardness testing. The results of this study showed that acidic drinks did not reduce the surface hardness of composite restoratives.

Hahnel, S *et al.*, (2010) [56], investigated the aging behavior of dental composites. Five composites were subjected to various artificial aging protocols (storage in distilled water/ethanol/artificial saliva /thermal cycling). The results showed generally, artificial aging leads to a significant decrease in mechanical properties, which underlines the relevance of continuous improvement of dental composites.

Pieniak, D *et al.*, (2012) [57], evaluate a long-term impact of the humid environment and cyclic thermal loads on the microhardness of new silorane-based composites and two methacrylate-based composites. The studies of hardness were carried out after each month of the exposure time. No long-term impact of normal saline environment with constant temperature on the microhardness of the studied materials has been noticed.

III. Hardness

Hardness is a surface property defined as the resistance of a material to permanent indentation or penetration.

Studies have shown that the harder a material is, the greater its wear resistance [58]. Microhardness depends largely on the filler particles (size, volume percent and weight percent) and the chemical composition [59].

Erosy *et al.,* (2004) [60], determined the micro hardness of two packable composite and one ion-releasing composite and two hybrid composite one of them was Filtek Z250. The results showed that the hybrid composite Filtek Z250 had statistically highest micro hardness than other tested composite.

Ilie and Hickel (2006) [61] examined the hardness of silorane based composite material, and thereby compared the examined characteristics against those of well-known methacrylate-based composites. The results showed that no statistically significant differences between silorane-based material and methacrylate composite in terms of hardness.

Ceballos *et al.,* (2009) [62], compared the micro hardness of two resin composites Filtek Z250 and spectrum TPH. The results showed Flitek Z250 composites exhibited higher Vickers micro hardness values than Spectrum TPH under each experimental condition evaluated.

Ilie and Hickel (2009) [63] analyzed the hardness of the silorane-based composite in comparison to six methacrylate-based composites after storing the materials for 24h in distilled water as well as after ageing (thermo cycling and storing for 4 weeks in water, saliva or alcohol). Their results showed compared to 24 hours storage in distilled water except for the silorane-based composites, all methacrylate based materials showed a statistical significant decrease in hardness by storing in alcohol solution.

Poskus *et al.,* (2009) [64], evaluated the Vickers hardness number (VHN) of inlay restoration of three hybrid composites resins (Filtek Z250, Opallis and Esthet-X) subjected to two post-cure treatments. The results showed Filtek Z250 composite had higher hardness value than Opallis and Esthet-X for control and two test group.

Lien and Vandewalle (2009) [65], determined the hardness of a new silorane-based restorative material (Filtek-LS) in comparison to six restorative materials one of them was Filtek-Z250. The results showed that compared to the methacrylate-based composite resins, the new silorane –based restorative material had relatively lower Knoop hardness.

Marghalani (2009) [66], evaluated the effect of post-irradiation dry ageing at different periods of time on Vickers micro hardness of some dental composites based on various resin matrices. The results showed dental composites based on silorane resin matrices (Filtek P90) achieved higher Vickers micro hardness than those based on dimethacrylates resin.

Lien *et al.,* (2010) [67], distinguished the hardness of a new silorane-based restorative material (Filtek LS) in comparison to five methacrylate-based restorative materials one of them was Filtek Z250. The results showed that compared to methacrylate-based restorative materials the new, silorane-based material had relatively lower micro hardness in comparison to methacrylate based composite.

Waikasetkorn *et al.,* (2010) [68], evaluated and compared surface hardness of silorane composite with three conventional methacrylate composites. The results showed the silorane-based composite possessed lower surface micro-hardness compared to methacrylate composites.

Lien and Vandewalle (2010) [69], compared the hardness of silorane based composite (Filtek LS), to a new composite resin restorative material (GC Kalore) and five methacrylate composite restorative materials, one of them was Filtek-Z250. The results showed the methacrylate based composite (Filtek Z250) had a significantly higher hardness than Silorane based composite.

D'alpino *et al.,* (2010) [70], evaluated the influence of different polymerization techniques on hardness of silorane and methacrylate –based composite in Class II restorations. The results showed silorane-based composite presented significantly lower hardness values than methacrylate-based composite ($p<0.05$), regardless of the depth. When depth was considered, for silorane-based composite, hardness values were similar; while, for methacrylate-based composite, significantly higher hardness values was observed at 1 and 3mm, when compared to 2 and 4mm.

Yilmaz and Topbasi (2010) [71], evaluated the effects of 4 different bleaching agents with 2 different bleaching methods on the micro hardness of 4 different resin composites (Aelite-hybrid, Grandio nanohybrid, Clearfil Majesty-nano superfilled, Silorane-silorane based). The results showed that Silorane composite showed lowest surface hardness at base line and after exposure to the bleaching agents.

Alsati *et al.,* (2010) [72], determined micro hardness of silorane-based composite and methacrylate based composite Z-100 restorative polymerized with 2

LED-lights and 2 polymerization cycles. Micro hardness measurements were then made on upper and lower surfaces of each specimen using a hardness tester with Knoop indenter 50g load. The results showed the methacrylate based composite Z-100 had highest micro hardness ratios at all tested conditions.

Borges *et al.*, *(*2011) [73] evaluated the hardness of two dental composite resins: micro hybrid (Filtek-Z250) and nanofilled (Filtek-Supreme). Values were measured for bottom/top surfaces using a digital micro hardness tester. Their results showed Filtek-Z250 composite showed higher hardness value than those observed for the nanofilled (Filtek-Supreme) for all curing units except Blue phase, which revealed no significant difference in terms of hardness between the tested composite resins.

Hanhel *et al.*, (2011) [74], determined Vickers hardness of silorane based composite and 13 dental restorative materials (nano-,micro-,hybrid-, macro filled composites, compomer, ormocer) one of them was Filtek Z250, a veneering composite (Sinfony) and enamel were used for reference. The results showed the Filtek Z250 had higher Vickers hardness value than the Filtek P90.

Kusgoz A *et al.*, (2011) [75], determined the hardness of silorane-based composite (Filtek Silorane: FS) in comparison to two methacrylate-based composites. The hardness of every material was evaluated after 1, 7, and 30 days and the results revealed that Filtek Silorane exhibited lower hardness than methacrylate-based composites after 1 day of storage. But the hardness of Filtek Silorane remained unchanged during the storage period and revealed stable hardness in water that was comparable to methcrylate-based composites.

Tchorz *et al.*, (2011) [76], compared the micro hardness of three composite resins with different organic matrices in deep class II cavities. The cavities were restored using Filtek Supreme (FS), Quixfil (QF), and Filtek Silorane (SI). The results showed Quixfil(QF) yielded the highest micro hardness value whereas the Filtek silorance SI yielded the lowest one.

Borges *et al.*, (2011) [77], determined Vickers hardness of nanofilled composite Filtek Z350 and the new silorane-based composite Filtek P90 photo-activated with different LED lights. The results showed the composite Filtek Z350 showed statistically higher Vickers hardness values than Filtek P90 for both top and bottom surfaces.

D'Alpino *et al.*, (2011) [78], investigated the influence of the energy dose on the hardness, of silorane and methacrylate-based posterior composites in Class II restorations with different bonding approaches. Hardness was evaluated along the transversal section of the fillings (1-4mm below the restoration surface). The results of both resin composites were not affected by the depth of evaluation, but the influence of the material was significant.

Ilie and Hickel (2011) [18], evaluated the Vickers hardness of experimental flowable resin-based composite in comparison to four regular methacrylate-based as well as a low shrinkage silorane-based micro-hybrid composite (Filtek Silorane). The results showed for all analysed materials, there was no significant difference in the micro-mechanical properties between top and bottom.

Pieniak *et al.*, (2012) [79], evaluated a long-term impact of the humid environment and cyclic thermal loads on the micro hardness of new silorane-based composite and two methacrylate-based composites. Micro hardness was measured with Vicker's method before and after a series of 4000 thermal cycles. The results showed that micro hardness of silorane-based composites was not decreased under the influence of cyclic thermal loads compared to methacrylate-based composites.

IV. Flexural Strength

Flexural strength has been define as the maximum stress that a material can resist before failure when it is subjected to bending load and it is regarded as the most significant measure of strength of dental materials, as considerable flexural stresses may occur during the complex mastication process [56].

Higher flexural strength materials are less prone to bulk fracture of the filling as well as fracture of margins. High flexural strength values reflect a limited tendency to crazing and high resistance to surface defects and erosion. The in vitro three-point bending flexural test is recommended by ISO 4049/2008 specification for polymer-based materials and is widely used for comparative purposes [56].

Attar *et al.*, (2003) [80], determined the Flexural strength of two conventional methacrylate based composite (Filtek Z250 and Dyract AP) and nine flowable composite materials. The results showed the control composite Filtek Z250 had the highest flexraural strength value at 24 hours (117.4 MPa) and after 1 month (95.6 MPa).

Ersoy *et al.*, (2004) [60], determined the flexural strength of two packable composite and on ion-releasing composite and two hybrid composite one of them was Filtek Z250. The results showed the Filtek Z250 composite had statistically highest flexural strength than other tested composite.

Weinmann *et al.*, (2005) [15], compared the flexural strength of silorane based composite and four methacrylate based restorative materials one of them was Filtek Z250. The flexural strength was determined according to ISO 4049 and the results showed the flexural strength of silorane based composite was significantly lower than methacrylate based composite Filtek Z250.

Filho *et al.,* (2006) [81], evaluated the flexural strength of three composite resins recommended for direct esthetic restoration: a polyacid modified composite (Dyract AP), a unimodel composite resin (Filtek Z250) and a hybrid composite resin (Point 4). The results showed the Filtek Z250 presented the highest flexural strength (166.74 MPa) and Dyract AP presented the lowest one (129.76 MPa).

Rodrigues *et al.,* (2007) [82], determined the flexural strength of five commercial available composites – Supreme (3M/ESPE), a universal nanofilled composite; Esthet-X (Dentsply), Z-250 (3M/ESPE), Charisma (Heraeus Kulzer), universal hybrid composites; and Helio Fill (Vigodent), a microfine composite. The results showed the Filtek Z250 had statistically highest flexural strength than other tested composites.

Ilie and Hickel (2007) [83], studied the flexural strength of new and classical composite formulations with different matrix structures, after thermo-cycling and ageing. Six composite materils were considered: one silorane based (Hermes/3M-ESPE, 4 conventional dimethacrylate composite containing Bis-GMA, TEGDMA, UDMA (Tetric, TetricCeram, TetricCeramHB, TetricEvoCeram) and reapectively one Bis-GMA,Bis-EMA and TEGDMA based composite (EsthetX). The results showed the new-matrix composite silorane and the Bis-GMA, Bis-EMA and TEGDMA based composite revealed a stable strength in the tested solvents.

Bona *et al.,* (2008) [84], assessed the flexural strength of four methacrylate composite resin (Filtek Z250-3M-ESPE; Amelogen, Ultradent; Vit-1-escence, Ultradent; Esthet-X, Dentsply). The flexural strength was measured using the three point bending test, in which bar shaped specimen were loaded at a cross hard spead of 0.5mm/min, in universal testing machine (EMIC 2000). The results showed the Esthet –X and Filtek Z250 had higher flexural strength than the other materials evaluated.

Melo Monteiro *et al.,* (2009) [85], determined the flexural strength of seven dental composite (Filtek Z350, Filtek Z250/3M ESPE; Grandio, Polofil Supra/VOCO; TPH Spectrum, TPH3, Esthet-X/Dentsply). The results showed Grandio composite had higher FS (141.07 MPa) which is not significantly different from Filtek Z250 (129.8MPa).

Ilie and Hickel (2009) [53] analyxed the flexural strength of silorane based composite and compared it against clinically used methacrylate-based composites after storing the materials for 24h in distilled water as well as after ageing (thermo cycling and storing for 4 weeks in water, saliva or alcohol). The results showed for the testing materials the flexural strength significantly reduced by storing in alcohol, where as 4 weeks storage in water or saliva did not significantly reduce the strength. Exceptions from this behavior were measured for Filtek Silorane which showing a constant strength over all storage conditions.

Lien and Vandewalle (2009) [65], determined the flexural strength of a new silorane-based restorative material (Filtek-LS) in comparison to six restorative materials one of them was Filtek-Z250. The reults showed there was no significant diference in terms of flexural strength between silorane-based composite (Filtek-LS) and methacrylate based composite (Filtek Z250).

Yesilyurt *et al.,* (2009) [54], investigated the effects of food simulating liquids (FSL) on the flexural strength (FS) of a new silorane based composite (Filtek Silorane) and compared it with three methacrylate-based composites (MBCs) one of them was Filtek Z250. The results showed that the FS of Filtek Silorane were not significantly affected by FSL. Conversely; the FS values of MBCs were significantly affected after conditioning in ethanol.

Hahnel *et al.,* (2010) [56], compared the ageing behavior of different dental composite materials with regard to flexural strength (FS), Rectangular specimens were prepared from five composites (Filtek Supreme XT, Filtek Silorane, CeramX, Quixfil, experimental ormocer) and subjected to various artificial ageing protocols (storage in distilled water/ethanol/artificial saliva for 7, 90 and 365 days; thermal cycling, 2 x 3000 cycles 5/55°C). FS were determined at baseline and after each ageing treatment. The results showed that, the significantly highest values were found for Filtek Silorane, and the lowest values were found for the experimental ormocer.

Lien *et al.,* (2010) [67], distinguished the flexural strength of a new silorane-based restorative material in comparison to five methacrylate-based restorative materials one of them was Filtek Z250. The results showed that the methacrylate-based restorative materials (Filtek Z250) had the highest flexural strength but was not significantly different from the new silorane –based material.

Waikasetkorn *et al.,* (2010) [68], evaluated and compared the flexural strength of silorane based composite (Filtek LS.3M/ESPE) with three conventional methacrylate composite. The results showed the conventional methacrylate composites (Filtek Supreme plus) had significantly higher flexural strength than the silorane-based composite.

Lien and Vandewalle (2010) [69], compared the flexural strength of a silorane based composite (Filtek LS) to a new composite resin restorative materials (Kalore) and five restorative materials one of them was Filtek-Z250. The results showed there was no significant differences between silorane based composite and conventional methacrylate composite Filtek Z250 in terms of flexural strength.

Leprince J *et al.,* (2010) [86], compared the flexural strength of nine different composite materials, two Ormocers (Admira and an experimental Ormocer V35694), one ring –opening composite (Filtek Silorane) and five methacrylate-based composites and an experimental material, (V34930). The flexural strength was determined by a three-point mending method. The results showed there was no significant difference in flexural strength between material types.

Soler *et al.,* (2010) [87], investigated the time dependent changes of the flexural strength of different composites. Eight materials were used: Heliomolar, Gradia Direct Post, Grandio Flow X-Flow, Grandio, Filtek 250, Filtek Silorane and GC Kalore. The samples were prepared according to ISO standard 4049 and stored in artificial saliva. The flexural strength of each material was measured after 24 hours and after fifteen days of storage with (Shimadzu AGS-J, 500N). The results suggested that storage in artificial saliva for fifteen days causes significant increase in flexural strength for Filtek Silorane.

Lohbauer (2010) [88], assessed the in vitro cyclic fatigue resistance of modern universal resin composites for posterior indications one silorane based composite (Filtek Silorane, 3M ESPE, (SI) and eight commercial composite resins one of them was (Filtek Z250,3M ESPE, (FZ). Flexural strength was measured after 14 days water storage at 37°C. The results showed the initial strength level was 112.2 for the Filtek silorane and 131.5 for Filtek Z250 composite. He concluded that cyclic flexural loading significantly reduces strength of composite resins, for the Filtek silorane the strength was 62.6 and for Filtek Z250 was 55.2.

Wilner *et al.,* (2010) [89], compared the flexural strength of ten different filling composites. Filtek Supreme XT (sup, 3M ESPE), Filtek Silorane (sil, 3M ESPE), Tetric EvoCeram (tec, Ivoclar Vivadent), Venus Diamond (ven, Heraeus Kulzer), Ceram X Mono (cxm, Dentsply DeTrey), Clearfil Majesty posterior (cmp, Kuraray), Herculite UltraXRV (hux,Kerr) Synerg D6 (syn,Coltene Whaledent), Grandio (grd, VOCO) and an experimental composite (exp, VOCO). The results showed superior properties for the highly filled composite exp, grd,cmp, and ven than Filtek Silorane.

Lien *et al.,* (2011) [90], evaluated the effect of high-heat storage on the flexural strength of four composite restorative materials (Filtek Supreme, Filtek LS, esthet-x and Durafil). The results showed no significant difference between groups per property based on storage temperature, however, significant differences were found based on composite type.

V. Modulus of Elasticity

Definition: The ratio of stress to corresponding strain below the proportional limit of a material.

The relationship between modulus of elasticity and polymerization shrinkage is the main challenge for maintenance of the adhesive interface, thus composites presenting high shrinkage values, associated with a high modulus of elasticity tend to disrupt the adhesive interface under polymerization [86].

Ilie and Hickel (2006) [61], examined the modulus of elasticity of silorane based composite material, and thereby compared the examined characteristics against those of well-known methacrylate-based composites. Modulus of elasticity of the silorane-based material was slightly lower.

Melo Monteiro *et al.,* (2009) [85], determined the modulus of elasticity (ME)of seven dental composite (Filtek Z350, Filtek Z250/3M ESPE; Grandio, Polofil Supra/VOCO; TPH Spectrum, TPH3, Esthet-X/Dentsply). The results showed Grandio composite had higher ME (13.91 GPa), Z350 (8.82 GPa) and Z250 (8.73GPa).

Ilie and Hickel (2009) [63], analyzed the modulus of elasticity of the silorane-based composite in comparison to six methacrylate-based composites after storing the materials for 24h in distilled water as well as after ageing (thermo cycling and storing for 4 weeks in water, saliva or alcohol). The modulus of elasticity from most analyzed composites decreased by storing in alcohol solution

Boaro *et al.,* (2010) [90], compared currently available low-shrinkage composites with others regarding shrinkage rate and elastic modulus. Seven BisGMA-based composites (Durafill/DU, Filtek Z250/FZ, Heliomolar/HM, Aelite LS Posterior/AP, Point 4/P4, Filtek Supreme/SU, ELS/EL), a silorane-based (Filtek LS, LS), a urethane-based (Venus Diamond, VD) and one based on a dimethacrylate-derivative of dimer acid (N'Durance, ND) were tested. Significance: Not all low-shrinkage composites demonstrated reduced polymerization shrinkage. Also, in order to effectively reduce polymerization stress, a low post-gel shrinkage must be associated to a relatively low elastic modulus.

Min *et al.,* (2010) [93], investigated the influence of shrinkage strain, modulus, and instrument compliance on polymerization stress of light-cured composites. A universal hybrid (Z250), flowable (Z350), and silorane-based (P90) composite were examined (n=5). A bonded disc method was used to measure the axial shrinkage strain. A dynamic oscillatory shear test using a custom-made rheometer was undertaken to measure the initial modulus development during light curing. At 10 s after light curing, modulus was highest for Z250 (466.2MPa), followed by Z350 (154.1MPa) and P90 (130.7MPa).

Youngchul *et al.,* (2012) [94], investigated the effect of layering methods, flowable composite liner and use of low shrinkage silorane-based composite on the polymerization shrinkage stress. A universal hybrid methacrylate-based composite (Z250), a flowable composite (Z350 flowable), and a silorane-based composite (P90) were used to fill the cavities. Cavities were restored using four different filling protocols. Results showed that the flexural modulus of P90 was 10.1 (0.9), Z250 was 13.6 (2.0), and that of Z350 flowable was 7.6 (0.9) GPa.

VI. The creep

Definition: time dependent plastic deformation occurring under stresses lower than the yielding stress.

A reduced amount of the resin matrix due to a high filler content and homogeneous filler distribution has been found to lead to higher creep resistance [95].

Ilie and Hickel (2006) [61] examined the creep of silorane based composite material, and thereby compared the examined characteristics against those of well-known methacrylate-based composites. The test procedure was carried out force controlled; the test load increased and decreased with constant speed between 0.4mN and 500mN. The load and the penetration depth of the indenter were continuously measured during the load–unload-hysteresis. By measuring the change in indentation depth with constant test force, a relative change in the indentation depth can be calculated. This is a value for the creep of the materials. Silorane based composite material achieved the lowest creep, the experimental flowable material showed high creep values (more plastic) as the regular flowable materials.

Watts and Marghalani (2007) [96], studied the time dependent viscoelastic deformation (creep and recovery) of new composite formulations with different matrix structures, under compressive load, after aging in food-simulating solvents of different solubility parameter. Four composite materials were studied, formulated with three different matrix structures. These were, respectively: silorane (Hermes, 3M-Espe); Ormocer (Experimental, Voco) and conventional dimethacrylate (Tetric-CeramHB, Ivoclar; X-trafil, Voco). The creep-strain ranged from minima of 0.72 (0.01) for the silorane and 0.82 (0.01) for the Ormocer, both in water, to the maximum of 3.48 (0.30) for Tetric-Ceram,silorane and Ormocer, exhibited viscoelastic stability in food-simulating solvents. But this behaviour was closely matched by one highly-filled dimethacrylate material.

Watts and Marghalani (2008) [93], compared several measures of material stability of a silorane-matrix composite with representative dimethacrylate-matrix composites. These included setting shrinkage and stress, visco-elastic creep/recovery and stability in solvents. Four groups of cylindrical specimens (4 x 6 mm) were prepared in molds, by complete light irradiation, and then conditioned in 3 solvents: methyl ethyl ketone (MEK), ethanol, and water for 1 month at 37 ^{o}C. The creep-strain under 35 MPa compressive stresses in 37 ^{o}C water was recorded continuously for 2 h and then the unloaded recovery-strain for 2 h. The data were statistically analyzed by ANOVA, Bonferroni's test and by linear regression. Silorane-matrix composite exhibited high dimensional and stress stability during and following photo-polymerization. This benefit was also found in visco-elastic-strain stability in compression after extended exposure to conditioning solvents of increasing power.

Ilie and Hickel (2009) [63], analyzed the creep of the silorane-based composite in comparison to six methacrylate-based composites after storing the materials for 24h in distilled water as well as after ageing (thermo cycling and storing for 4 weeks in water, saliva or alcohol). The creep was evaluated on samples using an automatic microhardness indenter (Fischerscope H100C, Fischer, Germany).The test procedure was carried out force controlled. The test load increased and decreased with constant speed between 0.4 and 500mN. The load and the penetration depth of the indenter were continuously measured during the load–unload-hysteresis. Their results showed increase in creep values for all methacrylate-based composites stored in alcohol. Only the creep values of the silorane-based material remained constant during all four types of storage.

Papadogiannis *et al.,* (2011) [98], evaluated the viscoelastic properties of novel low-shrinking composites and compare them to those of packable composites. Six materials were tested one of them Filtek Silorane (FS) materials were tested dry and wet at different temperatures (21°C to 50°C). Creep recovery was calculated among others. Significant differences were found both between the two groups and between materials belonging to the same group. All materials were softened by an increase of temperature, while Filtek Silorane was the least affected by water.

Ilie *et al.,* (2012) [99], analyzed differences in mechanical properties within and between modern flowable and non-flowable nano-hybrid and microhybrid RBCs by measuring mechanical properties at macro- and micro-scale. Thirty-four RBCs one of them Filtek Silorane. Creep was measured after the samples had been stored in water for 24 h at 37°C. The category of micro- and nano-hybrid RBCs performed in all properties superior compared to the flowable RBCs.

AIM OF THE WORK

The aim of the present work is to investigate selected mechanical properties at different storage conditions (distilled water, alcohol or artificial saliva) on three brands of composite (Silorane –based, Microhybrid methacrylate based and Nanohybrid methacrylate based):

A) Macro-mechanical characteristic:

1. The flexural strength.
2. The modulus of elasticity.

B) Micro-mechanical characteristic:

1. The Vickers hardness

MATERIALS

This study was designed to investigate the effect of different storage media namely, distilled water, Alcohol (Ethanol 70%) and artificial saliva (PH 7.0) on various properties of three resin composite.

Experimental Materials

Three composite restoratives from the same manufacturer were selected for this study

1. Silorane -based composite resin (Filtek^{TM} p90) *. (Fig.1)
2. Nanohybrid methacrylate based composite (Filtek Z250XT)*.(Fig.2)
3. Microhybrid methacrylate based composite (Filtek Z250)*. (Fig.3)

Shade A2 was Standardized for all tested materials. The chemical composition and the technical profiles of different materials according to the manufactures formulation are listed in (Table 1).

Table 1: Chemical composition of the tested materials

Material	Chemical composition
Filtek^{TM} P90	Matrix: New ring-opening Silorane Filler: Epoxy functional silane treated SiO2 and ytterbium fluoride (0.1-2 μm) Filler volume: 55% Filler by weight: 76%
Filtek^{TM} Z250 XT	Matrix: Bis-GMA, UDMA, Bis-EMA, PEGDMA, and TEGDMA. Filler: Surface-modified zirconia/silica (0. 1-10 μm) and 20 nm surface-modified silica. Filler by volume: 67.8% Filler by weight: 81.8%
Filtek^{TM} Z250	Matrix: Bis-GMA, UDMA, and Bis-EMA. Filler: zirconia/silica (0.01-3.5 μm). Filler by volume: 60% Filler by weight: 82%

*3M ESPE, st, Paul, MN, USA

The Storage Conditions

1. Artificial Saliva

Preparation of artificial saliva:

Artificial Saliva was prepared according to (Haeckel *et al.*, 1989) [100]. It was made according to the following composition

- Sodium chloride (Nacl)	0.4 gm
- Potassium chloride (Kcl)	1.21 gm
- Sodium hypophosphate (NaH2P04.2H20)	0.78 gm
- Sodium hyposulfied (Na2S.9 H20)	0.005 gm
- Urea (C0"NH2"2)	1.0 gm
- Deionized water	100 ml

The adjustment of the Ph of simulated saliva was performed by the addition of concentrated hydrochloric acid (94%) drop by drop with continuous mixing and using glass combined electrode of the pH meter until the desired pH was obtained.

2. Alcohol (Ethanol 70%)

3. Distilled Water

Celluloid Strips *

Curing Unit** (Fig.4)

Only one light curing unit was used in the study LED unit**, the diameter of the light curing tip was 8mm and the intensity was 1200 Mw/cm^2.

Thermocycling Machine*.** (Fig.5)

*Polyester matrix band, Microdont, Brazil

**HL-IV, 1200 mW/ cm^2, Guilin Woodpecker Medical Instrument co., ltd, China

*** Biomaterial Department Alexandria University

Fig. 1: Filtek ^{TM} Silorane P90 Composite Resin

Fig. 2: Filtek ^{TM} Z250XT Nanohybrid Composite Resin

Fig. 3: Filtek ^{TM} Z250 Microhybrid Composite Resin

Fig. 4: Curing unit

METHODS

I. Sample Gouping

A total of 189 specimens were prepared, equally divided into three main groups of 63 specimens each group. The first group was used to study the flexural strength and modulus of elasticity, while second group was used to study the Vickers micro-hardness test whereas; the third group was used to study the creep .Each group was randomly subdivided into three main subgroups of 21 each according to the restorative material used (A); where "A1": Filtek p90, "A2": Filtek Z250XT and "A3": Filtek Z250. The three main subgroups were further subdivided into three equal categories according to the storage medium (B); where "B1": distilled water, "B2": artificial saliva (pH.7) and "B3": alcohol, as shown in (Tables 2.A and 2.B).

Table 2.A: Variables of the study

	Variables	Symbol	Referred to
1	Restorative material	A_1 A_2 A_3	Filtek p90 Filtek Z250XT Filtek Z250
2	Storage medium	B_1 B_2 B_3	Distilled water Artificial saliva Alcohol

Table 2.B: Interaction of variables

Condition Test	**Distilled water**	**Artificial saliva**	**Alcohol**	**total**
Flexural Strength, Modulus Elasticity.	7samples of Filtek p90 (A_1,B_1)	7 samples of Filtek p90 (A_1,B_2)	7 samples of Filtek p90 (A_1,B_3)	**63**
	7samples of F Z250XT (A_2,B_1)	7samples of F Z250XT (A_2,B_2)	7 samples of F Z250XT (A_2,B_3)	
	7 samples of F Z250 (A_3,B_1)	7 samples of F Z250 (A_3,B_2)	7 samples of F Z250 (A_3,B_3)	
Vickers micro-hardness test	7 samples of Filtek p90 (A_1,B_1)	7 samples of Filtek p90 (A_1,B_2)	7 samples of Filtek p90 (A_1,B_3)	**63**
	7 samples of F Z250XT (A_2,B_1)	7 samples of F Z250XT (A_2,B_2)	7 samples of F Z250XT (A_2,B_3)	
	7 samples of F Z250 (A3,B1)	7 samples of F Z250 (A3,B2)	7 samples of F Z250 (A3,B3)	
The Creep	7 samples of Filtek p90 (A_1,B_1)	7 samples of Filtek p90 (A_1,B_2)	7 samples of Filtek p90 (A_1,B_3)	**63**
	7 samples of F Z250XT (A_2,B_1)	7 samples of F Z250XT (A_2,B_2)	7 samples of F Z250XT (A_2,B_3)	
	7 samples of F Z250 (A_3,B_1)	7 samples of F Z250 (A_3,B_2)	7 samples of F Z250 (A_3,B_3)	

II. Macro-Mechanical Characteristic

A. The Flexural Strength.

B. B. The Modulus of elasticity.

Mold Construction

A specially designed split Teflon mold ***(Fig.6)*** was fabricated to form rectangular bar specimens ***(Fig.7)*** with dimensions of 25mm length, 2mm in breadth and 2mm thickness. These dimensions were determined according to the recommendations of ISO (International Standards Organization) No 4049 specification (2000b) [115].

Specimens Preparation

The material was packed into the mold using a plastic spatula until the mold was slightly overfilled. A Celluloid strip was placed and gently pressed on top of the surface of the mold to remove any excess material, to avoid the presence of air bubbles and to ensure smooth surface finish. Each specimen was light cured for 20sec according to the manufacturer's instructions for material using visible light curing unit. After curing of the specimen, the Celluloid strip was removed and the mold was split to remove the specimen. The specimens were checked visually for any entrapment or chipped edges. Defective specimens were discarded, while any flashes were removed by using finishing disc grit 4000 to remove all flashes and to obtain smooth borders.

Storage Conditions

Each seven specimens of each restorative material were thermo-cycled in thermo cycling machine [1] (500 cycles at 5–55 ◦C) before transferred to a plastic tube containing 10ml distilled water or artificial saliva (PH.7) or Alcohol (Ethanol 70%). Then all specimens were stored in an incubator at 37± 1 Cº at 95-100% relative humidity for periods of 24 hours (distilled water) and one month (artificial saliva and Ethanol 70%) [63]. At the end of the storage periods, specimens were removed from their containers and gently blotted dry using cotton pellet. The specimens were marked with a pencil before testing to ensure proper adjustment on the machine; a central mark and a mark 2.5mm from each end of for the supporting ends. Flexure strength was measured at 24 hours and one month after storage to compare the effect of aging on it.

A. Flexural Strength Testing(FS)

Flexural strength, or bend strength, is defined as a material's ability to resist deformation under load. The flexural strength represents the highest stress experienced within the material at its moment of rupture. It is measured in terms of stress. Three point bend tests are commonly used to determine the flexural strength of a specimen [116].

The three point's flexural strength test was performed using a universal testing machine ** ***(fig.8)***. Each specimen was individually and horizontally mounted in a custom made fixture [three point bend test assembly; two parallel rods with span supporting the specimen, with the damage site centrally located on the tensile side] (fig.3&4) on a computer controlled materials testing machine with a load cell and data will recorded using computer software then the samples will statically compression loaded until fracture. The specimens were loaded to fracture using a cross head speed of 0.75mm/min. The maximum force necessary to produce fracture of the specimens was recorded in Newton s from the stress-strain curve.

** Lloyd mechartical testing machine

The Stress-strain curves will be recorded with computer software from the stress-strain curve, the flexural strength (FS).

FS represents the limiting stress at which failure or instability is imminent.

The calculation of FS is guided by the formulae:

FBS (ó) =3F (L)

$2wh^2$

Where;

F is the maximum load at the point of fracture,

L is pan (the distance between the two supports in millimeters)

W is the width of the sample

h is height,

B. The Modulus of Elasticity (E)

The measure of elasticity of a material is described by the term elastic modulus also referred to as modulus of elasticity or young's modulus. The elastic modulus represents the stiffness of a material within the elastic rang, it can be determined from The Stress-strain curves by calculation the ratio of stress/strain [117].

The modulus of elasticity was calculated from The Stress-strain curves record with computer software at same time during flexural strength test.

The value of E is the slope of the linear portion of the stress-strain curve expressed as the ratio of stress/strain.

The calculation of (E) is guided by the formulae:

E =L3/4 wh3 (P/Y)

Where;

L is pan (the distance between the two supports in millimeters)

W is the width of the sample

h is height,

P/Y is the slope of the linear part of the stress–strain curve within the elastic portion.

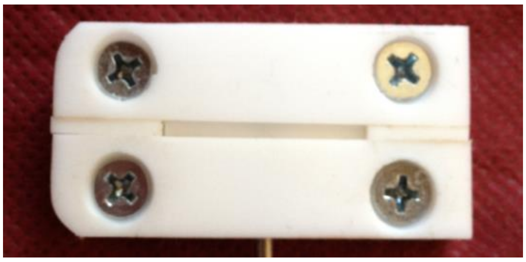


Fig. 6: Teflon mold 2×2×25 mm used for sample preparation

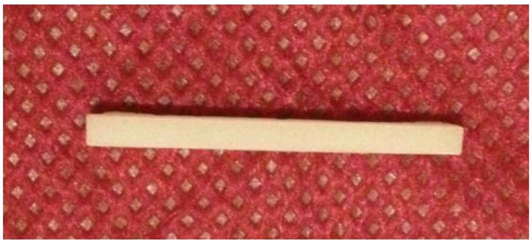


Fig.7: Bar shape specimen

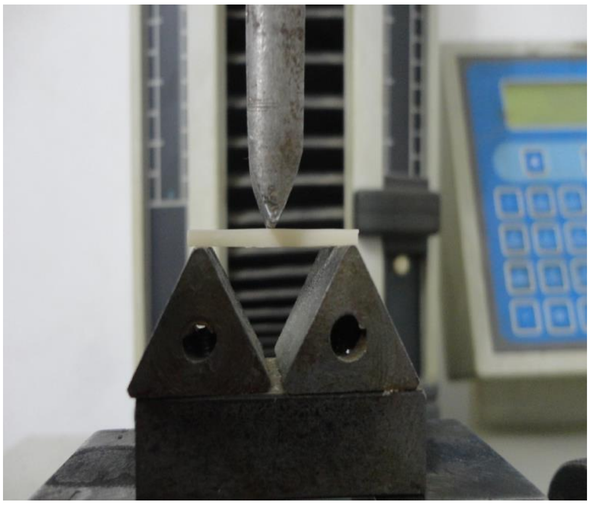


Fig. 8: The three point's test on universal testing machine

III. Micro-Mechanical Characteristic Hardness

Mold Construction

A specially designed split Teflon mold (Fig 9) was fabricated to form disc specimens (Fig.10) with dimensions of of size 6mm in diameter and 2mm in height.

Specimen Preparation

The composite material were condensed incrementally in the Teflon mold and covered with celluloid strip, then cured for 40 seconds for each increment by LED light curing unit with, it's curing tip placed on ^{2}direct contact with mold and perpendicular to it.

Storage Conditions

Each seven specimens of each restorative material were thermo-cycled(500 cycles at 5–55 ◦C) before transferred to a plastic tube containing 10ml distilled water or artificial saliva (PH.7) or Alcohol (Ethanol 70%). Then all specimens were stored in an incubator at 37± 1 Cº at 95-100% relative humidity for periods of 24 hours (distilled water) and one month (artificial saliva and Ethanol 70%) [63]. At the end of the storage periods, specimens were removed from their containers and gently blotted dry using cotton pellet.

The Vickers Hardness Test Procedure

Surface Micro-hardness of the specimens was determined using Digital Display Vickers Micro-hardness Tester [*](fig.11) with a Vickers diamond indenter. A load of 200g was applied to the surface of the specimens for 10 seconds. Five indentations, which were equally placed over a circle and not closer than 0.5 mm to the adjacent indentations, were made on the surface of each specimen. The diagonals length of the indentations were measured by built in scaled microscope[**] 20X objective lens and Vickers values were converted into micro-hardness values [20].

Micro-Hardness Calculation

Micro-hardness was obtained using the following equation:

HV=1.854 P/d2

Where,

HV is Vickers hardness in Kgf/mm2

P is the load in Kgf

d is the average diagonals lengths in mm

*(Model HVS-50, Laizhou Huayin Testing Instrument Co., Ltd. China).

** Shimadzu Ft/IR-8400 - Spectrophotometer, Japan.

Fig. 9: Teflon mold 2×6mm diameter

Fig.10: Disc shape specimen

Fig. 11: Vickers Micro-hardness Tester

IV. Time Dependent Mechanical Property (The Creep)

Mold Construction

A specially designed split Teflon mold (Fig.12) was fabricated to form cylinderical shape specimens (Fig.13) with dimensions of 4mm in diameter and 6mm in height [97].

Specimen Preparation

The Teflon mold was screwed over the transparent plastic slab. (Fig.5) The material was packed incrementally inside the mold until it was overfilled. A Celluloid strip was placed and gently pressed on top of the surface of the mold to remove any excess material, to avoid the presence of air bubbles and to ensure smooth surface finish. The photo-polymerization was initiated by illuminating the two surfaces (top & bottom) of the mold for 20 sec using visible light curing unit. Then the cylindrical specimen was taken off the mold and exposed to light from each side laterally for additional 20 sec in order to ensure polymerization depth [117].

Storage Conditions

Each seven specimens of each restorative material were thermo-cycled(500 cycles at 5–55 ◦C) before transferred to a plastic tube containing 10ml distilled water or artificial saliva (PH.7) or Alcohol (Ethanol 70%). Then all specimens were stored in an incubator at 37± 1 Cº at 95-100% relative humidity for periods of 24 hours (distilled water) and one month (artificial saliva and Ethanol 70%) [63]. At the end of the storage periods, specimens were removed from their containers and gently blotted dry using cotton pellet.

The Creep Test

It is defined as the time-dependent plastic deformation, or strain, of a material under a static load well below its conventional elastic limit [95].

It was performed on cylindrical-shaped specimen (6mm thickness and 4mm diameter) at constant load 50 MPa on a uniaxial compression rig of a universal testing machine. The stresses were maintained for 2 hours and the resultant percentage compressive strains were recorded as a function of time [97] (fig.14).

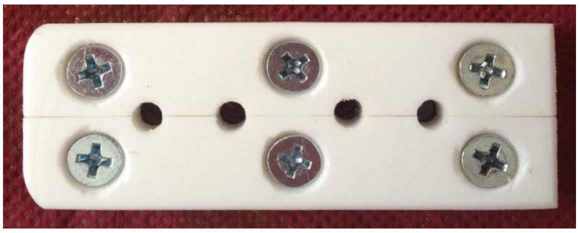


Fig. 12: Teflon mold 4×6mm diameter used for sample preparation

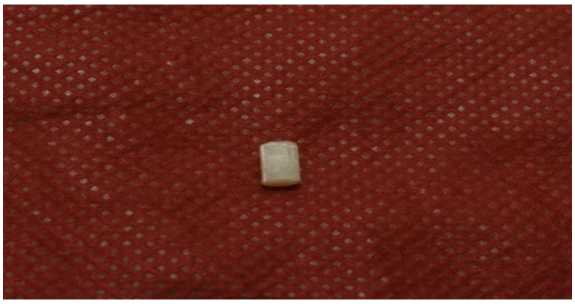


Fig. 13: Cylindrical shape specime

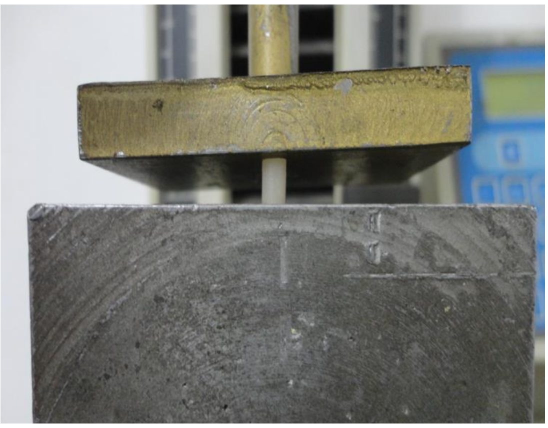


Fig. 14: The Creep test

Fig 5: Thermocycling machine

RESULTS

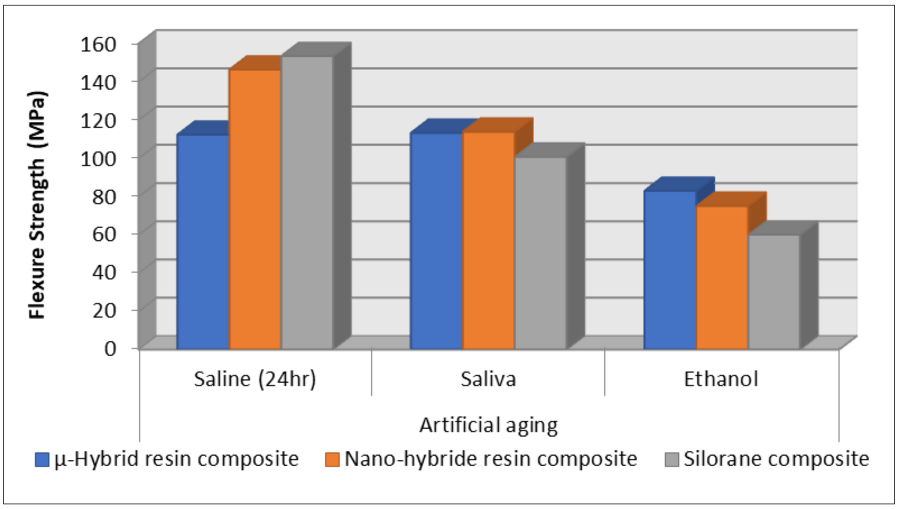
Data analysis was performed in several steps. Initially, descriptive statistics for each subgroup results. Two-way analysis of variance ANOVA tests of significance comparing variables affecting mean values. One way ANOVA followed by pair-wise Tukey's post-hoc tests were performed to detect significance between composite groups at different aging media. Statistical analysis was performed using Aasistat 7.6 statistics software for Windows. P values ≤ 0.05 are considered to be statistically significant in all tests.

1) Flexure Strength

The mean values and standard deviation of flexure strength (MPa) for all composite groups as function of artificial aging are summarized in table (3) and graphically drawn in ***(fig.12)***.

Table 3: Flexure strength results (Mean values± SDs) for all composite groups as function of artificial aging

Variables		**Artificial aging**		
		Saline (24hr)	Saliva	Ethanol
composite	Silorane composite	153.4 ± 11.21	100.8 ± 23.1	60.1 ± 4.34
	Nano-hybride resin composite	146.4 ± 13.62	113.9 ± 6.69	75.2 ± 8.77
	µ-Hybrid resin composite	113.3 ± 12.15	112.4 ± 3.23	82.9 ± 6.03



Graph 1: Histogram of flexure strength mean values for all composite groups as function of artificial aging

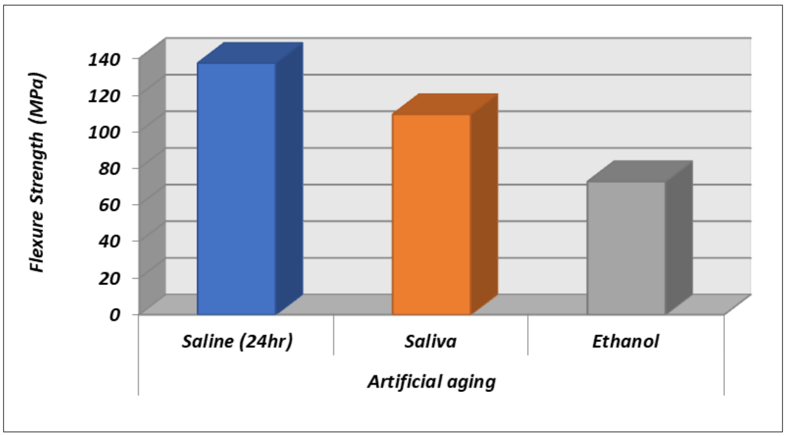
Effect of Artificial Aging

Irrespective of composite, totally it was found that Saline (24hr) aging group recorded the highest flexure strength mean value (137.4 ± 12.68 MPa) followed by Saliva aging group (109.3 ± 4.262 MPa) while Ethanol aging group recorded the lowest mean value (72.73 ± 6.725 MPa). The difference in flexure strength mean values between composite groups was statistically significant (P<0.05).

Table 4: Comparison of total flexure strength results (Mean values± SDs) as function of artificial aging

Variable		Mean ± SD	Tukey's rank	Statistics
Artificial aging	Saline (24hr)	137.4 ± 12.68	A	P value
	Saliva	109.3 ± 4.262	B	<0.0001*
	Ethanol	72.73 ± 6.725	C	

Same letter in the same column indicating non-statistically significant difference (Tukey's test ;*p* > 0.05).
*; significant (*p* < 0.05) ns; non-significant (p>0.05)



Graph 2: A column chart of total flexure strength mean values for different artificial aging

Interaction between Variables

- **Saline (24hr) aging;** it was found that Silorane composite group recorded the highest flexure strength mean value followed by Nano-hybride resin composite group while μ-Hybrid resin composite group recorded the lowest mean value. The difference in flexure strength mean values between composite groups was statistically significant (P<0.05).
- **Saliva aging;** it was found that Nano-hybride resin group recorded the highest flexure strength mean value followed by μ-Hybrid resin composite group while Silorane composite group recorded the lowest mean value. The difference in flexure strength mean values between composite groups was statistically non-significant (P>0.05).
- **Ethanol aging;** it was found that μ-Hybrid resin group recorded the highest flexure strength mean value followed by Nano-hybride resin composite group while Silorane composite group recorded the lowest mean value. The difference in flexure

strength mean values between composite groups was statistically non-significant (P>0.05).

- **µ-Hybrid resin composite;** it was found that Saline (24hr) aging group recorded the highest flexure strength mean value followed by Saliva aging group while Ethanol aging group recorded the lowest mean value. The difference in flexure strength mean values between composite groups was statistically significant (P<0.05).
- **Nano-hybrid resin composite;** it was found that Saline (24hr) aging group recorded the highest flexure strength mean value followed by Saliva aging group while Ethanol aging group recorded the lowest mean value. The difference in flexure strength mean values between composite groups was statistically significant (P<0.05).
- **Silorane composite;** it was found that Saline (24hr) aging group recorded the highest flexure strength mean value followed by Saliva aging group while Ethanol aging group recorded the lowest mean value. The difference in flexure strength mean values between composite groups was statistically significant (P<0.05).

Table 5: Comparison of flexure strength results (Mean values± SDs) as function of composite groups and artificial aging

Composite / **Artificial aging**	**Saline (24hr)**	**Saliva**	**Ethanol**	**ANOVA**
Silorane composite	$153.4^{A}_{a} \pm 11.21$	$100.8^{A}_{b} \pm 23.1$	$60.1^{A}_{c} \pm 4.34$	<0.0001*
Nano-hybride resin composite	$146.4^{A}_{a} \pm 13.62$	$113.9^{A}_{b} \pm 6.69$	$75.2^{A}_{c} \pm 8.77$	<0.0001*
µ-Hybrid resin composite	$113.3^{B}_{a} \pm 12.15$	$112.4^{A}_{a} \pm 3.23$	$82.9^{A}_{b} \pm 6.03$	0.0009*
ANOVA	0.0044*	0.5450 ns	0.0692 ns	

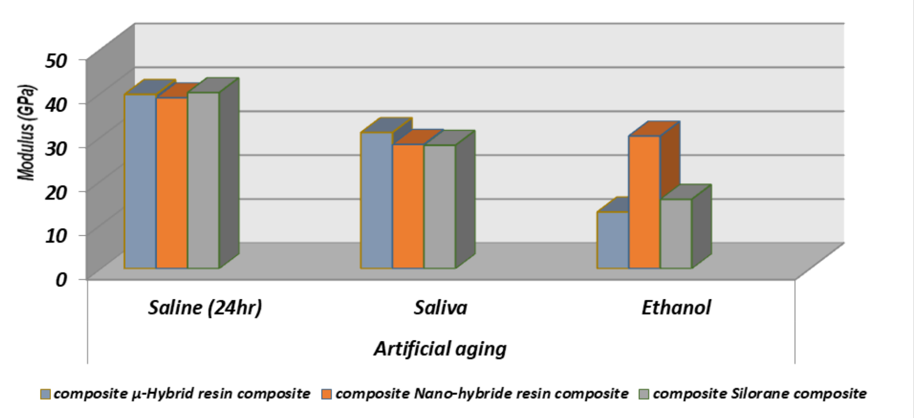
Different superscript letter in the same column indicating statistically significant between composite (Tukey's test ;$p < 0.05$). Different subscript letter in the same row indicating statistically significant between aging (Tukey's test ;$p > 0.05$). *; significant ($p < 0.05$) ns; non-significant ($p > 0.05$)

2) Young's Modulus

The mean values and standard deviation of Young's Modulus (GPa) for all composite groups as function of artificial aging are summarized in table (6) and graphically drawn in ***(fig.14).***

Table 6: Young's Modulus results (Mean values± SDs) for all composite groups as function of artificial aging

Variables		**Artificial aging**		
		Saline (24hr)	Saliva	Ethanol
composite	Silorane composite	40.1 ± 1.9	28.1± 3.7	15.8 ± 1.24
	Nano-hybride resin composite	38.9 ± 4.19	28.3 ± 1.75	30.2 ± 3.1
	µ-Hybrid resin composite	9.7 ± 0.78	31.01 ± 1.14	12. 9 ± 1.67



Graph 3: Histogram of Young's Modulus mean values for all composite groups as function of artificial aging

Effect of Artificial Aging

Irrespective of composite, totally it was found that Saline (24hr) aging group recorded the highest Young's Modulus mean value (29.57 ± 4.2 GPa) followed by Saliva aging group (29.13 ± 0.39 GPa) while Ethanol aging group recorded the lowest mean value (19.64 ± 2.26 GPa). The difference in Young's Modulus mean values between composite groups was statistically significant (P<0.05). Pair-wise Tukey's post-hoc showed no significant difference between saline and saliva aging (p>0.05).

Table 7: Comparison of total Young's Modulus results (Mean values± SDs) as function of artificial aging

Variable		**Mean ± SD**	**Tukey's rank**	**Statistics**
Artificial aging	Saline (24hr)	29.57 ± 4.2	A	P value
	Saliva	29.13 ± 0.39	A	<0.0001*
	Ethanol	19.64 ± 2.26	B	

Same letter in the same column indicating non-statistically significant difference (Tukey's test ;$p > 0.05$).
*; significant ($p < 0.05$) ns; non-significant (p>0.05)

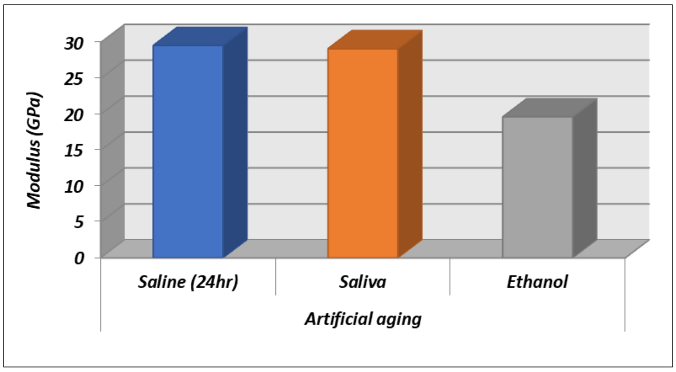


Figure 4: A column chart of total Young's Modulus mean values for different artificial aging

Interaction between Variables

- **Saline (24hr) aging;** it was found that Silorane composite group recorded the highest Young's Modulus mean value followed by Nano-hybride resin composite group while µ-Hybrid resin composite group recorded the lowest mean value. The difference in Young's Modulus mean values between composite groups was statistically significant (P<0.05).
- **Saliva aging;** it was found that µ-Hybrid resin group recorded the highest Young's Modulus mean value followed by Nano-hybride resin composite group while Silorane composite group recorded the lowest mean value. The difference in Young's Modulus mean values between composite groups was statistically non-significant (P>0.05).
- **Ethanol aging;** it was found that Nano-hybride resin group recorded the highest Young's Modulus mean value followed by Silorane composite group while µ-Hybrid resin composite group recorded the lowest mean value. The difference in Young's Modulus mean values between composite groups was statistically significant (P<0.05).
- **µ-Hybrid resin composite;** it was found that Saliva aging group recorded the highest Young's Modulus mean value followed by Ethanol aging group while Saline (24hr) aging group recorded the lowest mean value. The difference in Young's Modulus mean values between composite groups was statistically significant (P<0.05).
- **Nano-hybrid resin composite;** it was found that Saline (24hr) aging group recorded the highest Young's Modulus mean value followed by Ethanol aging group while Saliva aging group recorded the lowest mean value. The difference in Young's Modulus mean values between composite groups was statistically non-significant (P>0.05).
- **Silorane composite;** it was found that Saline (24hr) aging group recorded the highest Young's Modulus mean value followed by Saliva aging group while **Ethanol** aging group recorded the lowest mean value. The difference in Young's Modulus mean values between composite groups was statistically significant (P<0.05).

Table 8: Comparison of Young's Modulus results (Mean values± SDs) as function of composite groups and artificial aging

Composite Artificial aging	Saline (24hr)	Saliva	Ethanol	ANOVA
µ-Hybrid resin composite	39.7^{B}_{b} ± 0.78	31.01^{A}_{a} ± 1.14	$12.\ 9^{B}_{b}$ ± 1.67	<0.0001*
Nano-hybride resin composite	38.9^{A}_{a} ± 4.19	28.3^{A}_{a} ± 1.75	30.2^{A}_{a} ± 3.1	0.0704 ns
Silorane composite	40.1^{A}_{a} ± 1.9	28.1^{A}_{b}± 3.7	15.8^{B}_{c} ± 1.24	<0.0001*
ANOVA	<0.0001*	0.6284ns	0.0014*	

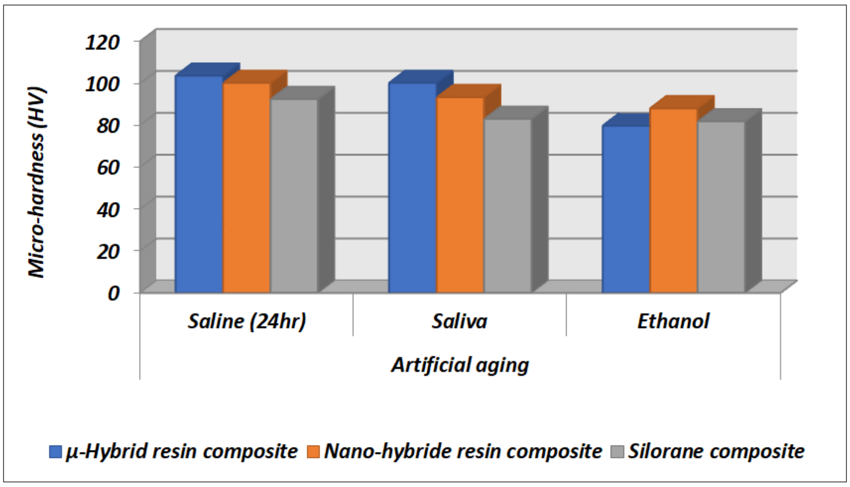
Different superscript letter in the same column indicating statistically significant between composite (Tukey's test ;$p < 0.05$). different subscript letter in the same row indicating statistically significant between aging (Tukey's test ;$p > 0.05$).
*; significant ($p < 0.05$) ns; non-significant (p>0.05)

3) Micro-Hardness

The mean values and standard deviation of micro-hardness (HV) for all composite groups as function of artificial aging are summarized in table (9) and graphically drawn in figures (16).

Table 9: Micro-hardness results (Mean values± SDs) for all composite groups as function of artificial aging

Variables		**Artificial aging**		
		Saline (24hr)	Saliva	Ethanol
Composite	Silorane composite	92.5 ± 2.27	82.99 ± 0.42	81.8 ± 1.78
	Nano-hybride resin composite	100.2 ± 1.48	93.4 ± 1.37	88.03 ± 0.4
	µ-Hybrid resin composite	103.6 ± 1.61	100.2 ± 1.88	79.7 ± 0.77



Graph 5: Histogram of micro-hardness mean values for all composite groups as function of artificial aging

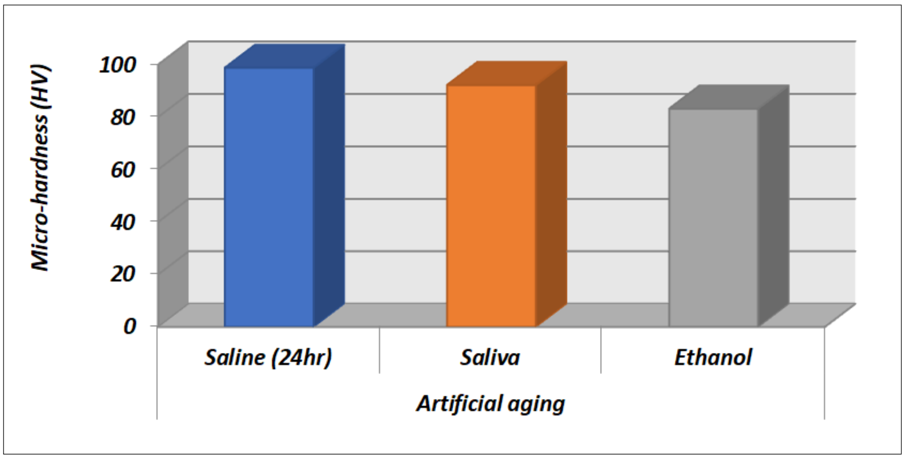
Effect of Artificial Aging

Irrespective of composite, totally it was found that Saline (24hr) aging group recorded the highest micro-hardness mean value (98.74 ± 1.387 HV) followed by Saliva aging group (92.17 ± 2.109 HV) while Ethanol aging group recorded the lowest mean value (83.19 ± 1.053 HV). The difference in micro-hardness mean values between composite groups was statistically significant (P<0.05).

Table 10: Comparison of total micro-hardness results (Mean values± SDs) as function of artificial aging

Variable		Mean ± SD	Tukey’s rank	Statistics
Artificial aging	Saline (24hr)	98.74 ± 1.387	A	P value
	Saliva	92.17 ± 2.109	B	<0.0001*
	Ethanol	83.19 ± 1.053	C	

Same letter in the same column indicating non-statistically significant difference (Tukey’s test ;$p > 0.05$).
*; significant ($p < 0.05$) ns; non-significant (p>0.05)



Graph 6: A column chart of total micro-hardness mean values for different artificial aging

Interaction between Variables

- **Saline (24hr) aging;** it was found that µ-Hybrid resin composite group recorded the highest micro-hardness mean value followed by Nano-hybride resin composite group while Silorane composite group recorded the lowest mean value. The difference in micro-hardness mean values between composite groups was statistically significant (P<0.05). Pair-wise Tukey's post hoc test showed no significant difference between µ-Hybrid and Nano-hybride resin composite group (p>0.05)
- **Saliva aging;** it was found that µ-Hybrid resin composite group recorded the highest micro-hardness mean value followed by Nano-hybride resin composite group while Silorane composite group recorded the lowest mean value. The difference in micro-hardness mean values between composite groups was statistically significant (P<0.05).
- **Ethanol aging;** it was found that Nano-hybride resin group recorded the highest micro-hardness mean value followed by Silorane composite group while µ-Hybrid resin composite group recorded the lowest mean value. The difference in micro-hardness mean values between composite groups was statistically non-significant (P>0.05). Pair-wise Tukey's post hoc test showed no significant difference between µ-Hybrid and Silorane composite group (p>0.05)
- **µ-Hybrid resin composite;** it was found that Saline (24hr) aging group recorded the highest micro-hardness mean value followed by Saliva aging group while Ethanol aging group recorded the lowest mean value. The difference in micro-hardness mean values between composite groups was statistically significant (P<0.05).
- **Nano-hybrid resin composite;** it was found that Saline (24hr) aging group recorded the highest micro-hardness mean value followed by Saliva aging group while Ethanol aging group recorded the lowest mean value. The difference in micro-hardness mean values between composite groups was statistically significant (P<0.05).
- **Silorane composite;** it was found that Saline (24hr) aging group recorded the highest micro-hardness mean value followed by Saliva aging group while Ethanol aging group recorded the lowest mean value. The difference in micro-hardness mean values between composite groups was statistically significant (P<0.05). Pair-wise Tukey's post hoc test showed no significant difference between Saliva and ethanol aging (p>0.05)

Table 11: Comparison of micro-hardness results (Mean values± SDs) as function of composite groups and artificial aging

Composite **Artificial aging**	**Saline (24hr)**	**Saliva**	**Ethanol**	**ANOVA**
Silorane composite	92.5^{B}_{a} ± 2.27	82.99^{C}_{b} ± 0.42	81.8^{B}_{b} ± 1.78	0.0002*
Nano-hybride resin composite	100.2^{A}_{a} ± 1.48	93.4^{B}_{b} ± 1.37	88.03^{A}_{c} ± 0.4	<0.0001*
µ-Hybrid resin composite	103.6^{A}_{a} ± 1.61	100.2^{A}_{b} ± 1.88	79.7^{B}_{c} ± 0.77	<0.0001*
ANOVA	0.0003*	<0.0001*	<0.0001*	

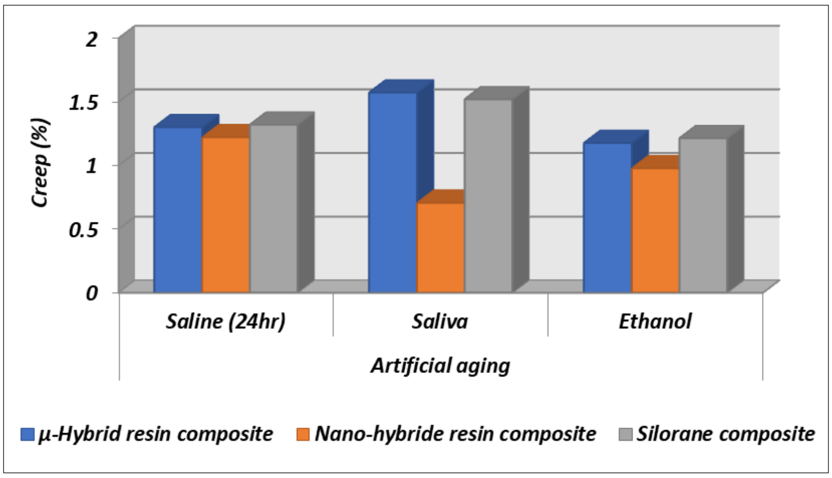
Different superscript letter in the same column indicating statistically significant between composite (Tukey's test ;$p < 0.05$)different subscript letter in the same row indicating statistically significant between aging (Tukey's test ;$p > 0.05$). *; significant ($p < 0.05$) ns; non-significant (p>0.05)

4) Creep

The mean values and standard deviation of creep (%) for all composite groups as function of artificial aging are summarized in table (12) and graphically drawn in ***(fig.18)***.

Table 12: Creep results (Mean values± SDs) for all composite groups as function of artificial aging

Variables		**Artificial aging**		
		Saline (24hr)	Saliva	Ethanol
composite	Silorane composite	1.32 ± 0.05	1.520 ± 0.04	1.214± 0.19
	Nano-hybride resin composite	1.222 ± 0.04	0.707 ± 0.09	0.978± 0.01
	µ-Hybrid resin composite	1.301 ± 0.01	1.572 ± 0.12	1.176 ± 0.1



Graph 7: Histogram of creep mean values for all composite groups as function of artificial aging

Effect of Artificial Aging

Irrespective of composite, totally it was found that Saline (24hr) aging group recorded the highest creep mean value (1.281 ± 0.021 %) followed by Saliva aging group (1.266 ± 0.198 %) while Ethanol aging group recorded the lowest mean value (1.123 ± 0.051 %). The difference in creep mean values between composite groups was statistically non-significant (P>0.05).

Table 13: Comparison of total creep results (Mean values± SDs) as function of artificial aging

Variable		**Mean ± SD**	**Tukey's rank**	**Statistics**
Artificial aging	Saline (24hr)	1.281 ± 0.021	A	P value
	Saliva	1.266 ± 0.198	A	0.1342 ns
	Ethanol	1.123 ± 0.051	A	

Same letter in the same column indicating non-statistically significant difference (Tukey's test ;$p > 0.05$).
*; significant ($p < 0.05$) ns; non-significant (p>0.05)

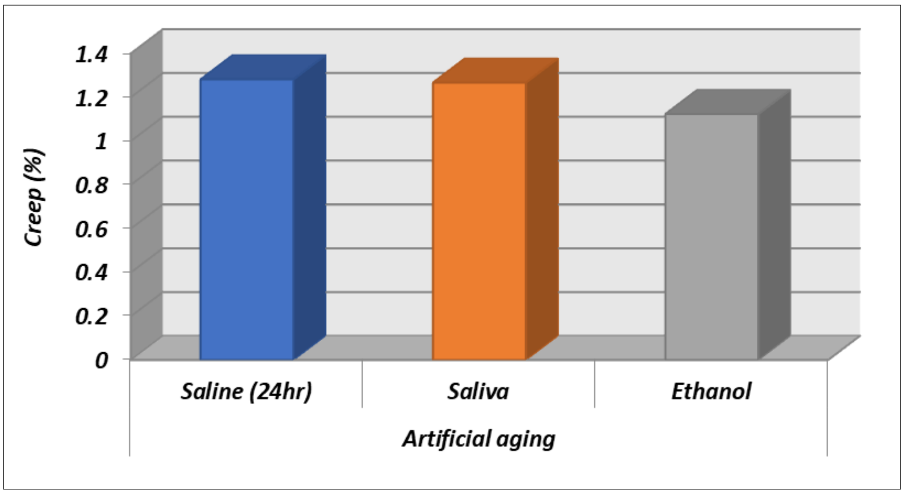


Figure 8: A column chart of total creep mean values for different artificial aging

Interaction between Variables

- **Saline (24hr) aging;** it was found that Silorane composite group recorded the highest creep mean value followed by µ-Hybrid resin composite group while Nano-hybride resin composite group recorded the lowest mean value. The difference in creep mean values between composite groups was statistically non-significant (P>0.05).
- **Saliva aging;** it was found that µ-Hybrid resin composite group recorded the highest creep mean value followed by Silorane composite group while Nano-hybride resin composite group recorded the lowest mean value. The difference in creep mean values between composite groups was statistically significant (P<0.05). Pair-wise Tukey's post hoc test showed no significant difference between µ-Hybrid and Silorane composite group (p>0.05)
- **Ethanol aging;** it was found that Silorane composite group recorded the highest creep mean value followed by µ-Hybrid resin composite group

while Nano-hybride resin composite group recorded the lowest mean value. The difference in creep mean values between composite groups was statistically non-significant (P>0.05).

- **µ-Hybrid resin composite;** it was found that Saliva aging group recorded the highest creep mean value followed by Saline (24hr) aging group while Ethanol aging group recorded the lowest mean value. The difference in creep mean values between composite groups was statistically non-significant (P>0.05).
- **Nano-hybrid resin composite;** it was found that Saline (24hr) aging group recorded the highest creep mean value followed by Ethanol aging group while Saliva aging group recorded the lowest mean value. The difference in creep mean values between composite groups was statistically non-significant (P>0.05).
- **Silorane composite;** it was found that Saliva aging group recorded the highest creep mean value followed by Saline (24hr) aging group while Ethanol aging group recorded the lowest mean value. The difference in creep mean values between composite groups was statistically non-significant (P>0.05).

Table 14: Comparison of creep results (Mean values± SDs) as function of composite groups and artificial aging

Composite **Artificial aging**	**Saline (24hr)**	**Saliva**	**Ethanol**	**ANOVA**
Silorane composite	$1.32^{A}_{a} \pm 0.05$	$1.520^{A}_{a} \pm 0.04$	$1.214^{A}_{a} \pm 0.19$	0.6718 ns
Nano-hybride resin composite	$1.222^{A}_{a} \pm 0.04$	$0.707^{B}_{b} \pm 0.09$	$0.978^{A}_{ab} \pm 0.01$	0.1459 ns
µ-Hybrid resin composite	$1.301^{A}_{ab} \pm 0.01$	$1.572^{A}_{a} \pm 0.12$	$1.176^{A}_{b} \pm 0.1$	0.1289 ns
ANOVA	0.1960 ns	<0.0001*	0.0779 ns	

Different superscript letter in the same column indicating statistically significant between composite (Tukey's test ;$p < 0.05$). Different subscript letter in the same row indicating statistically significant between aging (Tukey's test ;$p > 0.05$).
*; significant ($p < 0.05$) ns; non-significant (p>0.05)

DISCUSSION

The demand for direct esthetic restorations are still increasing consequently the improvement of resin composite properties is essential to obtain reliable and long-lasting clinical results. The absorption of water molecules by hydrophilic moieties within a resin composite material on exposure to the oral environment may result in plasticization of the resin polymer network thereby decreasing the mechanical properties of the material [101]. The present investigation combined mechanical properties testing after immersion of materials in different storage solutions to study the effect environmental conditions on biodegradation of different composite brands.

A) Macro-Mechanical Characteristic

1. Flexural Strength

The three-point bending test is based on the International Organization for Standardization (ISO) specification No 4049 (2000b) for polymer-based restoratives (25 x 2 x2mm) and was widely employed in dental research [107]. Flexural Strength is a more clinically relevant test of material strength and is especially important if the material used for Class I, II and IV cavities, which are usually subjected to high forces.

The flexural bending test, classified as opening mode test or Mode I, is usually recommended because the specimen fabrication and the load application are quite simple [106]. Although some studies have suggested alternative flexural test designs the three-point bending test still the choice for evaluating composites flexural strength due to the lower standard deviation, the lower coefficient of variation and the less complex crack distribution produced by it when compared to those produced by other test designs [107].

The results of present study showed that there was significant difference in terms of flexural strength between the silorane composite based which is higher than methacrylate based composite after 24 hours storage in distilled water.

The results of the present study were in agreement with the study of Lien and Vandewalle (2010) [69], they said The silorane-based material had relatively higher flexural strength than methacrylate-based composites.

Could be due to the silorane-based composite revealed a decreased water sorption, solubility and associated diffusion coefficient compared with conventional methacrylate-based composites [63]. Silorane-based composite exhibiting no change or the smallest changes when stored in water for 24 h, this stability is attributed to its monomer chemistry, The siloxane backbone of silorane is highly hydrophobic thus making the reactive to water oxirane groups inaccessible to water molecules [120].

The results of the present study were in dissimilarity with the study of Weinmann *et al.,* (2005) [115] who found that the flexural strength of silorane based composite was significantly lower than methacrylate based composite, this finding could be due to difference in methodology as the sample was not stored for 24 h the first measurement was performed 5 min after curing.

But after thermo cycling and storage in saliva for four weeks the results of the present study showed the microhybrid methacrylate composite exhibited more stable strength than silorane composite which showed significant reduction in the strength compared to 24 hours storage in distilled water.

This could be attributed to the difference in chemical composition of the organic matrix between the two composite materials. the methacrylate based composite had resin matrices composed of Bis-GMA, Bis-EMA, UDMA, and TEGDMA. Except for Bis-EMA, which is an ethoxylated version of Bis-GMA, othermolecules (Bis-GMA, UDMA, and TEGDMA) have hydroxyl groups which promote water sorption. As for Filtek Silorane, it had 3,4-epoxycyclohexyl-cyclopolymethylsiloxane. The cyclosiloxane backbone imparted hydrophobicity, thereby curtailing water sorption, [15] so may be rate of water sorption was different in methacrylate based composite it was high in first 24h and increased gradually for silorane over 4 weeks storage which explain the difference.

The results of the present study were in disagreement with the study of Ilie and Hickel (2009) [63] who found the new matrix silorane based composite revealed a stable strength after 4 weeks of storage in the tested saliva compared to the methacrylate based composite which showed significant reduction in the strength after storage. This finding could be due to difference in methodology as they measured the flexural strength according to (ISO/DIN 4049:1988) on bar-shaped specimens (16mm×2mm×2mm).

The results of the present study showed, after thermo cycling and 4 weeks storage in alcohol that, the methacrylate composite and silorane based composite exhibited reduction in the strength after storage for 4 weeks in alcohol. Our results were in agreement with Letícia C. B *et al.,* (2013) [110] who found the methacrylate composite and silorane composite presented different behaviors regarding aging in ethanol, as evidenced by the large range in values of percentage reduction for flexural strength (25–86%).

Zhang and Xu (2008) [121] Indeed, the flexural strength and hardness of methacrylate composite decreased drastically in the aqueous ethanol solution, as compared to the extent in decrease for all the composites in distilled water.

As organic solvents like ethanol have the potential for polymer damage. It can penetrate the resin matrix fully and promote the release of inreacted monomers. The partial dissolving of the resin matrix may result in the degradation of the filler-matrix interface, thereby impairing the flexural strength [54].

The results of the present study were in dissimilarity with the study of Yesilyurt *et al.,* (2009) [54] who found that after storage in alcohol the flexural strength of silorane based composite were not significantly affected and show stable strength in the tested solvent. Conversely, the flexural strength of the methacrylate composite showed significant reduction after conditioning in the tested solvent. , this difference may be due to shortening of the storage period to 7 days.

Also the present finding was in incongruity with the study of Ilie and Hickel (2009) [63] who found the new matrix silorane based composite revealed a stable strength after 4 weeks of storage in the ethanol compared to the methacrylate based composite which showed significant reduction in the strength after storage. this finding could be due to difference in methodology as they measured the flexural strength according to (ISO/DIN 4049:1988) on bar-shaped specimens (16mm×2mm×2mm).

2. Modulus of Elasticity

Another important mechanical parameter provided by the flexural test is the modulus of elasticity, which describes the rigidity of the material. Different clinical situations demand resin-based restorative materials with different moduli of elasticity. Class V (cervical) cavities; for example, demand a low modulus restorative material to flex with the tooth. A relatively high modulus, on the other hand, is expected from posterior composites to withstand the occlusal forces and preserve the adhesive interface [108]. Several authors have reported a significant correlation between the modulus of elasticity and the percentage of filler by volume (vol %) 3, 7 or by weight (wt %) [109].

Regardless to aging (24h saline), this study showed that was significant difference in Young's Modulus of silorane based composite and that of methacrylate based composite ,which in agreement Lien and Vandewalle (2010) [67] who found silorane based composite has higher Modulus than micro hybride methacrylate based composite.

Probably because the tested materials have dissimilar filler chemical compositions, the inorganic contents of Filtek LS are quartz particles, whose spatial orientation can be described as a crystalline solid of interconnected SiO4 tetrahedra and classified as tectosilicate. Conversely, Filtek Z250 include glassand zirconia-filler particles. It is not clear how filler-matrix molecular dynamics and spatial configuration of chemical bonds between SiO4 atoms can dictate the macroscopic stiffness of matter and influence overall material properties such as flexural strength and flexural modulus [67].

The results of the present study showed that no significant difference between the effect of saline and saliva aging on both the silorane based composite and methacrylate based composite in term of Young's Modulus.

While Ethanol aging group recorded the lowest mean value, The difference in Young's Modulus between saline, saliva and the effect of Ethanol on both the silorane based composite and methacrylate based composite was statistically significant. Our results were in agreement with Letícia C. B *et al.,* (2013) [110] who found the methacrylate composite and silorane composite presented different behaviors regarding aging in ethanol, as evidenced by the large range in values of percentage reduction for elastic modulus (26–75%).

The results of the present study are in dissimilarity with the study of Ilie and Hickel (2009) [63] who found the new matrix silorane based composite revealed a stable Modulus after 4 weeks of storage in the ethanol compared to the methacrylate based composite which showed significant reduction in the Modulus after storage. This finding could be due to difference in methodology as they measured the flexural strength according to (ISO/DIN 4049:1988) on bar-shaped specimens (16mm×2mm×2mm).

B) Micro-Mechanical Characteristic

- **Vickers Hardness Testing**

Hardness is considered to be related to wear resistance and is the most commonly examined mechanical property indicator for synthetic restorative materials.(103) Composite-related factors affecting strongly surface hardness of the material include filler particle size, type, morphology, distribution, volume, fraction and diluent's and inorganic filler content of resin composites. Composites with harder filler particles and higher filler load exhibit higher surface hardness [104].

Additionally, resin composition, type and degree of polymerization significantly affect the hardness of the restorative materials [105].

Vickers microhardness test was selected for this study because it is relatively a simple technique, very popular and reliable for obtaining the results. Additionally, it is considered by several authors as an indicator for the degree of polymerization of resin materials and used commonly as indirect method to evaluate degree of cure [66].

The result of the present study showed that, the silorane based composite had lower surface micro hardness than methacrylate based composite after 24 hours storage in distilled water this.

Palin and colleagues (2005) [118] reported that the degree of converging of experimental Silorane H1 averaged 50.2% after 24 hours.

Kusgoz *et al.,* (2011) [75] reveled that the degree of converging of Filtek Silorane (43.5%-56.4%) was significantly less when compared with methacrylate composite (63.5%- 67.5%). The decrease in the degree of converging of Filtek Silorane compared with methacrylate composite could be related to the difference in monomer chemistry. Additionally, variety in the filler size, filler volume, and filler type of the methacrylate and silorane which has less filler (76%) than methacrylate (82,84%) could explain the difference.

The results of Hahnel *et al.,* (2010) [56] Atali and Topbasi (2010) [71] Hahnel *et al.,* (2011) [74] revealed that the Filtek silorane composite showed low microhardness when compared to microhybrid and Nanohybrid methacrylate composite. These results were in agreement with the present study.

In controversy the results of the present study was in disagreement with the study of Ilie and Hickel (2006) [61] who found no significant difference between methacrylate composite and silorane based composite in terms of hardness. This difference may be due to difference in the methodology of specimens preparation, the specimens cured and then stored in 37ºC distilled water for 24 hours, then sectioned in the middle prior to testing with a slow-speed diamond saw under water cooling, and polished with a diamond suspension (mean grain size 1µm).Ten measurement were made starting at 0.1mm under the surface with 100-µm intervals between the measuring points.

Additionally the results of the present study was in incongruity with the study of Marghalani (2010) [66] who found that the silorane based composite had higher surface hardness than methacrylate based composite this finding may be due to difference in methodology as they measured the hardness on the upper and lower surface of (8x2 mm) samples that were finished manually from both sides after the preparation, with 1000 grit silorane carbide (SiC) abrasive paper as well as 5 and 1µm aluminum oxide slurry pastes for 5 seconds each step. Then the specimens were aged-dry either in dark bottles at 23 ± 0.5ºC or incubator at 37 ± 0.5ºC.

Also the results of the present study was in disagreement with the study of Hooshmand *et al.,* (2013) [112] they said there was no statistically significant difference among microhardness of microhybrid and nanohybrid methacrylate composite and silorane based composite This could be attributed to difference in the methodology . They used total of 50 non-carious extracted human teeth after debridement and standard Class V cavities were prepared.

But after thermo cycling and storage in Saliva and aging for four weeks the results of the present study showed the silorane based and methacrylate based composite exhibited significant reduction in surface hardness compared to 24 hours storage in distilled water. Our results were in agreement with Mayworm *et al.,* (2008) [102] Hahnel *et al.,* (2010) [56] and Jiang *et al.,* (2011) [113].

This finding was in inconsistency with the study of Yesilyurt *et al.,* (2009) [54] who found the hardness of methacrylate based composite significantly decreased while the silorane composite revealed stable hardness after conditioning in saliva, this difference may be due to shortening of the storage period to 7 days.

Also the present finding was in disagreement with the study of Ilie and Hickel (2009) [63] who found that except for the silorane-based composite, all methacrylate based composite showed a statistical significant decrease in hardness by storing in saliva solution. This difference may be due to they used bar-shaped specimens not disc like this study and different load force 500mN.

The results of the present study showed both type of composite exhibited a significant decrease in hardness after thermo cycling and storage in alcohol (ethanol) for 4 weeks, as compared to the extent in decrease in distilled water.

As organic solvents like ethanol have the potential for polymer damageing. It could 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, thereby impairing the hardness [54].

The present finding was in disagreement with the study of Yesilyurt *et al.,* (2009) [54] who found the hardness of methacrylate based composite significantly decreased while the silorane composite revealed stable hardness after conditioning in ethanol, this difference may be due to shortening of the storage period to 7 days.

C) Time Dependent Mechanical Property

- **The Creep**

The creep deformation is a significant aspect of the mechanical performance of polymer-based materials. It has been reported that if a resin-based material has high creep strain, this will adversely affect its resistance to mechanical stress and thus influence the long-term clinical durability of the restorations [114]. Therefore, this study was conducted to evaluate the creep deformation of some newly introduced resin-composites.

An inherent mechanical problem of polymeric filled materials such as composite resins is their viscoelasticity, which reduces their capacity to resist deformational change under load, especially under conditions of moisture from saliva and fluctuating temperatures. Thus, creep is defined as the time-dependent plastic deformation, or strain, of a material under a static load well below its conventional elastic limit [95]. The response to load application is dependent on the composite's filler content, matrix chemistry, and degree of conversion. Strain capacity is inversely related to inorganic filler content. Considering the relatively narrow range of volumetric shrinkage values displayed by hybrids and microfilled composites with regular consistency, contraction stress onset and its final value are directly affected by the composite's inorganic content [95].

The results of creep test in this study showed that all materials exhibited classic creep. For all matrix structures, the principal creep parameters were no significantly different for each solvent condition. All materials showed slight higher creep-strain in water than in ethanol. These results were in agreement with Watts D.C. and Marghalani H.Y (2007) [96].

Filler content plays an important role in the creep resistance of composite resins as silorane-based composite possesses the lowest filler content when compared to both methacrylate based materials, the reason for its same creep resistance should be attributed to other factors possibly to its monomer composition [119] or filler coupling.

Siloranes were developed as monomers with four polymerisable cycloaliphatic oxirane moieties [15], giving a higher cross-link density and thus a better creep resistance [99].

This finding is in inconsistency with the study of Ilie and Hickel (2009) [63] revealed that Except for the silorane-based composite, all methacrylatebased materials showed a statistical significant decrease in creep by storing in alcohol solution. This difference may be due to the difference in the methodology because they measure creep by (The indentation modulus was calculated from the slope of the tangent of indentation depth-curve at maximum force. By measuring the change in indentation depth for 5 s with a constant test force of 500mN, a relative change in the indentation depth was calculated. This is a value for the creep of the materials).

The results of the present study are in disagreement with the study of S. El-Safty *et al.,* (2012) [11] who found that Creep deformation of all studied resin-composites increased with wet storage. This difference may be due to the difference in the methodology because they compare between dry and wet aging for 24h.

The absorption of moisture by the resin-composites leads to the degradation of both strength and stiffness of composites [121]. The effect of water on creep behavior in terms of plasticization is evident in the literature. The presence of water and other fluids has the potential to induce swelling and peeling stress in the structure, in addition to a plasticizing effect on the polymer matrix as well as debonding of the filler from the matrix, all of which can lead to increased creep formation [119].

CONCLUSIONS

Within the limitations of the current in vitro study, the following were concluded:

1. The flexural strength and hardness of the silorane based and methacrylate based composite were significantly influenced by saliva or ethanol.
2. The Young's Modulus of the silorane based and methacrylate based composite were not significantly influenced by saliva and significantly influenced by ethanol.
3. The creep silorane based and methacrylate based composite were not significantly influenced by saliva or ethanol.

RECOMMENDATIONS

Clinical Recommendations

In dentistry there is no one biomaterial that has the same physical, mechanical and optical properties as tooth structure (i.e., dentin, enamel, cementum) and possesses the physiological characteristics of intact teeth in function. So we recomend using materials that result in a mimicked biological effect.

- A biomimetic material should match the part of the tooth that it's replacing in several important ways, including same physical, mechanical ,optical properties and function of the respective areas (e.g., pulp, dentin, enamel, dentoenamel junction)

Suggestions for Future Research

More studies should be conducted to evaluate:

- Other properties of these materials
- Effect of other storage media (e.g., citric acid).
- Effect of Extending the period of storage 3-6 months

To have full understanding about the reliability of these materials as being capable of producing acceptable restorations.

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