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Effect of immersion in different media on the mechanical properties of dental composite resins

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Abstract
This article was undertaken to investigate the effect of various media and storage time on the mechanical properties of dental composites resins. Three dental composite resins Competence universal, Spectrum and Bright light were tested. Forty-eight Specimens of each composites were prepared for flexural strength and Forty-eight Specimens of each composites were prepared for diametral tensile strength. The specimens were storage in Different solutions: distilled water, artificial saliva, ethanol/water and mouthwash at 37°C, after that; flexural strength (FS) and diametral tensile strength (DTS) tests were measured on 24 hours, 15, 30, 60 and 90 days. The time immersion and the solutions have effect on the flexural strength and modulus and on DTS, all composites exhibited lowest mechanical properties at 90 days. Composite spectrum has highest mechanical properties, the flexural strength ranged from 99.1 to 44.84 MPa when composites immersion in ethanol/water. While composite Bright light recorded lowest properties.

Keywords: dental composites, mechanical properties, flexural strength, diametral tensile strength, Ethanol, Chlorhexidine

1. Introduction
Dental composites restoration is used for restoring damage teeth or to fix orthodontic appliances inside the oral cavity, the success of any dental composites restoration depends completely on its physical, chemical and mechanical properties. The durability and long life of dental composite resins are substantially influenced by the characteristics of the oral environment [1, 2] and the presence of degradation in composite restoration.
In the oral cavity, many factors that can causes to the degradation of dental composites restoration and occurs in areas that are unexposed to abrasion and compression, chemical degradation must be present [3]. Water, saliva [4, 5] drinks and food [6], these factors can cause changes in the physical and mechanical properties of the dental composite restoration [7-9]. other factors degradation of composites such as pH of immersed solution [10], liquid environment and temperature can affect the physical properties [11]. However mechanical properties of the composites restoration decreases by water because it degrades the silane interface of the composite resins [12]. Also alcohol is a good dimethacrylate solvent which most composites content [7] alcohol can soften the matrix of composite by increased the amount of unreacted monomers and oligomers that diffuse out of the material [13, 14]. In other hand when Composite were decreased the dental properties after immersion in the mouthwashes [15, 16].
Previous studies have shown that the mixtures of ethanol and water lead to increased composite wear [17], and the study on mouth rinse with an alcohol and a low pH, decrease in hardness of resin composite [7, 18], increase the sorption and solubility of resin composites [1, 19]. Material such as dental composites restorative which under stress in oral cavity are exposed to tension and compression forces are evaluated by measure of properties of flexural strength, flexural modulus [20, 21]. The diametral tensile strength are providing information about the behavior of brittle dental composites, It is therefore a clinically relevant factor since composites would be expected to fail under tensile stresses during mastication because of the forces they are subjected to in functional areas [22, 23]. Solution media were chosen as the food simulating, distilled water simulates the wet oral environment provided by saliva and water, the ethanol solution simulates certain beverages, including alcohol, vegetables, fruits, candies and syrups.

The purpose of this article to evaluate the effect of solution media (pH 4.0 – 7.4) storage times on the mechanical properties of different dental composite.

2. Materials and Methods

2.1. Dental composites and Immersion media

To conduct this article, three different dental composites was used which described in Table_1. All The materials were base Bis_GMA matrix composition and different filler type.

Four different solution were used in the study: 1. Distill water (DW), 2. Artificial saliva (AS) had an electrolyte composition similar to that human saliva. It was composed of (carboxymethyl cellulose, Sodium chloride NaCl 125.64, Potassium chloride KCl 963.9, Potassium thiocyanate KSCN 189.2, Potassium Dihydrogen orthophosphate KH_2PO_4 654.5, Urea $CO(NH_2)_2$ 200, Calcium chloride dehydrate $CaCl_2$. $2H_2O$ 227.8, Sodium sulphate $Na_2SO_4.10H_2O$ 763.2, Sodium Hydrogen Carbonate $NaHCO_3$ 630.8 and Ammonium chloride NH_4Cl 178 mg/L) [24]. 3. Ethanol/Water (EW) Solution 40% ethanol, and 4 mouth wishes (MW) Chlorhexidine 0.2% Phliadephia Pharmaceuticals / Jordon. Distill water was used as the control. The PH measured with a PH meater (TRANS BP 3001) the PH value of each solution is show in Table 2.

2.2 Preparation of Specimens

48 specimens were fabricated for each type of composites in a stainless steel mold for each test (FS, DTS), Flexural strength test specimens (25 mm × 2 mm × 2 mm) were fabricated according ISO 4049, and cylindrical specimens 2.5mm in diameter and 6mm in height were prepared to DTS test, both held between two glass slabs, all specimens were polymerized by curing light unit with a light emitting diode LED source (Woodpecker china) using an irradiance of intensity was 600 W/cm^2 with time 40 seconds of exposure to top and the distance between curing light and specimen 1 mm, after polymerization was completed the specimens were extracted from the molds and ground wet with silicon carbide paper 1200-grit. Specimen's dimensions were measured with a digital caliper (INSIZE China) of 0.001 sensitivity. The specimens were randomly divided into four test groups.

All the specimens were stored in distilled water at 37 ± 1°C for 24 hours before test. Then, the specimens were immersed in 250 ml of each four solutions. The different storage times (1, 30, 60 and 90 days) remained under constant tempter at 37°C.

Table 1: Main composition of composite resin tested in this study.

Composite	Code	Composition	Filler content Weight% (Vol %)	Manufacturer
Competence universal	CU	Bis_GMA*, TEGDMA**,inorganic filler parties of (0.02-1.5 µm)	76% (57%)	Willmann & Pein GmbH Hamburg, Germany
Bright light	BL	Bis_GMA, TEGDMA, inorganic filler parties of (0.05-1.5 µm)	80% (65%)	DMF LTD, E. U.
Spectrum	SP	Bis_GMA, Bis_EMA*** TEGDMA, Ba-Al-borosilicate glass/colloidal silica	76% (57%)	DENSPLY, Konstanz, Germany

*Bis_GMA: Bisphenol A Diglycidyl Methacrylate, **TEGDMA: TriethyleneGlycol Dimethacrylate, ***Bis_EMA: Ethoxylated Bisphenol A Glycol Dimethacrylate.

Table 2: immersion media used in the study.

Immersion media	pH
Distilled water (DW)	7.2
Artificial Saliva (AS)	6.92
Ethanol/water (EW)	7.99
Chlorhexidine (MW)	5.25

2.3 Flexural strength testing and flexural modulus

Flexural strength and flexural modulus were measured according to 2049:2008. The flexural strength testing and flexural modulus were measured by using a 3-point bending testing device in universal testing machine (Zwick/roell BT1-FR2.5TN Germany) at a cross-head speed of 1 mm/min. Specimens were loaded until fracture occurred. The flexural strength was calculated as [25]

$$Fs = \frac{3PL}{2bd^2}$$

where FS flexural strength (MPa), *P* load at fracture (N), *L* distance between the supporting wedges (mm), *b* width of the specimen (mm), and *d* thickness of the specimen (mm). Flexural modulus E (MPa) was calculated using the following formula:

$$E = \frac{3PL^3}{4bd^3D}$$

P load at fracture (N), *L* distance between the supporting wedges (mm), *b* width of the specimen (mm), *d* thickness of the specimen (mm) and *D* the deformation of the specimen at P, also flexural modulus was determined from the slope of initial part of stress-strain carve.

2.4 Diametral tensile strength test (DTS)

Diametral tensile strength were performed using a universal testing machine (Zwick/roell BT1-FR2.5TN Germany). The disk specimens were loaded until fracture at a crosshead speed of 1mm/min. It was being compressed between the two supporting plates of the machine. The formula used to diametral tensile strength was:

$$DTS = \frac{2P}{\pi DT}$$

Where *P* load at fracture (N), *D* diameter (mm) and *T* thickness (mm) of specimens.

3. Result

The results of flexural strength testing (FS) are summarized in Table 3 and Figure 1-A, B, and C, flexural modulus Table 4 and Diametral tensile strength test (DTS) Table 5. There is a slight decrease in flexural strength test of composites when immersed in distill water, a major decrease founded in composites test when immersed in ethanol/water, Its ranged from 91.51±3.05 to 33.55±2.8, 72.561±4.26 to 28.2±5.9 and 68.74 to 99.106±2.83 to 44.84±5.01 MPa at one day to 90days for CU, BL and SP respectively. Artificial saliva and moth

wishes (Chlorhexidine) media presented same range of flexural strength, ranking from lowest to highest was as follows BL<CU<SP for 90days. Figure 2-A, B, and C show flexural modulus proportional with time and breakdown the composites when immersion in ethanol/water media, for BL composites decreases from 13.509±1.24 to 2.9±1.18GPa.

There is slight decrease on composites when immersed in distill water, artificial saliva and moth wishes (Chlorhexidine) media presented same value at 90days for Diametral tensile strength test (DTS) Figure 3_A, B and C, ranking from lowest to highest value DW<MW<AS media. There is downfall in the DTS of composites when immersed in ethanol/water, Its decreased 40.92±2.33 to 18.46±4.57, 38.25±1.43 to16.92±5.36 and 43.815±3.07 to 26.28±2.9 MPa at 90 days for CU, BL and SP composites respectively.

Table 3: Mean (standard deviation) of flexural strength tested (MPa) of dental composites testing after immersion in different media.

Immersion media	Time (Day)	Dental composites test (n=3)		
		Competence universal	Bright Light	Spectrum
Distill water (DW)	1	91.513 (3.05)	72.561 (4.26)	99.106 (2.83)
	15	90.921 (0.9)	70.012 (2.23)	94.823 (3.21)
	30	90.770 (1.72)	63.223 (4.97)	93.264 (3.22)
	60	88.385 (1.58)	56.39 (1.32)	88.385 (1.58)
	90	85.95 (2.4)	52.019 (3.51)	85.956 (2.4)
Artificial Saliva (AS)	1	91.513 (3.05)	72.561 (4.26)	99.106 (2.83)
	15	88.602 (1.2)	64.201 (1.91)	94.401 (2.6)
	30	86.824 (6.67)	59.96 (3.44)	87.187 (2.97)
	60	66.14 (3.03)	50.003 (1.6)	82.187 (2.4)
	90	59.362 (4.39)	45.301 (2.47)	74.072 (4.2)
Ethanol/water (EW)	1	91.513 (3.05)	72.561 (4.26)	99.106 (2.83)
	15	82.335 (1.203)	60.368 (2.33)	89.523 (1.72)
	30	53.008 (3.54)	40.591 (3.12)	63.331 (2.6)
	60	36.112 (2.19)	33.15 (3.71)	50.914 (2.16)
	90	33.554 (2.8)	28.277 (5.9)	44.845 (5.01)
Chlorhexidine (MW)	1	91.513 (3.05)	72.561 (4.26)	99.106 (2.83)
	15	88.251 (1.66)	64.09 (4.82)	93.771 (4.82)
	30	63.701 (2.23)	59.663 (3.56)	90.22 (1.76)
	60	52.497 (12.7)	53.872 (4.72)	83.977 (3.92)
	90	55.857 (6.65)	47.022 (5.68)	71.636 (4.38)

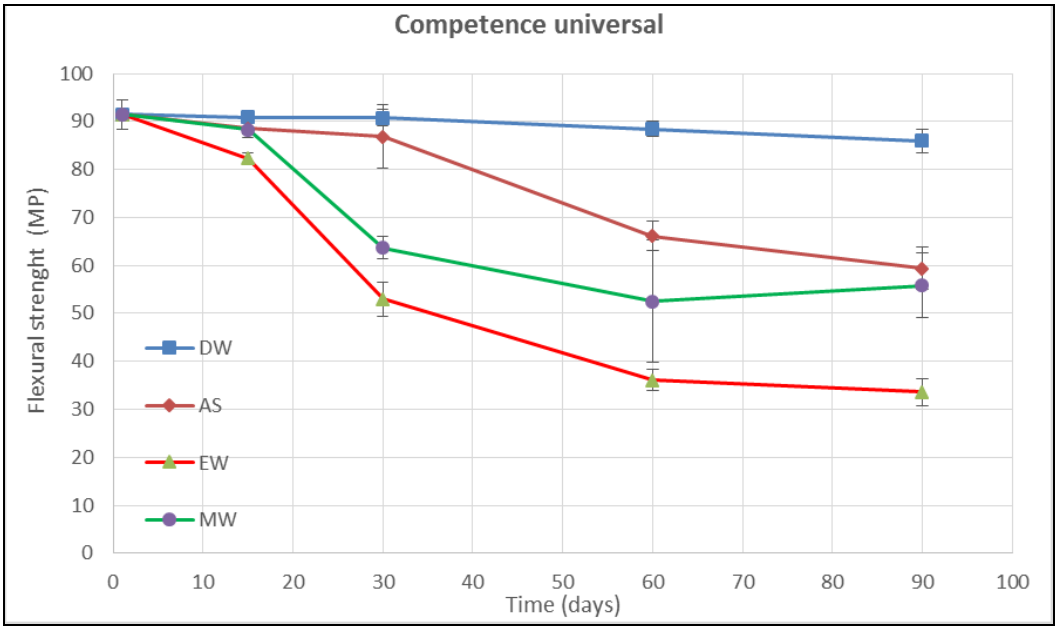

Fig 1A: Mean flexural strength of the Competence universal composites.

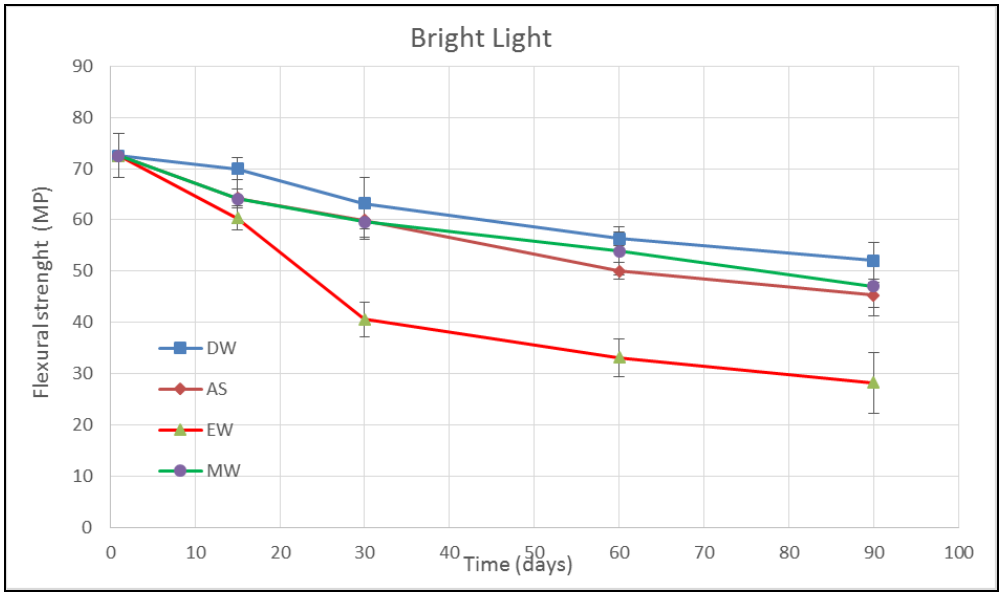

Fig 1B: Mean flexural strength of the Bright Light composites.

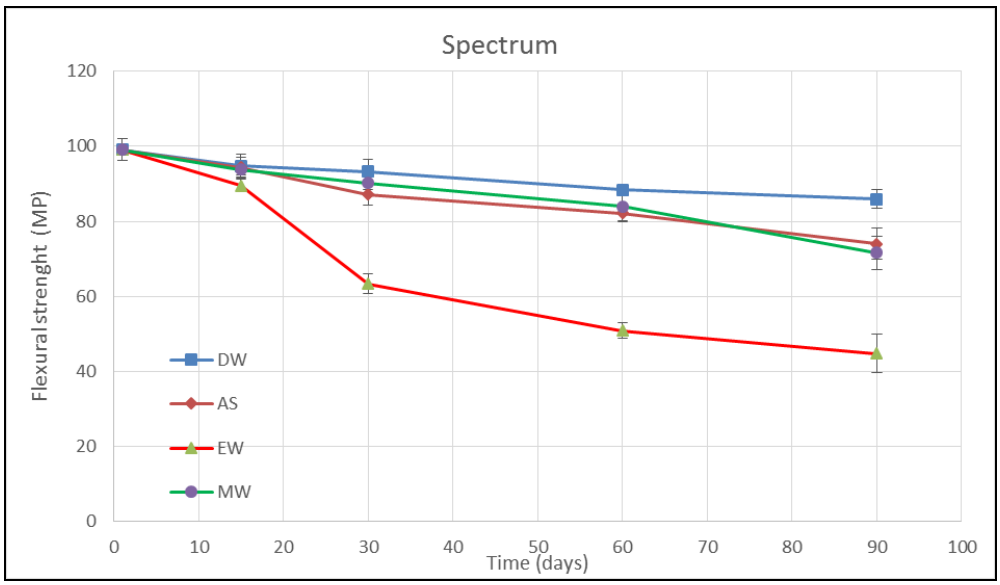

Fig 1C: Mean flexural strength of the Spectrum composites.

Table 4: Mean (standard deviation) of flexural modulus (GPa) of dental composites testing after immersion in different media.

Immersion media	Time (Day)	Dental composites test (n=3)		
		Competence universal	Bright Light	Spectrum
Distill water (DW)	1	16.872	13.509	18.634
	15	15.665	12.465	17.076
	30	14.305	10.562	16.755
	60	13.238	9.856	15.905
	90	12.922	8.308	14.153
Artificial Saliva (AS)	1	16.872	13.509	18.634
	15	15.721	11.536	16.937
	30	14.653	10.838	15.496
	60	12.016	8.308	14.465
	90	11.789	7.434	13.237
Ethanol/water (EW)	1	16.872	13.509	18.634
	15	13.837	8.621	15.721
	30	10.793	5.836	10.426
	60	7.741	3.156	8.73
	90	4.029	2.901	5.092
Chlorhexidine (MW)	1	16.872	13.509	18.634
	15	15.668	11.634	16.924
	30	13.837	10.119	15.721
	60	11.939	10.093	13.509
	90	10.793	9.858	12.167

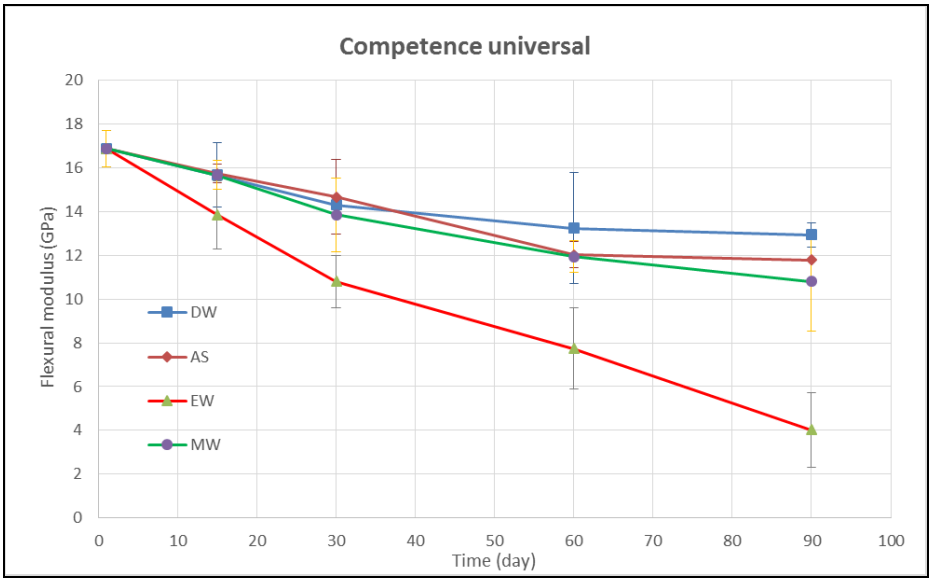

Fig 2A: Mean flexural modulus of the Competence universal composites.

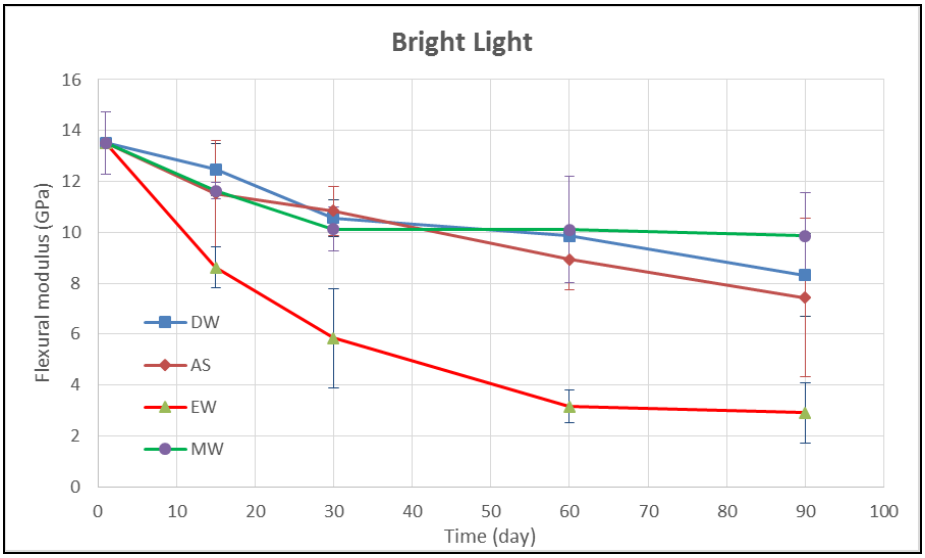

Fig 2B: Mean flexural modulus of the Bright Light composites.

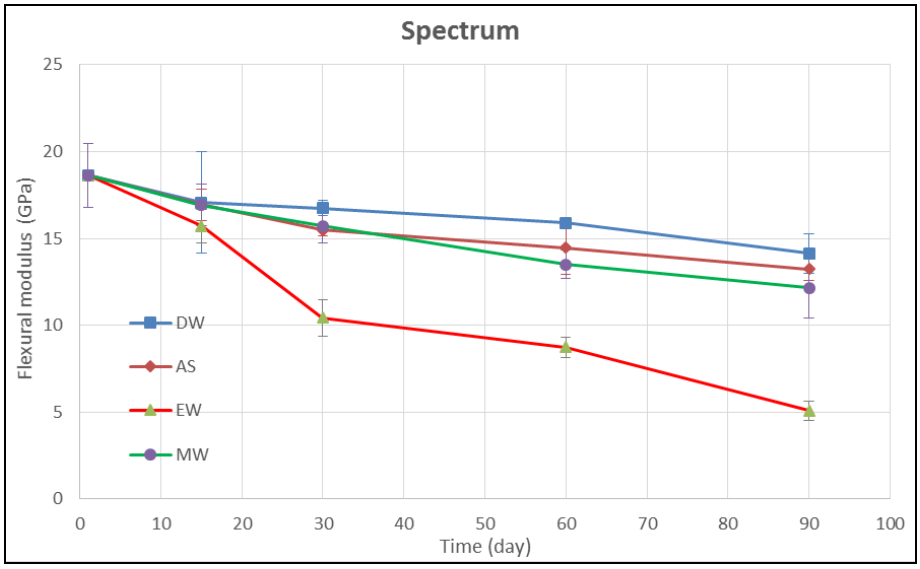

Fig 2C: Mean flexural modulus of the Spectrum composites.

Table 5: Mean (standard deviation) of diametral tensile strength (MPa) of dental composites testing after immersion in different media.

Immersion media	Time (Day)	Dental composites test (n=3)		
		Competence universal	Bright Light	Spectrum
Distill water (DW)	1	40.924 (2.33)	38.25 (1.43)	43.815 (3.07)
	15	38.362 (1.22)	35.061 (4.02)	41.64 (2.41)
	30	32.243 (2.67)	29.963 (2.67)	36.152 (3.99)
	60	25.971 (6.4)	24.726 (4.54)	35.091 (4.33)
	90	24.2617 (9.0)	22.983 (5.96)	34.300 (2.99)
Artificial Saliva (AS)	1	40.924 (2.33)	38.25 (1.43)	43.8152 (3.07)
	15	38.631 (3.81)	34.336 (2.66)	40.73 (1.95)
	30	34.462 (1.97)	29.62 (4.18)	38.973 (1.41)
	60	32.07 (1.2)	27.667 (3.74)	37.084 (2.79)
	90	25.004 (3.00)	23.522 (3.28)	35.673 (4.4)
Ethanol/water (EW)	1	40.924 (2.33)	38.25 (1.43)	43.815 (3.07)
	15	36.014 (4.21)	33.116 (3.97)	39.011 (3.15)
	30	33.417 (3.71)	28.689 (4.57)	35.331 (8.3)
	60	20.421 (3.67)	19.926 (3.78)	28.117 (2.25)
	90	18.461 (4.57)	19.922 (5.36)	26.2847 (2.9)
Chlorhexidine (MW)	1	40.924 (2.33)	38.25 (1.43)	43.815 (3.07)
	15	37.941 (1.7)	34.012 (2.21)	41.351 (2.44)
	30	33.290 (2.75)	30.633 (4.62)	40.591 (5.43)
	60	28.614 (3.3)	26.793 (1.51)	38.554 (1.91)
	90	24.622 (2.03)	24.632 (2.82)	33.3185 (5.16)

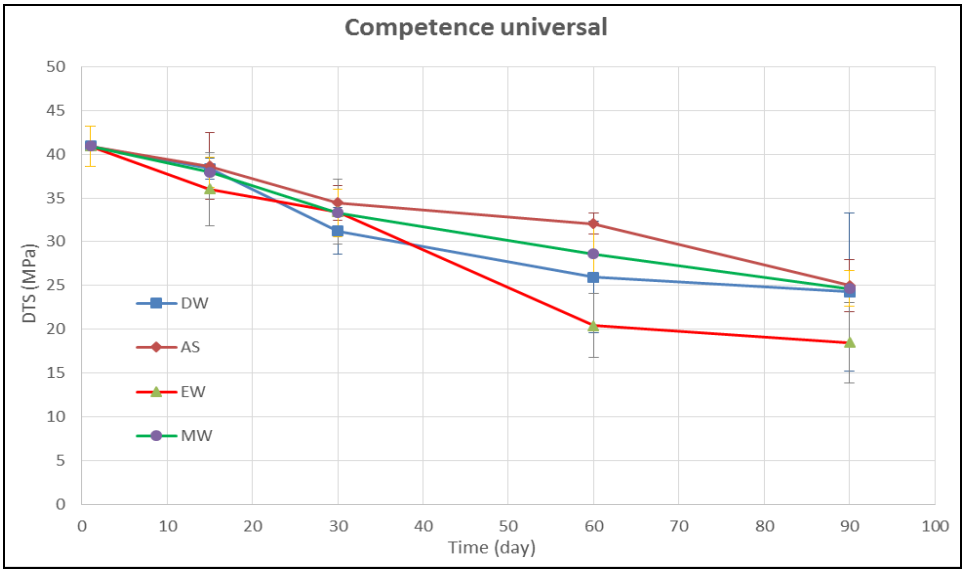

Fig 3A: Mean DTS of the Competence universal composites.

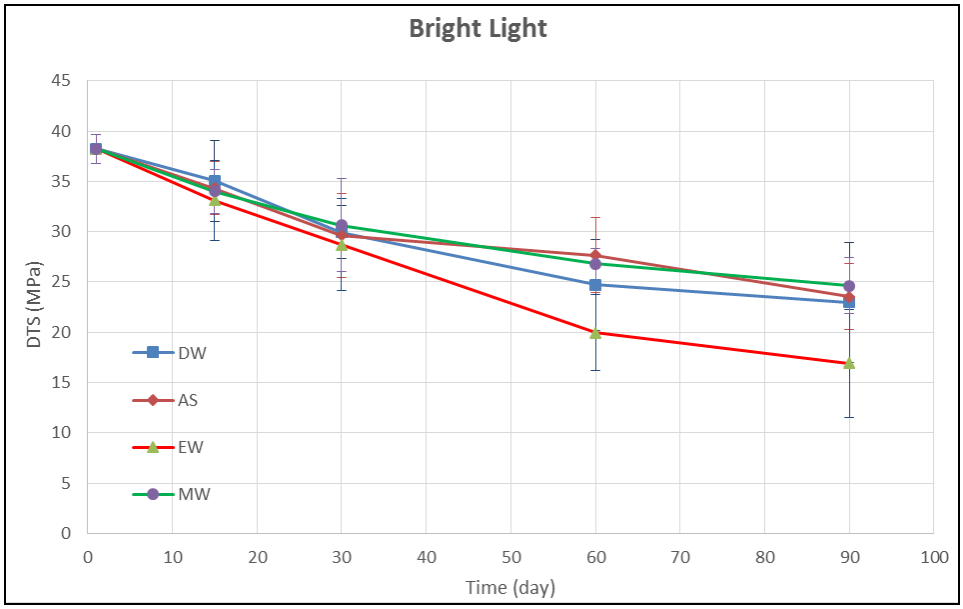

Fig 3B: Mean DTS of the Bright Light composites.

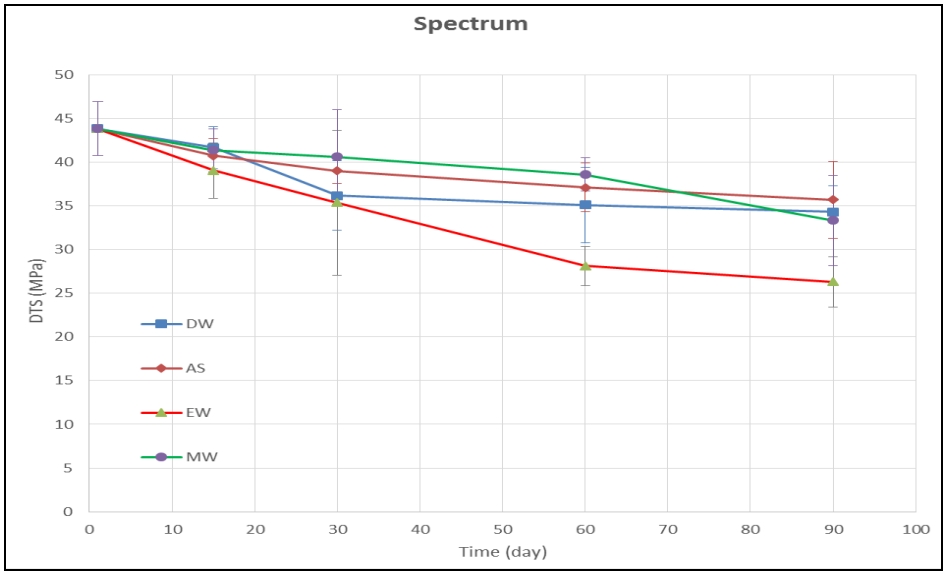

Fig 3C: Mean DTS of the Spectrum composites.

4. Discussion

A good restorative material should satisfy demand for products with good mechanical properties to replace natural tooth tissue. In oral environment, dental composites are exposed in interrupted form or continuously to chemical agents found in saliva, food and drinks may be cause to the chemical degradation and loss many of dental properties. The degradation resistance of dental composites in the oral environment plays an important role in long life of composites. In current study specifically evaluated the effect of media oral cavity on the FS and DTS of three composites with those of a hybrid composite and a microfill composite and compared between this composites, results were listed to a 90-day period. Three dental composite restorative were selected for this study, all test composites were kept in different solution for 90 days, the CU composites stay constant (small change) in flexural strength while it's decreased of other composite test, and SP had highest flexural strength while BR had the lowest flexural strength. Also for flexural modulus BR recorded lower value for all time, while SP presented highest modulus. The slight decreases in flexural strength and diametral tensile strength of composites after immersion in water can be explained by respect of the filler-matrix interaction, because water destroys some of the filler-matrix bonds.

The result shows the intensive effect of Ethanol/water 40% on the mechanical properties of composites comparing to distill water, artificial saliva, and mothwishes (Chlorhexidine), this came from organophilic nature, which may produce probable degradation of matrix and disintegration of the bond between the filler and the silane seems to occur after 30days storage, which was continued up to 90 days.

The flexural strength measured in this study were within the values reported in previous studies, Rodrigues *el al.* [26] study flexural strength of two composite resins and one compomer for 30 days, Nuran *et al.* [21] and Balkenhol *et al.* [27]. The diametral tensile strength DTS are used to understand the behavior of brittle materials, DTS (MPa) measured in this study are similar to previous studies recorded as Rehman *et al.* [23], Aguiar *et al.* [28] and Christine Schmidt & Nicoleta Ilie [29]. Study at least confirms the water, artificial saliva and mouthwishes (Chlorhexidine) had the same effect on mechanical properties after 90days storage, also the possible damage by ethanol, a possibly damaging factor of which the public should be aware.

5. Conclusions

Under the conditions of this study the following conclusions were drawn:

1. The effect of immersion media on flexural strength, modulus and Diametral tensile strength of dental composites were dependent.
2. A marked reduction in the Flexural strength and modulus of the composites has been showed when its immersed in ethanol/water media. Also he results showed a small decrease in the mechanical properties when the composites are immersed in artificial saliva and chlorhexidine comparing to distilled water.
3. Continuity and the clinical durableness of dental composites may be compromised due to changes in mechanical properties with immersion media.

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