

RESEARCH PAPER

Flexural Properties of New Nanocomposites for Dental Restorative that Based on (Bis-GMA) Material and Polymerized by Light Curing

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ABSTRACT

Six types of new nanocomposites materials were synthesizing as dental composites, that based on the different sets of unsaturated monomers (where they know by groups name of (A, B, C, D, E, and E)) in addition to 2, 2 propyl bisphenol glycidyl dimethacrylate (Bis-GMA) and inorganic Nanofillers [SiO₂, ZrO₂, HA, and Al₂O₃] which adding individually, were characterized for the reason of assessed their promising applications as restorative materials in the dentistry field. The flexural strength and flexural modulus of the prepared dental nanocomposites were examined and the results were widely discussed and connected to the composition materials of these composites. The results show that the composites with specific monomers (BIS-GMA, meth acrylamide, methacrylic acid, and 1-6 hexanediol methacrylate) for the group (D) that reinforced with silica nanoparticle have elevated flexural properties as compared with other types of the prepared nanocomposite, where these values reached to (144 MPa and 5.4 GPa) for flexural strength and flexural modulus respectively.

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INTRODUCTION

Last 45 years, the field of used dental restorative composite materials has gradually improved year by year and have more advanced due to the high demand by dental patients for tooth coloured and restoration.

Resin-based composites have broadly used in the medicine field especially in dentistry due to their excellent properties such as mechanical, biocompatibility, and aesthetic, characteristics as well as high coherence possibility with tooth structure, suitable handling and reasonable cost, etc. [1].

Resin-based composites that used in dental applications are mostly composing of four essential components polymer matrix, filler particles, coupling agent, and initiator- accelerator system [2].

The polymer matrix is an organic constituent consists of monomers that usually are polymerized by visible light activation, whilst fillers are an inorganic component, typically used for increasing the physical and mechanical properties of the resin matrix [3].

In the current time, in spite of the important development of resin-based composites, the

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restorative composite reveals more limitations causing actual impair in their permanence and service life that due to unsatisfactory mechanical strength and high polymerization shrinkage [4].

Mechanical properties of resin-based composites are basically dependent upon their microstructure, composition, and the filler particles distribution in the composite [5].

A large number of authors have reported a considerable connection between the mechanical properties of composites and the use of different types of monomers as resin base and select the more appropriate fillers in these composites to reach more sufficient mechanical properties.

In 2018, R A C Razali¹, et.al they study the influence of different weight fraction (0, 5, 15, 20 wt%) of natural hydroxyapatite (NHAp) and silica as inorganic fillers in dental resin (50%bisphenol A glycidyl methacrylate (BisGMA)/ 25%triethylene glycol dimethacrylate (TEGDMA)/ 25%hydroxyethyl methacrylate (HEMA)), by added silica to NHAp and mixed with organic matrix resin. The results can be signified that the mechanical properties depend on the weight fraction of HAp and silica in composite and the best composition (85 wt% of HAp and 15 wt% of silicate) acquired the highest values of the flexural and compressive strength [6].

M. S. Al Ajely et.al in 2018, prepare a new dental composite from Bis GMA/TEGDMA in a composition ratio of 70/30 as a matrix resin and adding different ratios of calcium fluoroaluminosilicate glass filler. The static flexural strength and modulus were measured by a three-point bending test that was done according to the ISO-4049 specification. The result showed the highest flexural strength 90.35 MPa, and flexural modulus (11.29 GPa) have been obtained [7].

In 2019 Sukriti Yadav & Swati Gangwar, prepared dental resin composites by photo-curable and with varying ratios of functionalized nHAPs (0, 4, 8, and 12%wt.). The matrixes of this composite were bisphenol A-glycidyl methacrylate (Bis-GMA) used as a base monomer with Tri-ethylene glycol dimethacrylate (TEGDMA) and 2-hydroxyethyl-methacrylate (HEMA) were as a diluents monomer. The results show that hardness and compressive strength were increasing with an increase in the filler particles, while the flexural strength of the composite decreases with increases in the amount of filler, although this value is still higher than that of a satisfactory limit of flexural strength in

dentistry application (i.e. 74.2MPa) [8].

Another group at the same year studies the effect of filler size on the aggregation of these fillers, subsequently, their effect on the mechanical properties of dental resin composites that calculated at different filler loadings (20 wt% up to 76.5 wt %.) of silica particles (SiNP) has 80 nm nano size, and amorphous barium-alumina borosilicate (BaAlBoSi) microparticles (1.0 μm), added to resin matrix composed of triethylene glycoldimethacrylate (TEGDMA), urethane dimethylacrylate (UDMA), bisphenol A polyethylene glycol diether dimethacrylate (BisEMA), and bisphenol A glycidyl methacrylate (BisGMA) at the weight ratio of (0.3:0.7:1:1), respectively. The paper concluded that at the lowest filler concentration (20 wt %) of Si nanoparticles, the flexural strengths were the highest, and at (BaAlBoSi) fillers were lower. The reduction in flexural strength of (BaAlBoSi) composites is associated with the increase in the size of fillers particle caused an excellent distribution of particle size [9].

The aim of the presented study is producing new types of dental composite materials, by using numerous new monomers together with Bis-GMA (such as methacrylamide, 2-ethylhexylmethacrylate, methacrylic acid, and methacrylate), which were reinforced with four types of inorganic nanoparticles (ZrO_2 , SiO_2 , HA, and Al_2O_3) individually. The characterizing of these composites was done by examining the flexural properties and study the effect of chemical composition of constituents of composites materials on the flexural properties, with comparing the results of all prepared composites to select the more suitable composites in dental applications.

MATERIALS AND METHODS

In this research six types of matrices were prepared, are listed in Table 1 in six groups (A, B, C, D, E, and F) each matrix was prepared from different monomers, where 2,2 propyl bisphenol glycidyl dimethacrylate (Bis-GMA) is the base material in all matrices, with other different monomers each has its function. As well, four types of inorganic nanoparticles (ZrO_2 , SiO_2 , HA and Al_2O_3) were used as nano-fillers for various purposes, each type of nanoparticles has different particle sizes are ((30-40), (10-20), 25, and (20-40) nm) respectively. All chemical materials were supplied by Sigma

Table 1. Chemical Composition of the Matrices Resins of Prepared Composites.

Groups	Monomers	Adding Ratio	Monomers Mixing Time
A	1. BIS-GMA	40%	50min
	2. 2-ethylhexylmethacrylate	20%	
	3. methylmethacrylate	20%	
	4. 1-6 hexanediol methacrylate	20%	
B	1. BIS-GMA	40%	50min
	2. methacrylic acid	20%	
	3. methacrylate	20%	
	4.1-6hexanediol methacrylate	20%	
C	1. BIS-GMA	40%	70min
	2. methacrylic acid	20%	
	3. polyethylene glycol 3500	20%	
	4.1-6hexanediol methacrylate	20%	
D	1. BIS-GMA	40%	90min
	2. methacrylamide	20%	
	3. methacrylic acid	20%	
	4. 1-6 hexanediol methacrylate	20%	
E	1. BIS-GMA	40%	70min
	2. 2-ethylhexylmethacrylate	20%	
	3. polyethylglycol	10%	
	4. .bisphenol adimethacrylate	10%	
	5. 1-6 hexanediol methacrylate	20%	
F	1. BIS-GMA	40%	70min
	2. methacryate	20%	
	3. polyethylene glycol	20%	
	4. 1-6 hexanediol methacrylate	20%	

Aldrich/USA and MERCK/Germany companies.

Nanocomposite Preparation

Each group described in Table (1) was mixed for approximately 90 minutes with the present of

N_2 gas for removing dissolved oxygen, after that the adding 0.4gm of zinc oxide as antimicrobial agent. Initially, one type of nano-fillers (HA) was added at different weight fractions (1, 2, 3, and 4%wt) to two groups of matrix monomers (D and

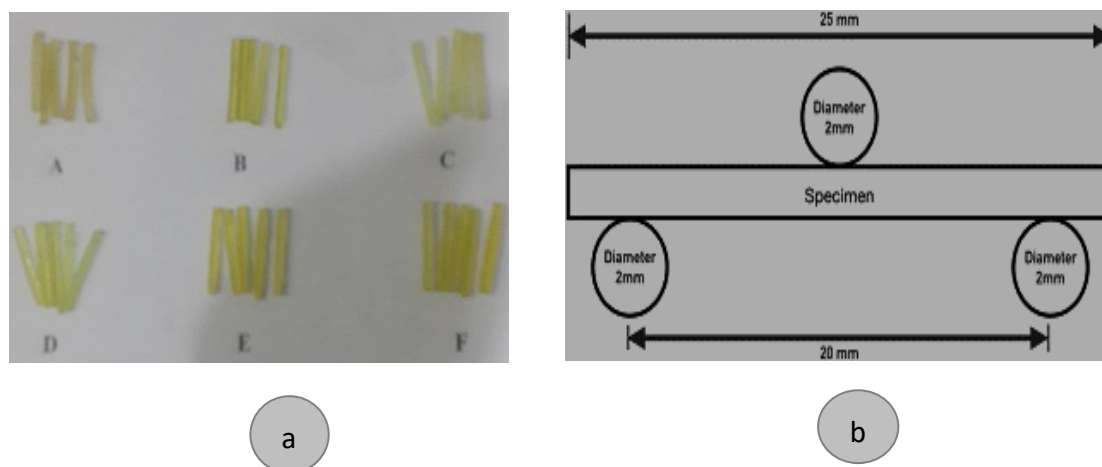


Fig. 1. Flexural test where (a) test specimens and (b) schematic illustration of the flexural strength test.

H) to choose the best percentage of addition. Then added nano-fillers (ZrO_2 , SiO_2 , HA, and Al_2O_3) individually to each group of matrix monomers mentioned earlier in Table 1 and at a constant ratio of weight fraction (2%wt). Also 0.5wt. % ratio of DMAEMA as accelerator and 0.5wt % of camphor quinone as initiator has put into the mixture and continue mixing for 20 min which considered as the last stage of mixing to producing the composite materials.

Then all the specimens were cured with light by using a light-curing unit of type (EliparFreelight2LED, 3 M ESPE) at 1500 mW/cm² intensity. The samples were exposed to irradiation in different positions for 60 s. After polymerization and prior to performing the flexural strength (FS) test, the samples were stocked up in distilled water at 37°C for 48h and 100% humidity.

The Flexural Test

The flexural strength test was performed according to ISO 4049:2019 by the three-point bending test [10]. Five specimens of each composite type were prepared with dimensioned (2mm × 2mm × 25mm) as shown in Fig. 1.

The samples were fitted between two supports with a 20 mm span between them the speed of the test was 0.75 mm/min until fracture has occurred. The flexural strength (MPa) was determined using Equation (1); [11]

$$\text{Flexural strength (MPa) for three - point bend test} = \sigma_{\text{bend}} = \frac{3FL}{2wh^2}$$

Where F is the max of applied load (N), is the span between the supports (20 mm), w is the width (2 mm), and h is the thickness (2 mm).

The flexural modulus (E_f), is calculated in the elastic region was determined from Equation 2:

$$\text{Flexural modulus} = E_f = \frac{FL^3}{4\delta wh^3} = \frac{mL^3}{4wh^3} \quad (2)$$

Where δ is the deflection of the beam when a force F is applied, m is the slope of the load (F) / deflection curve.

RESULTS AND DISCUSSION

The flexural properties of polymeric composites are dependent on the type of polymeric materials, fillers type, fillers ratio, distribution of fillers, and bonding of the fillers inside the composite material. To select the more suitable material from the prepared matrices to use as a matrix for dental composites, the prepared matrices were subjected to the flexural test, Figs. 2 and 3 show the values of flexural strength and flexural modulus, respectively, as a function of matrix type (A, B, C, D, E, and F). From these figures, it was observed difference values in the flexural strength and flexural modulus depending on the components of the copolymers prepared as matrices materials for dental composites. In addition, it was noticed that the highest values of the bending properties were acquired for the sample that has the matrix prepared from monomers group D which reach to 121MPa and 4.38GPa, respectively. While the lowest magnitudes of flexural strength and flexural modulus were for the two samples prepared from monomers of groups A and E which reached their values to 87MPa and 3.43GPa, respectively. These results are connected to the chemical composition of these matrices and the nature of chemical structures of copolymers chains and the type of

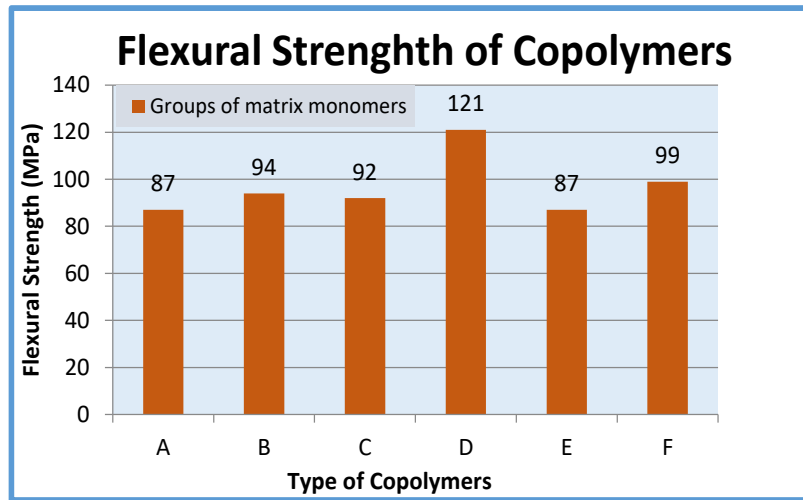


Fig. 2. Flexural strength of the prepared copolymers (matrices) as a function of monomers groups (A, B, C, D, E and F).

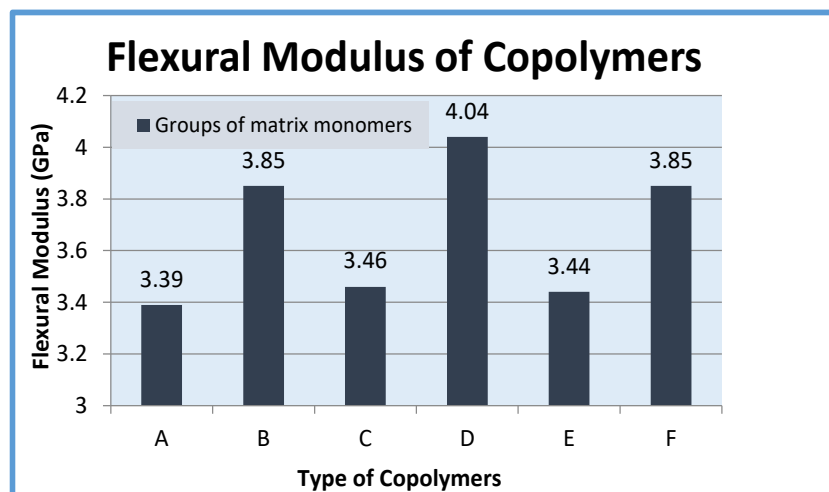


Fig. 3. Flexural modulus of the prepared copolymers (matrices) as a function of monomers groups (A, B, C, D, E and F).

chemically bonding that connect the monomers between them.

Based on the results obtained from Figs. 2 and 3, two samples of matrix materials were selected, one with the highest values in bending properties and the other having the lowest values, these samples were represented by two groups of matrix monomers D and E, respectively. These materials were reinforced with hydroxyapatite nanoparticles, to prepare nanocomposites for dental materials.

Effect of nanofillers content

The effect of adding different amounts of

nano-hydroxyapatite (0, 1, 2, 3, and 4) wt.% with a constant amount of zinc oxide (0.4gm) as an antimicrobial agent on the flexural strength and flexural modulus of the dental restorative hybrid nanocomposites materials, was shown in Figs. 4 and 5, respectively.

There is a primary increase in the flexural strength and flexural modulus of the hybrid nanocomposites when adding 1% ratio of nano-powder hydroxyapatite were the values of flexural strength and flexural modulus increased from 121 to 133MPa and from 4.04 to 4.67GPa respectively, for the matrix of monomers group D and these properties increased from 87 to 103MPa and from

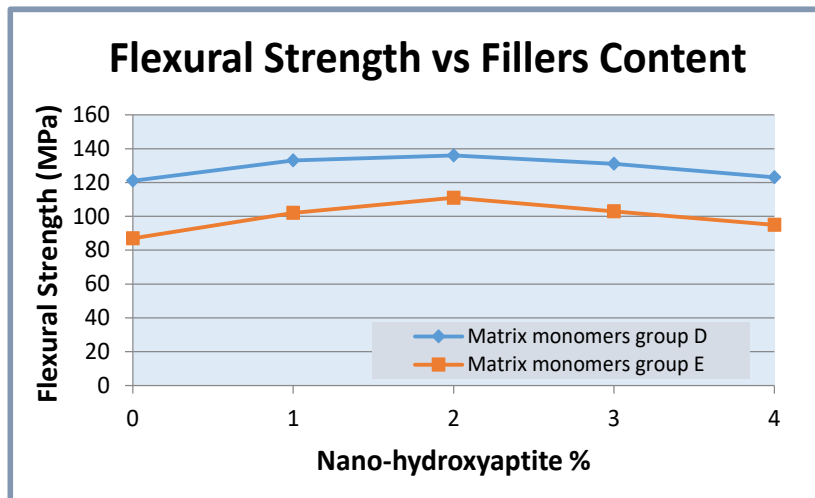


Fig. 4. Flexural Strength for hybrid nanocomposites based on the matrix of groups D or E as a function of hydroxyapatite nanoparticles content in the composite.

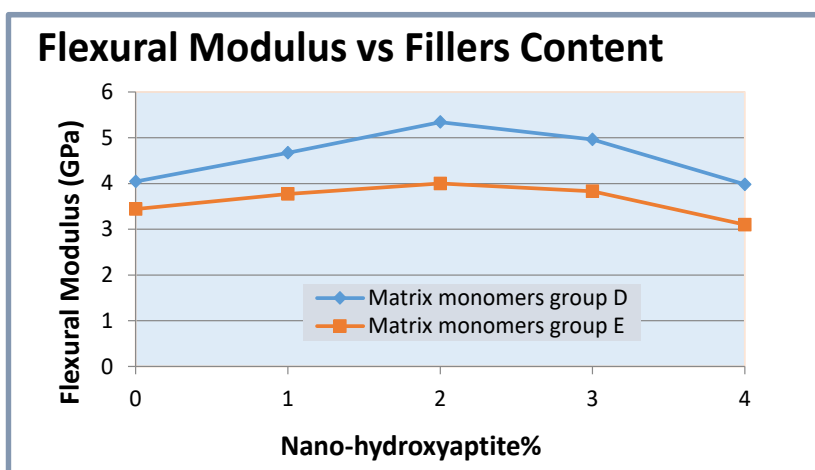


Fig. 5. Flexural modulus for hybrid nanocomposites based on the matrix of groups D or E as a function of hydroxyapatite nanoparticles content in the composite.

3.44 to 3.77GPa, respectively, for the matrix of monomers group E.

The maximum values of flexural strength and flexural modulus have been observed at ratio 2%wt of hydroxyapatite content, where it reached to 136MPa and 5.34GPa, respectively, for hybrid nanocomposites based on the matrix D, whereas, reached to 111MPa and 4GPa, respectively for hybrid nanocomposites based on the matrix E.

Other studied about the connecting between the properties of composites and the variation in filler ratios content, found that the composites with high filler content show better mechanical and physical properties [12]. These studies look to be agreeing with the results of the current study

but to limited content, it was found the hybrid nanocomposite resins that contain on 2%wt of hydroxyapatite nanoparticle showed significantly the higher values of flexural strength and flexural modulus as mentioned earlier, after this ratio of hydroxyapatite nanoparticle specifically at ratios (3 and 4) %wt., these properties decreased in their values.

A similar result was also shown by many researchers that found the extra increase in the used filler amount lead to a decrease in flexural strength [13]. These results indicated that the successful function of reinforcement by nanoparticle would not be accomplished at high ratios of weight fraction, perhaps these

large amounts can affect the formation of fillers agglomerates, which considered as weak points in composites (structural defects); next, the existence of such agglomerates would decrease the mechanical properties (particularly strength property) of dental composites that reinforced by nanoparticles. [14, 15]

Other reasons explained this behavior such as a decrease in the effective load-bearing cross-section of the resin matrix at a concentration of nano-filler higher than limited content [16]; as well as, high ratios of filler content in composite usually caused a poor adhesion between filler and matrix.

Moreover, it was noticed from Figs. 4 and 5 that the rate of increase in flexural strength and flexural modulus depends on matrix monomer content of groups D and E.

Therefore, it can be concluded that the content of fillers does not individually affect the mechanical properties, but other factors such as size and type of filler, matrix composition, amount of initiators, and microstructure morphology contribute to the improvement of physical and mechanical properties. [17].

Types of nano-filler: Based on foregoing results, it was choosing the best percentage (2%wt) nanofillers content. Then added different types of nano-fillers (ZrO_2 , SiO_2 , HA, and Al_2O_3) individually to each group of matrixes monomers (A, B, C, D, E, and F) mentioned earlier in Table 1, at a constant

ratio of weight fraction (2%wt). Figs. 6 and 7 show the flexural strength and flexural modulus of hybrid nanocomposite materials for dental restorative as a function of matrix type.

These figures showed a remarkable increase in flexural strength and flexural modulus with adding (2 %wt.) ratio of nanoparticles (ZrO_2 , SiO_2 , HA, and Al_2O_3) individually in the matrix monomers of the groups (A, B, C, D, E, and F), as compared with the analogs of the matrices from which it is built. The rate of increment in flexural strength and flexural modulus values are dependent on the type of the fillers, as well as, on the matrix type (copolymers materials), and their properties [18, 19].

As well as, it can be seen that, the flexural strength and flexural modulus values are varying within the individual group; this is related to the use the different types of nanoparticles from ceramic fillers.

Furthermore, it was established from these Figures that, the average values of flexural strength for all the prepared nanocomposite specimens range from the lowest value (87 MPa) for a composite that based on matrix group (E) and reinforced with ZrO_2 nanoparticles to the highest value (144MPa) for a composite that based on matrix group (D) and reinforced with SiO_2 nanoparticles.

Furthermore, was noticed that the matrix monomers of group D that reinforced by SiO_2 nanoparticles showed the highest value of elastic

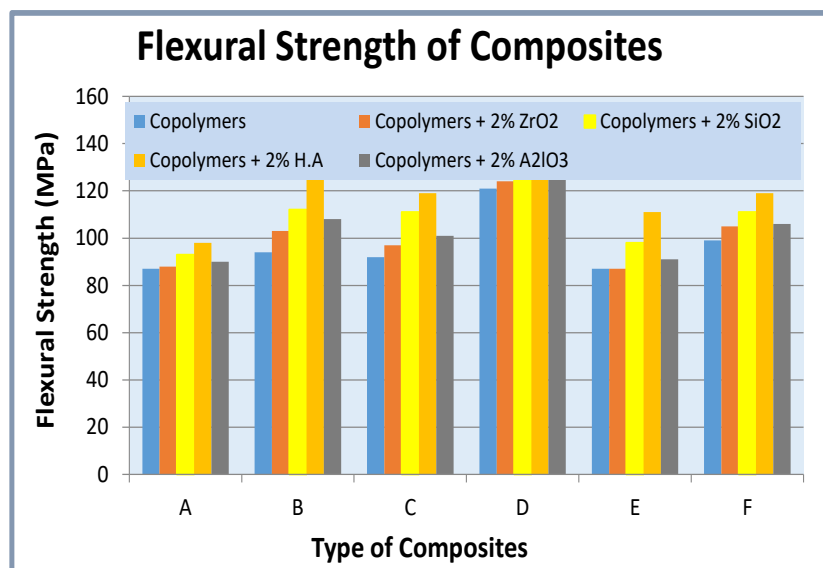


Fig. 6. Flexural strength of prepared hybrid nanocomposites as a function of fillers type and the type of matrix material (monomers group of copolymers materials).

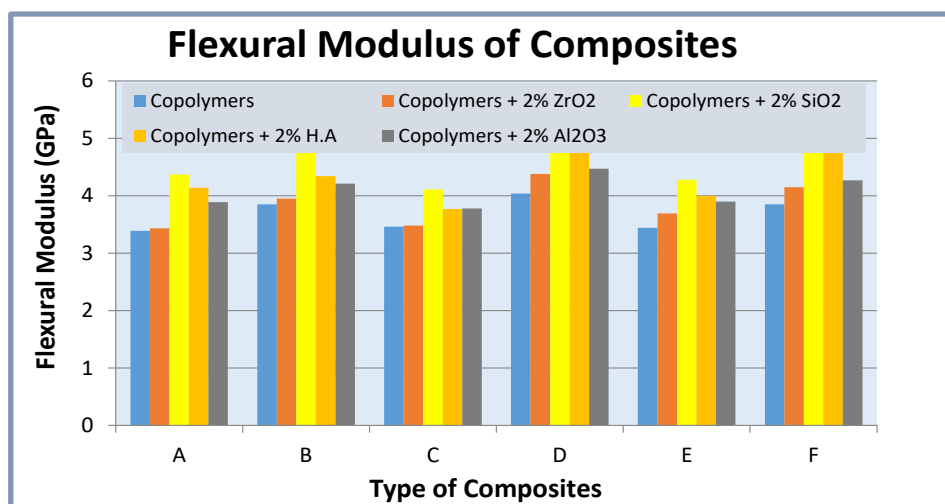


Fig. 7. Flexural modulus of prepared hybrid nanocomposites as a function of fillers type and the type of matrix material (monomers group of copolymers materials).

modulus (5.47GPa) in comparison with all other composites, while the lowest values (3.43GPa) were recorded with the matrix monomers group (A) that reinforced by ZrO₂ nanoparticles.

And upon it, all the prepared hybrid nanocomposite materials of dental restorative achieve higher flexural strength values than ones proposed by the ISO suggesting, so all these composites can further be used as direct restoration for the damaged dental materials.

On other hand, the results showed that the maximum value of flexural strength was obtained for all the prepared copolymers composites when reinforced there with silica nanoparticles followed by these reinforced by hydroxyapatite, alumina, and zirconia nanoparticles which were added to all the prepared matrices. This is related to the small size of silica and hydroxyapatite fillers particle which are (10-20 nm) and 25nm, respectively, contributes to increased mechanical strength due to increased contact surface area of these particles with organic resin [15].

From these results, it can be concluded that the prepared dental composites based on different types of new monomers of matrix material and containing nanoparticles have improved flexural properties.

These results are related to what the former reported about monomer properties (such as chemical composition and structure of chains) have a significant effect on resultant composites behavior where the more flexible composite resin is produced due to the high flexibility of the resin,

which increased the flexural strength and flexural modulus and helps to reduce brittleness. [20, 21]

The chemical composition of monomer content in the resin has a direct effect on the mechanical properties of the composites. Prior researches proved that flexural strength value rises after a part of the weight percentage of TEGDMA is replaced by 1-6 HDDA (Note Table No. 1). In addition, a decrease in flexural strength was viewed when Bis-GMA was mixed with other monomers [22] due to the higher viscosity of Bis-GMA than other monomers. This is an especial reason that might clarify the results of the current study.

Nanocomposites that contain methacrylate monomers in their matrix maybe explain the reason for the higher flexural properties with the same type of nano-filler material, therefore, the highest values of flexural strength and flexural modulus have been gained by nanocomposites that based on the monomers matrix that contains meth acrylamide of (group D) is (144 MPa) and (5.47 GPa), respectively, which was much higher than the other tested materials, flowed by groups B, C, F, E, and A respectively.

The second factor that affected the properties of composite is the addition of nanoparticles filler. Where it was observed from Figs. 6 and 7 that the addition of nanopowders generally increased the flexural properties of copolymer-composites and the rate of increase depends on the nature of reinforcing materials for the same matrix material and how strong is the adhesion between it and the matrix material.

One of the reasons is due to the ability of nanoparticle fillers to the formation of composites materials that have strong physical bonding; as a result, it is required higher flexural stress for breaking, which resulted in the increase of both flexural modulus and strength. Moreover, the increase is due to the ability of some nano-powders to increase the resistance in the formation of cracks, and it also tends to inhibit the growth of microcracks, as it is known that the presence of microcracks in the structure of composite resins causes a further decrease in the mechanical properties [23].

From the other reasons, that the strong interfacial interface between organic matrix and inorganic fillers and uniform dispersion of nano-fillers in the composite materials produced a homogeneous distribution of stresses subsequent to prevent of stress concentration, which causing a considerable increase in mechanical properties of the resultant composites. The homogeneous dispersion, jointly with the excellent interfacial adhesion between resin/fillers, enhances the mechanical properties of composites [23, 24].

In fact, the main factor of this variance in results is related to the properties and structure of each type of fillers together with their ability to support crystal deposition and development on designed teeth.

The strong bond between resin and fillers in dental composites is an essential issue, and it is assumed to be one of the most important factors that must be considered in the reinforcing of resin to produce the composites. Therefore the high flexural strength obtained in this study can be associated with the good interconnection between fillers and resin.

Although this variation in flexural properties data with the fillers type are interesting, the principal reasons for this result are probably quite complex and involved multiple factors connected with particle size and geometry, the morphology of fillers, and homogeneity of fillers distribution in the matrix [25].

CONCLUSION

From this study, can be concluded the following:

1. New dental restorative nanocomposites over that using at markets contain unique monomers and different fillers were successfully fabricated through the photopolymerization process.

2. It can also be confirmed that flexural properties of studied dental composites are in the satisfactory value for dental ISO standards.

3. The flexural strength and the flexural modulus were increased by the incorporation of the nanofillers into polymer resin according to the type of these nanofillers and the chemical composition of matrix monomers materials.

4. The various materials of the prepared nanocomposite showed different flexural properties that connected to their chemical composition and interfacial interaction between organic and inorganic phases.

5. The higher values of flexural properties obtain in this study, are due to existing methacrylamide and methacrylic acid monomers and silica nanoparticles in the dental restorative nanocomposite.

6. The dental restorative nanocomposite that prepared from matrix monomers group D and reinforced by 2% silica nanoparticles, can consider as one of the most promising samples in the restoration and repair of dental.

CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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