

Elastic Properties of substituted 45S5 Bioactive Glasses and Glass - Ceramics

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Abstract — CuO, Fe₂O₃, MnO₂ and ZnO substituted 45S5 bioactive - glasses were prepared. Glass - derived Bioactive Glass - ceramics were obtained through controlled crystallization of bioactive glasses. The formed crystalline phases in bioactive glass - ceramics were identified using X - ray diffraction (XRD) analysis. Density and ultrasonic wave velocities of bioactive glasses and glass - ceramics were measured and used to study elastic properties: Young's modulus, shear modulus, bulk modulus and Poisson's ratio, of bioactive glasses and glass - ceramics. The results indicate that the addition of CuO, Fe₂O₃, MnO₂ and ZnO contents in 45S5 bioactive glass enhances its density and elastic properties. The bioactive glass - ceramics exhibits higher density and elastic properties when compared with their corresponding bioactive glasses.

Index Terms — Bioceramics, Bioactive Glasses, Bioactive Glass - ceramics, Physical Properties, Bioactivity, Mechanical Properties, Elastic Modulus, Poisson's ratio

1 INTRODUCTION

The most widely researched bioactive material is 45S5 bioactive glass [Composition wt. % 45 SiO₂ - 24.5 Na₂O - 24.5 CaO - 6 P₂O₅], where S denotes the network former SiO₂ in 45% by weight followed by a specific Ca/P molar ratio 5 [1]. It was invented by Hench in 1969. The key compositional features that are responsible for the bioactivity of 45S5 bioactive glass are its low SiO₂ (glass network former) content, high Na₂O and CaO (glass network modifiers) content, and high CaO/P₂O₅ ratio [2].

An ideal bioactive material requires both good biochemical and biomechanical compatibility. A particular advantage of 45S5 bioactive glass is its ability to bond to both hard and soft tissues [3]. However, a disadvantage of 45S5 bioactive glass is its brittleness. As a consequence, it has poor mechanical properties and is generally restricted to clinical non - load bearing situation [4].

The mechanical strength of a glass depends on its elastic properties, shape, surface/internal defects and the character of the force to which it is subjected. These factors inevitably degrade the mechanical strength of a material to 1/10 or lower of its intrinsic (theoretical) strength [5]. Thus, one cannot obtain the intrinsic strength by using destructive mechanical test on a big specimen. The ultrasonic non - destructive testing has been found to be one of the best techniques to study the elastic properties of glasses.

The present work aims to measure the density and ultrasonic wave velocities (both longitudinal and shear) for CuO, Fe₂O₃, MnO₂ and ZnO substituted 45S5 bioactive glasses and glass - ceramics. These values have been used to evaluate Young's, bulk and shear modulus and Poisson's ratio of bioactive glasses and glass - ceramics.

2 EXPERIMENTAL

2.1 Preparation of Bioactive Glasses and Glass - ceramics

Fine grained quartz was used as the source of SiO₂ while Na₂O and CaO were introduced in the form of anhydrous sodium carbonate [Na₂CO₃] and anhydrous calcium carbonate [CaCO₃] respectively, P₂O₅ was added in the form of ammonium dihydrogen orthophosphate [NH₄H₂PO₄] and CuO, Fe₂O₃, MnO₂ and ZnO was added as such for preparation of bioactive glasses. All the batch materials were of analytical grade chemicals and used without further purification. The compositions of bioactive glasses are given in Table 1. The weighed batches were melted in alumina crucibles for 3 hours in an electric furnace at the temperature 1400 ± 10 °C. The homogeneous melts were cast into preheated stainless steel moulds of the required dimensions. The prepared bioactive glass samples were directly transferred to a regulated muffle furnace at the temperature 500 °C for annealing. After 1 h, the muffle furnace was left to cool to room temperature at a rate of 30 °C/h.

In order to obtain the bioactive glass - ceramics, the bioactive glass samples were heated in the muffle furnace in two step regime at the deduced temperatures and times as shown in Table 2. These temperatures were obtained from differential thermal analysis (DTA) of bioactive glasses. Each bioactive glass sample was heated slowly to the first nucleation temperature for the formation of sufficient nuclei sites and after holding for the definite time, was then further heated to reach the second chosen crystal growth temperature for performing the perfect crystal growth process and after a second hold for the specific time, the sample was left to cool inside the muffle furnace to room temperature at a rate of 20 °C/h.

Table 1: Composition of bioactive glasses

| Code for glasses | Composition (wt %) | | | | |
|------------------|---|-------------------|-------|-------------------------------|--------------------------------|
| | CuO substituted 45S5 bioactive glasses | | | | |
| | SiO ₂ | Na ₂ O | CaO | P ₂ O ₅ | CuO |
| C1 | 44.00 | 24.50 | 24.50 | 6.00 | 1.00 |
| C2 | 43.00 | 24.50 | 24.50 | 6.00 | 2.00 |
| C3 | 42.00 | 24.50 | 24.50 | 6.00 | 3.00 |
| C4 | 41.00 | 24.50 | 24.50 | 6.00 | 4.00 |
| | Fe ₂ O ₃ substituted 45S5 bioactive glasses | | | | |
| | SiO ₂ | Na ₂ O | CaO | P ₂ O ₅ | Fe ₂ O ₃ |
| F1 | 44.00 | 24.50 | 24.50 | 6.00 | 1.00 |
| F2 | 43.00 | 24.50 | 24.50 | 6.00 | 2.00 |
| F3 | 42.00 | 24.50 | 24.50 | 6.00 | 3.00 |
| F4 | 41.00 | 24.50 | 24.50 | 6.00 | 4.00 |
| | MnO ₂ substituted 45S5 bioactive glasses | | | | |
| | SiO ₂ | Na ₂ O | CaO | P ₂ O ₅ | MnO ₂ |
| M1 | 44.00 | 24.50 | 24.50 | 6.00 | 1.00 |
| M2 | 43.00 | 24.50 | 24.50 | 6.00 | 2.00 |
| M3 | 42.00 | 24.50 | 24.50 | 6.00 | 3.00 |
| M4 | 41.00 | 24.50 | 24.50 | 6.00 | 4.00 |
| | ZnO substituted 45S5 bioactive glasses | | | | |
| | SiO ₂ | Na ₂ O | CaO | P ₂ O ₅ | ZnO |
| Z1 | 44.00 | 24.50 | 24.50 | 6.00 | 1.00 |
| Z2 | 43.00 | 24.50 | 24.50 | 6.00 | 2.00 |
| Z3 | 42.00 | 24.50 | 24.50 | 6.00 | 3.00 |
| Z4 | 41.00 | 24.50 | 24.50 | 6.00 | 4.00 |
| | Base 45S5 bioactive glass | | | | |
| | SiO ₂ | Na ₂ O | CaO | P ₂ O ₅ | |
| 45S5 | 44.00 | 24.50 | 24.50 | 6.00 | |

Table 2: Heat treatment schedule for crystallization of bioactive glasses

| Sample | Nucleation | | Growth | |
|---|------------------|--------------|------------------|--------------|
| | Temperature (°C) | Time (hours) | Temperature (°C) | Time (hours) |
| CuO substituted 45S5 bioactive glasses | | | | |
| C1 | 518 | 6 | 675 | 3 |
| C2 | 510 | 6 | 651 | 3 |
| C3 | 504 | 6 | 631 | 3 |
| C4 | 499 | 6 | 616 | 3 |
| Fe ₂ O ₃ substituted 45S5 bioactive glasses | | | | |
| F1 | 527 | 6 | 682 | 3 |
| F2 | 523 | 6 | 665 | 3 |
| F3 | 519 | 6 | 650 | 3 |
| F4 | 516 | 6 | 640 | 3 |
| MnO ₂ substituted 45S5 bioactive glasses | | | | |
| M1 | 525 | 6 | 678 | 3 |
| M2 | 520 | 6 | 657 | 3 |
| M3 | 517 | 6 | 640 | 3 |
| M4 | 514 | 6 | 627 | 3 |
| ZnO substituted 45S5 bioactive glasses | | | | |
| Z1 | 528 | 6 | 686 | 3 |
| Z2 | 524 | 6 | 671 | 3 |
| Z3 | 521 | 6 | 661 | 3 |
| Z4 | 518 | 6 | 651 | 3 |
| Base 45S5 bioactive glass | | | | |
| 45S5 | 533 | 6 | 717 | 3 |

2.2 X - Ray Diffraction (XRD) Analysis

Identification of the crystalline phases after heat - treatment of bioactive glass samples was carried out by X - ray diffraction (XRD) analysis. The bioactive glass - ceramics were examined using a X - ray diffractometer, adopting Ni filter and Cu target with voltage of 40 KV and a current of 25 mA. The XRD patterns were recorded in a 2θ range of $10 - 70^\circ$. The JCPDS - International Center for Diffraction Data Cards was used as a reference data for the interpretation of XRD patterns in the present work.

2.3 Density Measurements

Archimedes principle was employed to obtain the density of bioactive glass and glass - ceramic samples using distilled water as buoyant. All the weight measurements have been made using a digital balance having an accuracy of ± 0.0001 g. Density (ρ) of sample was obtained employing the relation [6] given below [6]:

$$\rho = \frac{w_a}{w_a - w_b} \rho_b$$

where w_a is the weight of sample in air, w_b is the weight of sample in buoyant and ρ_b is the density of buoyant.

2.4 Elastic Properties Measurements

The ultrasonic wave velocities propagated in the polished bioactive glass and glass - ceramic specimens were obtained by measuring the elapsed time between the initiation and the receipt of the pulse appearing on the screen of an ultrasonic flaw detector via a standard electronic circuit. The size of the specimen was 20 mm x 10 mm x 3 mm according to ASTM Standard: E494 - 10. Two quartz transducers in this machine, which are X - cut transducer and Y - cut transducer, with has a resonant frequency of 5 MHz and acts as transmitter - receiver at the same time, were required to generate longitudinal and transverse ultrasonic waves respectively. Burnt honey was used as bonding material between samples and transducers. The ultrasonic wave velocity (V) can be calculated using the following equation [7]:

$$V = \frac{2X}{\Delta t}$$

where X is the sample thickness and Δt is the time interval. The elastic strain in an amorphous solid (such as glass) generated by a minor stress, can be described by two independent elastic constants, C_{11} and C_{44} [8]. For pure longitudinal ultrasonic waves, $C_{11} = \rho V_L^2$ and for pure transverse ultrasonic waves, $C_{44} = \rho V_T^2$, where V_L and V_T , respectively, are the longitudinal and transverse ultrasonic wave velocities. Elastic properties: Young's modulus (E), shear modulus (G), bulk modulus (K) and Poisson's ratio (σ), can be obtained by density (ρ) and longitudinal (V_L)

and shear (V_S) ultrasonic wave velocities by using the following standard equations [9]:

$$E = \rho V_L^2 \frac{3V_L^2 - 4V_T^2}{V_L^2 - V_T^2}$$

$$G = \rho V_T^2$$

$$K = \frac{1}{3} \rho (3V_L^2 - 4V_T^2)$$

$$\sigma = \frac{V_L^2 - 2V_T^2}{2(V_L^2 - V_T^2)}$$

3 RESULTS

3.1. X - Ray Diffraction (XRD) Analysis

The X - ray diffraction (XRD) patterns for CuO substituted 45S5 bioactive glass - ceramics are shown in Figure 1. The XRD patterns of all the bioactive glass - ceramics show the presence of crystalline phase of sodium calcium silicate [$\text{Na}_2\text{Ca}_2\text{Si}_3\text{O}_9$ (card number: PDF # 01 - 1078 & PDF # 02 - 0961), $\text{Na}_2\text{CaSi}_3\text{O}_8$ (card number: PDF # 12 - 0671)]. Each CuO substituted bioactive glass - ceramics (C1, C2, C3 and C4) also, show the presence of crystalline phase of calcium silicate [CaSiO_3 (card number: PDF # 42 - 0550)].

3.2 Density Measurements

Experimental values of density of bioactive glasses and glass ceramics are given in Table 3. It has been observed that the increase of CuO, Fe_2O_3 , MnO_2 and ZnO contents in the base bioactive glass (45S5) causes an increase in its density. It also has been observed that the density of bioactive glass - ceramics are higher than their respective bioactive glasses.

3.3 Elastic Properties Measurements

Values of longitudinal and shear ultrasonic wave velocities of investigated bioactive glasses and glass - ceramics are given in Table 3 while values of Young's, shear and bulk modulus and Poisson's ratio are listed in Table 4. Variation in Young's, shear and bulk modulus and Poisson's ratio of investigated bioactive glasses and glass - ceramics are shown in Figures 2, 3, 4 and 5 respectively. Both longitudinal and shear ultrasonic wave velocities and Young's, shear and bulk modulus of base bioactive glass (45S5) increases with the incorporation of CuO, Fe_2O_3 , MnO_2 and ZnO contents in it.while a slight decrease in Poisson's ratio was obtained. It also has been founded that the crystallization of bioactive glasses increases their both longitudinal and shear ultrasonic wave velocities, Young's, shear and bulk modulus while decreases the Poisson's ratio.

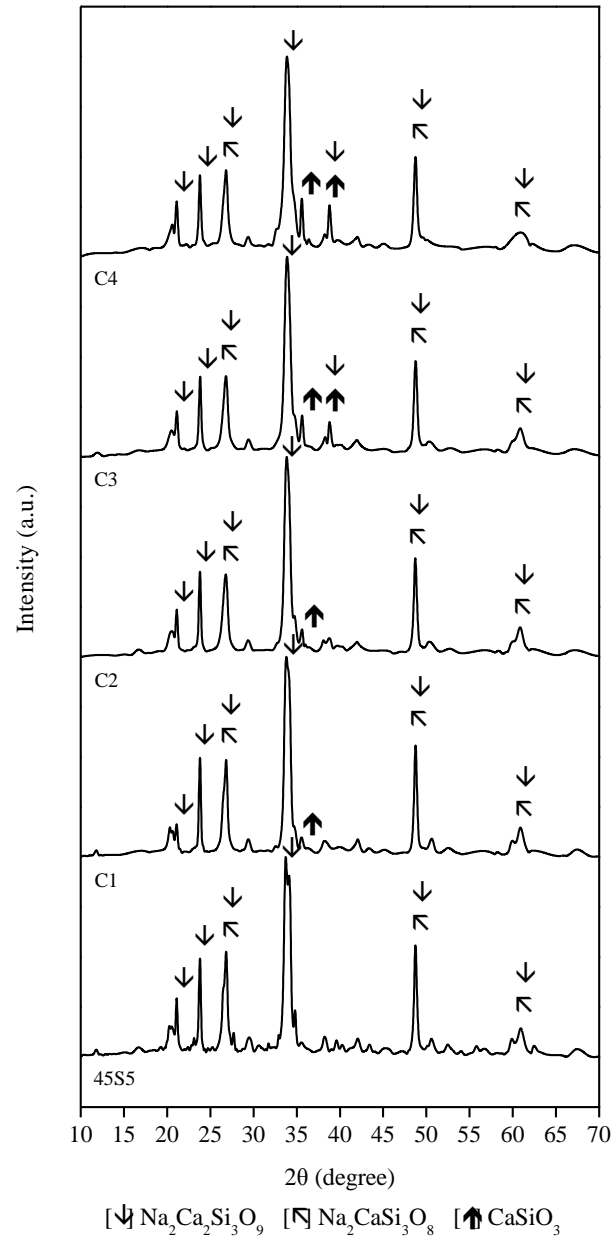


Figure 1: XRD Patterns for CuO

ceramics

substituted 45S5 bioactive glass -

Table 3: Density (ρ), longitudinal velocity (V_L) and transverse velocity (V_T) of bioactive glasses and glass ceramics

| Sample | Glasses | | | Glass - ceramics | | |
|--------|---|-------------|-------------|--|-------------|-------------|
| | ρ (g/cm ³) | V_L (m/s) | V_T (m/s) | ρ (g/cm ³) | V_L (m/s) | V_T (m/s) |
| | CuO substituted 45S5 bioactive glasses | | | CuO substituted 45S5 bioactive glasses - ceramics | | |
| C1 | 2.732 | 5915 | 3370 | 2.943 | 6519 | 3744 |
| C2 | 2.742 | 5927 | 3381 | 2.954 | 6531 | 3757 |
| C3 | 2.759 | 5940 | 3394 | 2.972 | 6547 | 3772 |
| C4 | 2.772 | 5961 | 3411 | 2.986 | 6569 | 3789 |
| | Fe ₂ O ₃ substituted 45S5 bioactive glasses | | | Fe ₂ O ₃ substituted 45S5 bioactive glasses - ceramics | | |
| F1 | 2.723 | 5913 | 3368 | 2.925 | 6517 | 3743 |
| F2 | 2.731 | 5925 | 3380 | 2.932 | 6530 | 3755 |
| F3 | 2.746 | 5937 | 3393 | 2.946 | 6544 | 3770 |
| F4 | 2.757 | 5958 | 3409 | 2.956 | 6562 | 3784 |
| | MnO ₂ substituted 45S5 bioactive glasses | | | MnO ₂ substituted 45S5 bioactive glasses - ceramics | | |
| M1 | 2.738 | 5927 | 3382 | 2.937 | 6533 | 3761 |
| M2 | 2.749 | 5941 | 3397 | 2.947 | 6550 | 3775 |
| M3 | 2.767 | 5965 | 3415 | 2.964 | 6571 | 3793 |
| M4 | 2.781 | 5983 | 3431 | 2.977 | 6587 | 3807 |
| | ZnO substituted 45S5 bioactive glasses | | | ZnO substituted 45S5 bioactive glasses - ceramics | | |
| Z1 | 2.727 | 5912 | 3366 | 2.928 | 6515 | 3741 |
| Z2 | 2.736 | 5921 | 3379 | 2.936 | 6527 | 3752 |
| Z3 | 2.752 | 5933 | 3387 | 2.951 | 6541 | 3766 |
| Z4 | 2.764 | 5947 | 3400 | 2.962 | 6556 | 3780 |
| | Base 45S5 bioactive glass | | | Base 45S5 bioactive glass - ceramics | | |
| 45S5 | 2.707 | 5904 | 3353 | 2.912 | 6507 | 3728 |

Table 4: Young's modulus (E), shear modulus (G), bulk modulus (K) and Poisson's ratio (σ) of bioactive glasses and glass ceramics

| Sample | Glasses | | | | Glass - ceramics | | | |
|--------|---|-----------|-----------|----------|--|-----------|-----------|----------|
| | E (GPa) | G (GPa) | K (GPa) | σ | E (GPa) | G (GPa) | K (GPa) | σ |
| | CuO substituted 45S5 bioactive glasses | | | | CuO substituted 45S5 bioactive glasses - ceramics | | | |
| C1 | 78.10 | 31.02 | 54.22 | 0.259 | 103.37 | 41.25 | 70.06 | 0.253 |
| C2 | 78.85 | 31.34 | 54.53 | 0.258 | 104.39 | 41.69 | 70.40 | 0.252 |
| C3 | 79.89 | 31.78 | 54.96 | 0.257 | 105.78 | 42.28 | 71.00 | 0.251 |
| C4 | 81.01 | 32.25 | 55.49 | 0.256 | 107.15 | 42.86 | 71.70 | 0.250 |
| | Fe ₂ O ₃ substituted 45S5 bioactive glasses | | | | Fe ₂ O ₃ substituted 45S5 bioactive glasses - ceramics | | | |
| F1 | 77.75 | 30.88 | 54.02 | 0.259 | 102.67 | 40.97 | 69.59 | 0.253 |
| F2 | 78.49 | 31.20 | 54.27 | 0.258 | 103.51 | 41.34 | 69.90 | 0.252 |
| F3 | 79.46 | 31.61 | 54.64 | 0.257 | 104.75 | 41.87 | 70.32 | 0.251 |
| F4 | 80.45 | 32.03 | 55.15 | 0.256 | 105.80 | 42.32 | 70.85 | 0.250 |
| | MnO ₂ substituted 45S5 bioactive glasses | | | | MnO ₂ substituted 45S5 bioactive glasses - ceramics | | | |
| M1 | 78.77 | 31.31 | 54.43 | 0.258 | 104.01 | 41.54 | 69.96 | 0.252 |
| M2 | 79.74 | 31.72 | 54.72 | 0.257 | 105.05 | 41.99 | 70.44 | 0.251 |
| M3 | 81.03 | 32.26 | 55.43 | 0.256 | 106.60 | 42.64 | 71.11 | 0.250 |
| M4 | 82.15 | 32.73 | 55.90 | 0.255 | 107.76 | 43.14 | 71.64 | 0.249 |
| | ZnO substituted 45S5 bioactive glasses | | | | ZnO substituted 45S5 bioactive glasses - ceramics | | | |
| Z1 | 77.84 | 30.89 | 54.12 | 0.260 | 102.75 | 40.97 | 69.64 | 0.254 |
| Z2 | 78.63 | 31.23 | 54.27 | 0.259 | 103.57 | 41.33 | 69.96 | 0.253 |
| Z3 | 79.43 | 31.57 | 54.77 | 0.258 | 104.79 | 41.85 | 70.45 | 0.252 |
| Z4 | 80.32 | 31.95 | 55.15 | 0.257 | 105.88 | 42.32 | 70.88 | 0.251 |
| | Base 45S5 bioactive glass | | | | Base 45S5 bioactive glass - ceramics | | | |
| 45S5 | 76.74 | 30.43 | 53.77 | 0.261 | 101.57 | 40.47 | 69.33 | 0.255 |

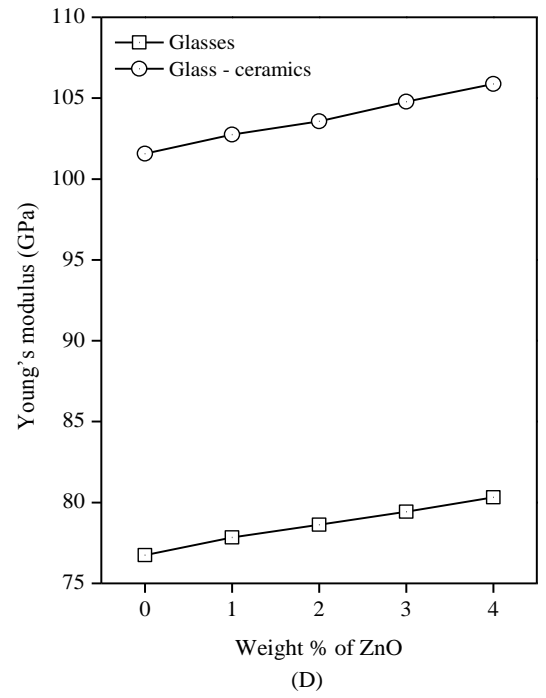
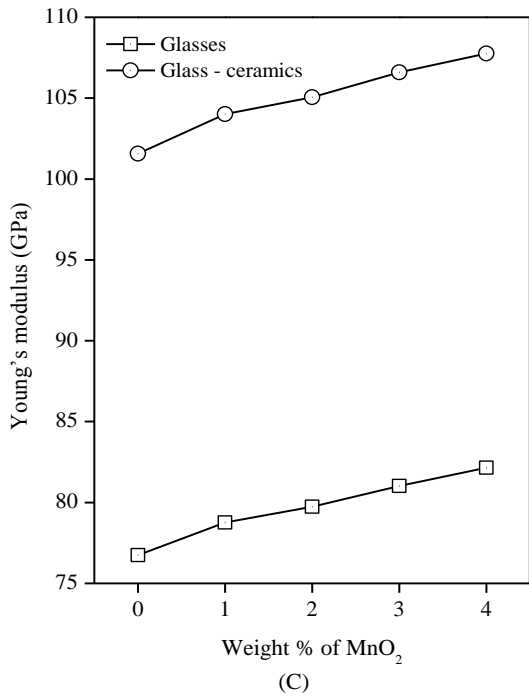
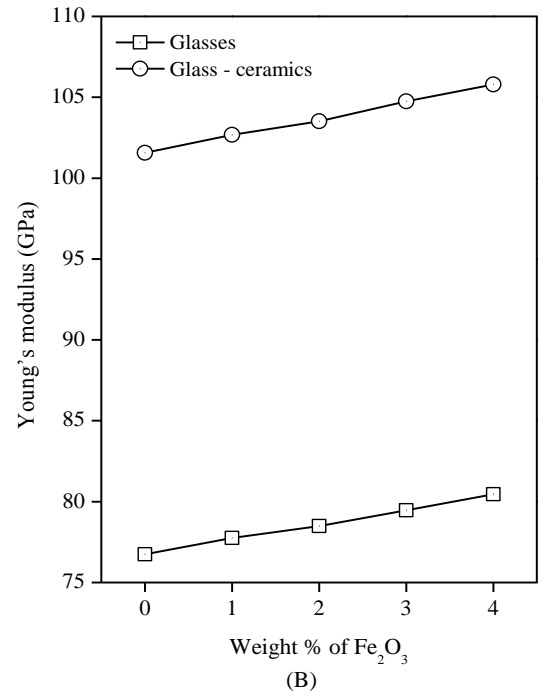
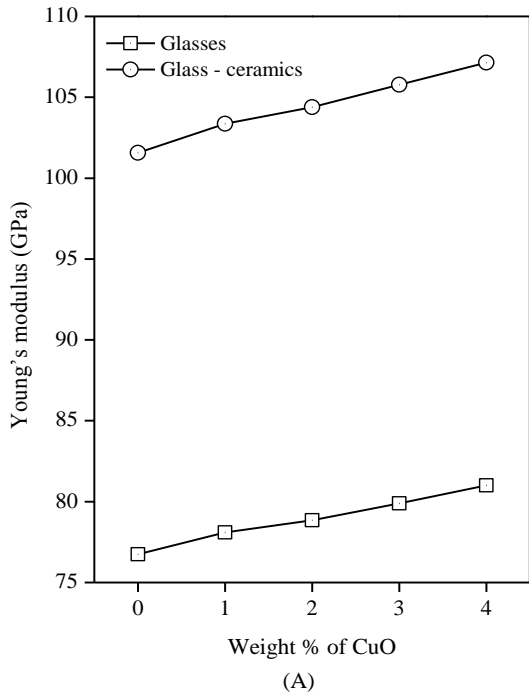


Figure 2: Variation in Young's modulus for (A) CuO, (B) Fe₂O₃, (C) MnO₂ and (D) ZnO substituted 45S5 bioactive glasses and glass - ceramics

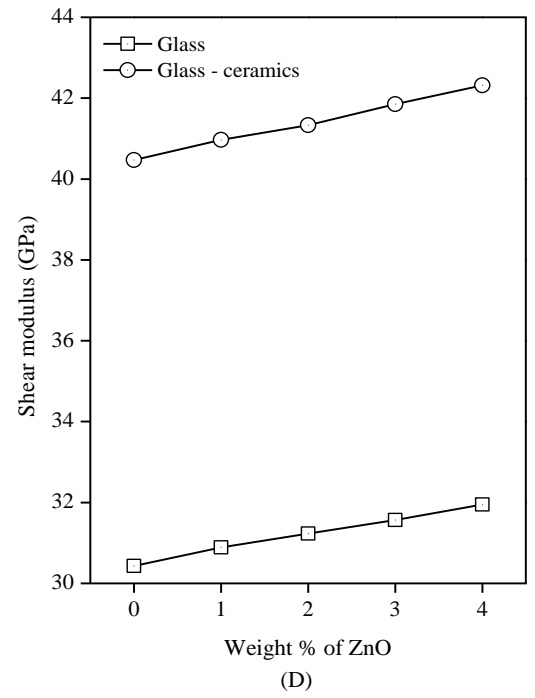
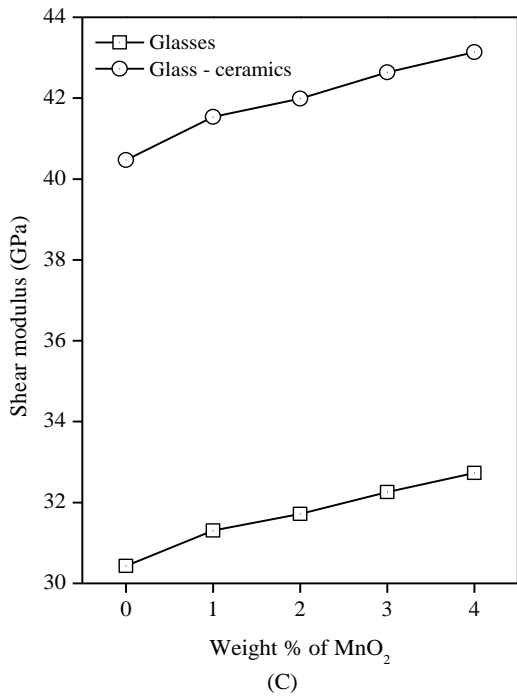
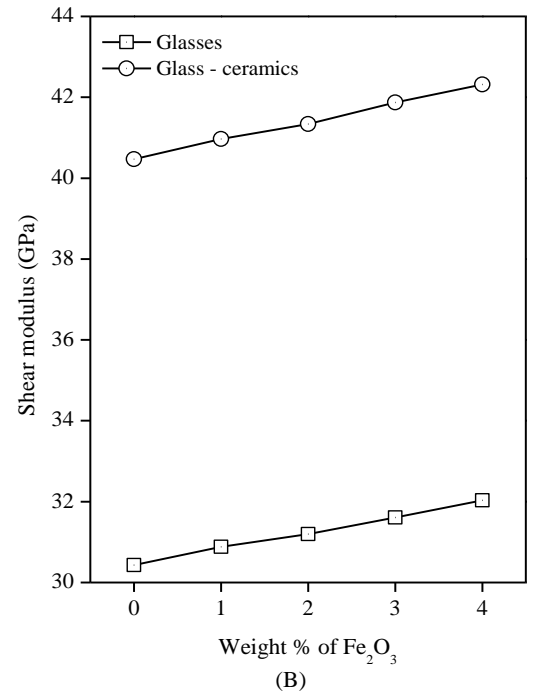
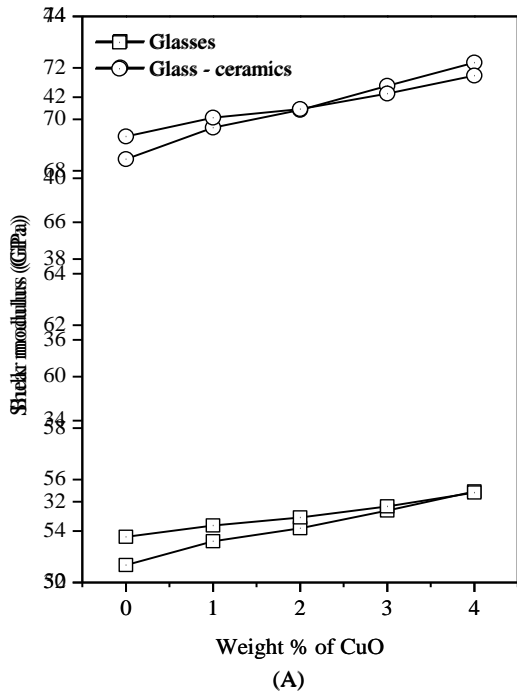


Figure 3: Variation in shear modulus for (A) CuO, (B) Fe₂O₃, (C) MnO₂ and (D) ZnO substituted 45S5 bioactive glasses and glass - ceramics

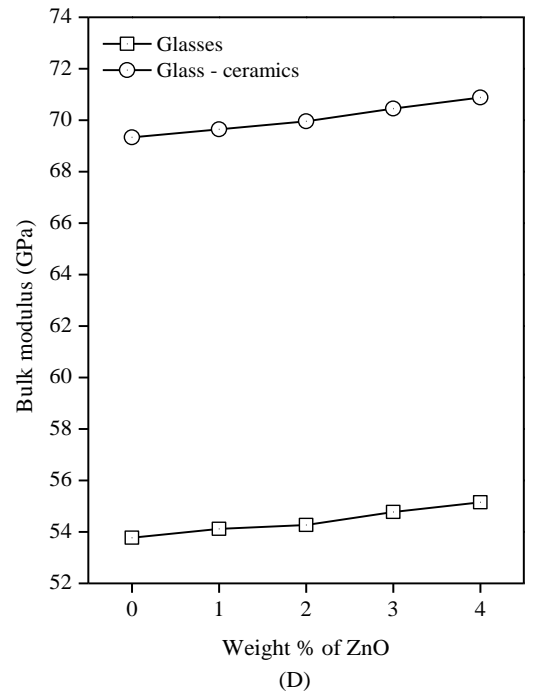
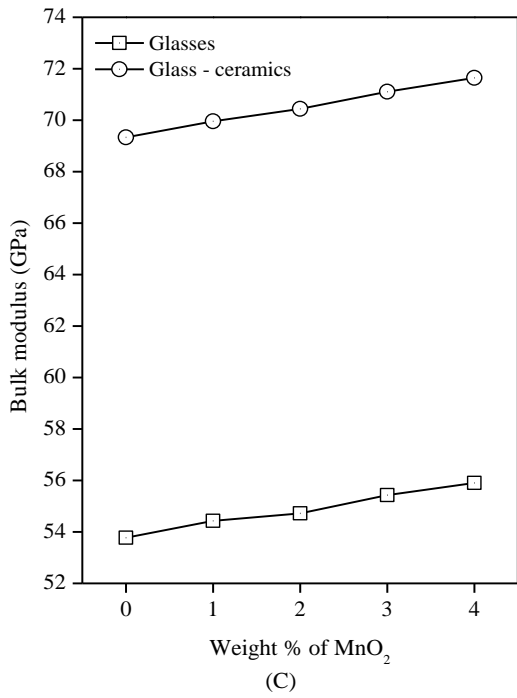
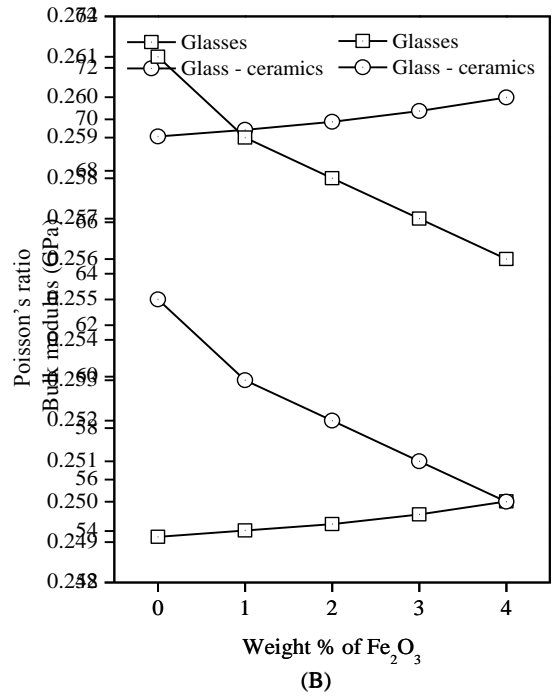
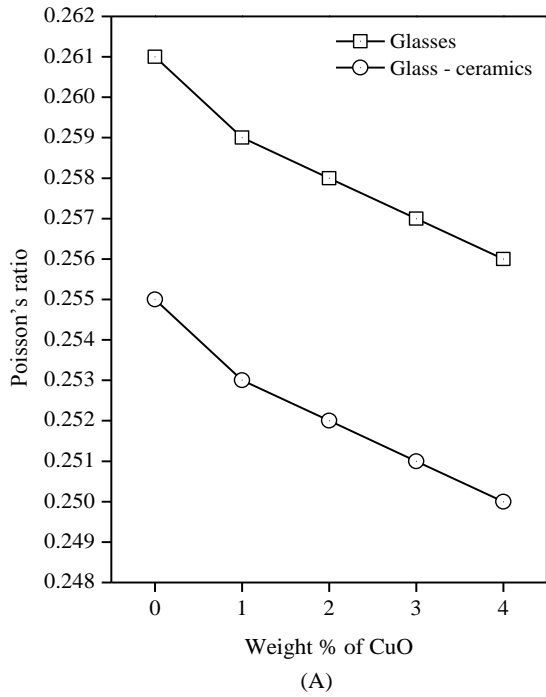


Figure 4: Variation in bulk modulus for (A) CuO, (B) Fe₂O₃, (C) MnO₂ and (D) ZnO substituted 45S5 bioactive glasses and glass - ceramics

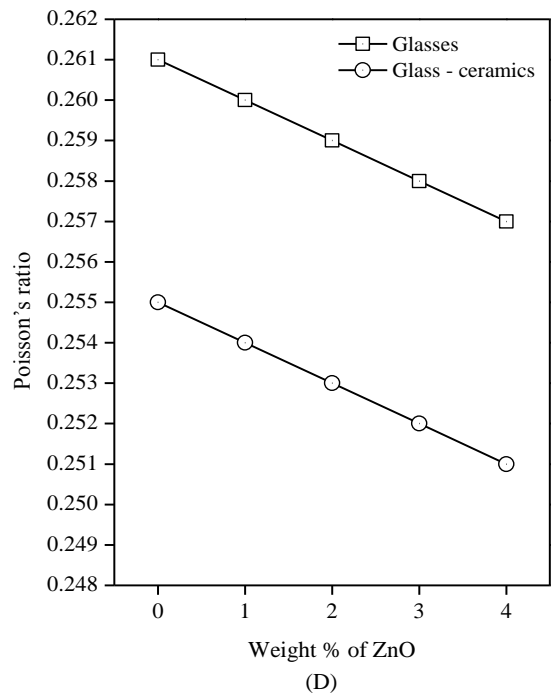
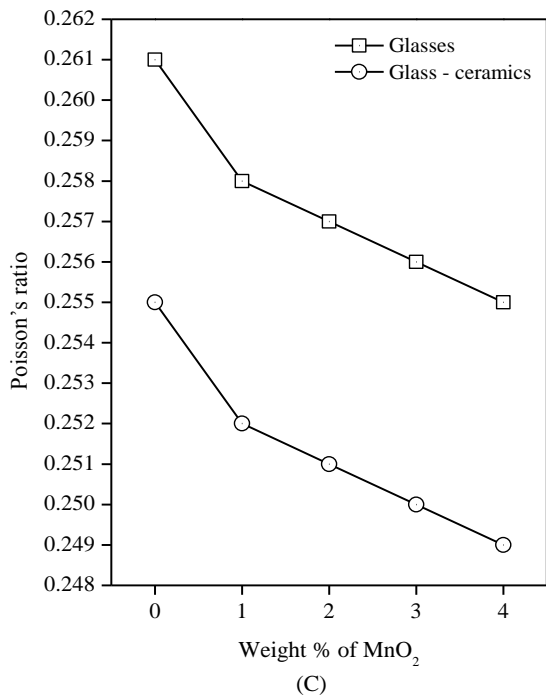


Figure 5: Variation in Poisson's ratio for (A) CuO, (B) Fe₂O₃, (C) MnO₂ and (D) ZnO substituted 45S5 bioactive glasses and glass - ceramics

4 DISCUSSIONS

4.1 X – Ray Diffraction (XRD) Analysis

The XRD patterns of all the bioactive glass - ceramics show the presence of crystalline phases. The reason for the ease of

crystallization of bioactive glasses can be correlated with the presence of silicate and phosphate network, as well as the possible phase separation even in micro scale of the two phases on heat - treatment. It is well known that the addition of a few percentage of P_2O_5 to silicate glass compositions, promotes the volume nucleation and glass - ceramic formation [10]. There is some evidence for precipitation of phosphate crystals which subsequently act as heterogeneous nucleation sites for the subsequent crystallization of the major phases, although the detailed role of P_2O_5 remains to be discussed [11]. Previous studies [12], [13], [14] have shown that the heat - treatment of 45S5 bioactive glass at a nucleation temperature of 550 °C and followed by heating at a crystallization temperature of 680 °C produces a bioactive glass - ceramic containing the sodium calcium silicate [$Na_2Ca_2Si_3O_9$] as main crystalline phase. In all the bioactive glass - ceramics sodium calcium silicate [$Na_2Ca_2 Si_3O_9$ & $Na_2CaSi_3O_8$] is present as main crystalline phase. Crystalline phase of calcium silicate [$CaSiO_3$] is also observed in each CuO substituted bioactive glass - ceramics (C1, C2, C3 and C4). The studied bioactive glass - ceramics did not contain Cu, Fe, Mn and Zn as separate crystalline phases. This can be related to their relatively low content in bioactive glass composition.

4.2 Density Measurements

. The increase of CuO, Fe_2O_3 , MnO_2 and ZnO contents in the base bioactive glass (45S5) leads to an increase its density because of replacement of a lighter element, Si (density = 2.33 g/cm³) with a heavier element, Cu (density = 8.96 g/cm³), Fe (density = 7.86 g/cm³), Mn (density = 7.21 g/cm³) and Zn (density = 7.14 g/cm³). The density of bioactive glass - ceramics are higher than their respective bioactive glasses due to densification.

4.3 Elastic Properties Measurements

In general, the increase in ultrasonic wave velocities (longitudinal and shear) and elastic modulus of glasses are related to the increase in connectivity of the glass network [15], [16]. An increase in longitudinal and shear ultrasonic wave velocities and Young's, shear and bulk modulus of 45S5 bioactive glass due to increase of CuO, Fe_2O_3 , MnO_2 and ZnO contents in it, can be explained by the decrease in the inter - atomic spacing which means that the copper, iron, manganese and zinc ions with octahedral coordination are involved in the glass network as modifiers by occupying the interstitial positions which cause the increase in the average number of the network bonds per unit volume. Therefore, it can be suggested that CuO, Fe_2O_3 , MnO_2 and ZnO modification leads to an increase in the network connectivity of the studied bioactive glasses. A decrease in Poisson's ratio of 45S5 bioactive glass with increasing of network modifiers is attributed to increase the

crosslink density (defined as the number of bridging bonds per cation) of glass network [17].

It is generally accepted that the controlled heat treatment of glasses above the glass transition temperature and crystallisation temperature leads the possible formation of crystalline phases along with the residual glassy phases [18]. The observed increase in ultrasonic wave velocities and elastic modulus, and decrease in Poisson's ratio of bioactive glass - ceramics is mainly due to the densification during thermal treatments.

5 CONCLUSIONS

Addition of CuO, Fe_2O_3 , MnO_2 and ZnO contents in 45S5 bioactive glass increases its density and elastic modulus: Young's, shear and bulk modulus while a decreases its Poisson's ratio. Crystallization of bioactive glasses enhances their density and elastic properties.

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