# Effect of Oxygen Plasma Treatment on Some Physical Properties of Nanoparticles Glass Ionomer Cement Doped With ZnO or TiO$_2$

**Nagy Abdulsamee[1]\*, Mohamed Y Hassaan[2], Osama Rashed[2], Islam E Soliman[3] and Taha M Tiama[4]**

[1]*Professor and Head of Dental Material Department, Faculty of Dentistry, Modern University for Technology and Information, Egypt*
[2]*Professor, Physics Department, Faculty of Science, Al-Azhar University, Nasr City, Cairo, Egypt*
[3]*Lecturer, Biophysics Department, Faculty of Science, Al-Azhar University, Nasr City, Cairo, Egypt*
[4]*Research, Assistant, Knowledge Center, Faculty Of Pharmacy, Misr University for Science and Technology, 6th Oct, Egypt*

**\*Corresponding Author:** Nagy Abdulsamee, Professor and Head of Dental Material Department, Faculty of Dentistry, Modern University for Technology and Information, Egypt.

## Abstract

Glass-ionomer cement (GIC) with the following composition: SiO$_2$, CaO, P$_2$O$_5$ with ZnO or TiO$_2$ nanoparticles (NPs) as additives were synthesized through a quick alkali mediated sol–gel method. The effect of adding ZnO or TiO$_2$NPs on the physical and mechanical properties of the prepared glass ionomer cement was examined and exposed to oxygen plasma at Low Temperature Plasma Technology of glass powder in GIC by means of dielectric barrier discharges (DBD). The obtained glass powder was then used to prepare the GICs. The ideal powder: liquid (P:L) ratio determined to prepare the experimental GICs was equal to 1.5:1 the used liquid was poly acrylic (PAA) acid. The chemical process allows the development of glass powder at 580°C which was the aim of the present study. The powders were characterized by thermal analysis (TG/DSC), X-ray diffraction (XRD), Fourier transforms infrared (FTIR) then was compared to the compressive, diametral tensile strengths and setting time of GICs before and after plasma Treatment. The results obtained showed that the addition of ZnO or TiO$_2$ has enhanced the mechanical characteristics of glass ionomer cement, especially after plasma treatment.

***Keywords:*** *GICS; Sol-Gel; Plasma Treatment; Differential Scanning Calorimetry and Thermogravimetric Analysis; X-Ray Diffraction; X-Ray Diffraction; FTIR; Diametral Tensile Strength; Compressive Strength; Setting Time*

## Introduction

Although GIC has been extensively used for several decades as a regenerated material in dentistry, e.g. in dental fillings, their physical properties were only improved from the early 1980s, thus making these materials more applicable as well as popular [1]. Their obvious safety and good biocompatibility led researchers and clinicians to consider them for use in different surgical applications as bone cements such as repair of cerebrospinal fluid fistulas and skull defects [2].

However, the presence of aluminium in the glass structure of commercially available GICs has confined with orthopaedic uses. Unfortunately, it was discovered that these aluminium ions causes defects in bone mineralization [3]. and has been implicated in the pathogenesis of degenerative brain diseases such as Parkinson's and Alzheimer's diseases [4,5] and that the release of aluminium from GICs used in reconstructive neurosurgery is considered as the main cause of one case of sub-acute fatal encephalopathy [6]. Noticeable efforts have been made to prepare aluminium-free glass composition with numerous filler components to improvements and enhancements of their physical and mechanical properties for use in GICs. These filler components have been added with optimum levels including; silver-

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amalgam particles, spherical silica, zirconia, glass fiber, hydroxyapatite, bioactive glass particles as pre-reacted glass ionomer particles to overcome the aforementioned drawbacks of GICs [7,8].

A dielectric barrier discharge (DBD) is an electrical discharge between two electrodes separated by an insulating dielectric barrier. The DBD discharges are predestined for applications of large volume plasma chemistry. Barrier discharges, known also as silent discharges, are characterized by the presence of at least one insulating layer between two planar or cylindrical electrodes connected to an AC power supply [9]. The main advantage of this type of electrical discharge is that non-equilibrium plasma conditions in atmospheric pressure gases can be generated in a more economic and reliable way. This feature has paved the way to a number of important DBD applications, including plasma-chemical vapor deposition, surface activation [10], and more recently in surface modification of diverse materials [11].

PAA reacts with the basic TiO$_2$ and ZnO forming a cross-linked metal polyacrylate salt containing residual TiO$_2$ and ZnO particles. These GICs are set at body temperature without undergoing any polymerization shrinkage and also without significant evolution of heat. Over 20 years of use, the excellence of GICs biocompatibility in the mouth in addition to its harmless effect, the development of glass-ionomer cement has become of greater concern in scientific research [12].

Since zinc is considered the second most prevalent trace element in the body and is essential for proper cellular and immune function. It is known that the body needs zinc to metabolize carbohydrates, fats, proteins, alcohol and to dispose of carbon dioxide. In addition, zinc is an essential trace element in the human body and has a stimulatory effect on bone formation. Human bone contains 0.0120 - 0.0250 wt % Zn, which is relatively high compared with the average Zn content of whole fat-free adult tissues (0.0030 Zn wt%) and that of plasma (0.78 - 1.0 Zn mg/L) [13,14].

On the other hand, soft chemistry has been used for synthesizing glasses because this route produces more homogeneous materials using lower processing temperatures than the conventional fusion method. Also, the sol–gel process has the potential to yield glasses which cannot be otherwise prepared by the conventional melting method since their melting points are high [15]. Considerable efforts have been exerted to improve the properties of GICs using other types of glass powders derived from calcium-silicate systems which have new components. The aim of this study was to prepare glass powder at 580°C and to investigate the physical and mechanical properties of glass ionomer cement GIC supplemented with TiO$_2$ or ZnO nanopowder at 0%, 5% and 10% (weight percent) before and after plasma treatment on its surface.

## Materials and Methods

Tetraethylorthosilicate (TEOS), calcium nitrate tetrahydrate Ca(NO)$_3$·H$_2$O and triethyl phosphate (TEP) (≥ 98%) were purchased from Fluka (Buchs, Switzerland). Ammonia solution, 33%, and nitric acid, 68%, were purchased from Merck, USA. Both nitric acid and ammonia solutions were diluted to 2 M using distilled water.

### Sol-gel Synthesis of ZnO and TiO$_2$- doped Glass Ionomer

Glass containing 0 wt% of ZnO and TiO$_2$ samples were synthesized through a quick alkali-mediated sol–gel technique, as shown in table 1 [16]. The solutions were made from mixing TEOS, distilled water and 2M nitric acid (as a hydrolysis catalyst) together in ethanol, and then the mixture was left to react for 30 min under continuous magnetic stirring for the acid hydrolysis of TEOS. After that, appropriate amounts of series reagents were added in the following sequence: (TEP) and Ca (NO)$_3$·H$_2$O, allowing 30 min for each reagent to react completely. After the final addition, the mixture of all reagents was left for 60 min to complete hydrolysis. Ammonia solution of 2M concentration (a gelation catalyst) was dropped into the mixture. The mixture was then manually agitated with glass rod (as a mechanical stirrer) to prevent the formation of a bulk gel. Finally, each prepared gel was left to dry at 100 - 120°C for 2 days and sintered at 580°C for 2 hr in thermal oven. ZnO and TiO$_2$ nanoparticles were added to the glass ionomer cement powder by mixing in a vortex for five minute.

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*Table 1:* Shows the nominal compositions and codes of the prepared bioactive glass.

| Glass | Glass base | | | Additives wt% | |
|---|---|---|---|---|---|
| | SiO$_2$ | P$_2$O$_5$ | CaO | TiO$_2$ | ZnO |
| ZT$_0$ | 60 | 5 | 35 | 0 | 0 |
| TiO$_2$ 5% | 60 | 5 | 30 | 5 | 0 |
| TiO$_2$ 10% | 55 | 5 | 30 | 10 | 0 |
| ZnO 5% | 60 | 5 | 30 | 0 | 5 |
| ZnO 10% | 55 | 5 | 30 | 0 | 10 |

### Cement Preparation

The powder prepared by the sol–gel process was passed through a sieve with a mesh opening of 45 μm, and then was used to produce the cement. The experimental GICs were prepared at room temperature by mixing the powder prepared by the sol–gel process with aqueous solutions 45 - 50% (m/m) of poly (acrylic acid) – PAA – MW 230,000. The specimens were made using the following to a powder: liquid ratio (P:L) 1.5:1 [17].

### Plasma Treatment

The schematic diagram of dielectric barrier discharge (DBD) cell used for treatment of experimental GICs powder is shown in figure 1. The DBD cell was consisted of two parallel plate electrodes. The upper electrode was Al- sheet of dimensions 25 × 25 cm$^2$ pasted on dielectric glass plate of thickness 1.5 mm and the lower electrode was stainless steel plate of dimensions 35 × 35 cm$^2$.
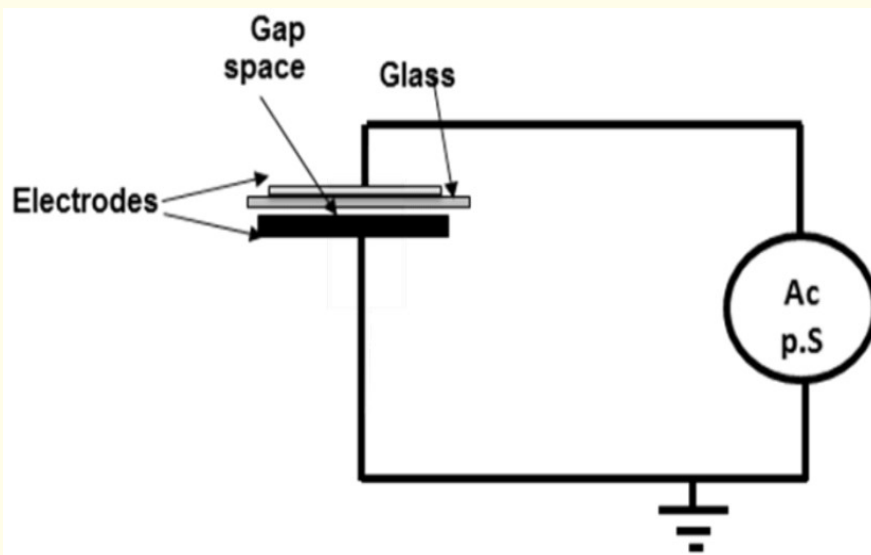


*Figure 1:* Schematic diagram of the DBD Experiment.

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The gap distance between the dielectric glass plate and lower electrode was 2 mm. Plasma discharges were operated in open air under atmospheric pressure and generated by a 15 kV/30 mA AC power supply of 50 Hz frequency. The two electrodes were connected to the high-voltage AC power supply. The powder was placed in the gap between two electrodes. Glass ionomer cement can be treated in this discharge at discharge current is 30 mA with exposure time for 30 min.

## Characterization

Thermogravimetric analyses (TGA), and differential calorimetric analyses (DSC) were performed for the dried gels using a computerized SETARAM labsys™ TG-DSC thermal analysis system. Scans were performed in the atmosphere, and in a temperature range of 50 - 1000°C, at a rate of 10°C min$^{-1}$. The materials were analyzed using aluminum oxide powder as a reference. The phase analysis of the samples was examined by X-ray diffractometer; model BRUKERaxs using Ni-filtered CuKα irradiation at 40 kV and 25 mA. The infrared spectra of the prepared glass were obtained using Fourier transform infrared spectrophotometer (FTIR) (Model 580, Perkin-Elmer). Each sample used for infrared spectroscopic analysis was prepared according to standard procedure by mixing about 2.00 mg of powder sample with 200 mg of KBr, which was subsequently pressed into pellet in an evacuated die.

## Physical Properties

### Diametral compressive

An alternative method of testing brittle materials, in which the ultimate tensile strength of a brittle material is determined through compressive testing, is popular because of its relative simplicity and reproducibility. The method is described in the literature as the diametral compression test for tension or the Brazilian method. In this test, a disk of the brittle material is compressed diametrically in a testing machine until fracture occurs, as shown in figure 2. The compressive stress applied to the specimen introduces a tensile stress in the material in the plane of the force application of the test machine because of the Poisson effect. The tensile stress (σx) is directly proportional to the applied force (F) applied in compression through the following formula:

$$\sigma x = 2F / \pi DT,$$

Where: F = load applied; D = diameter of the cylinder; T = thickness of the cylinder; π is a constant and equals 3.14 [18].
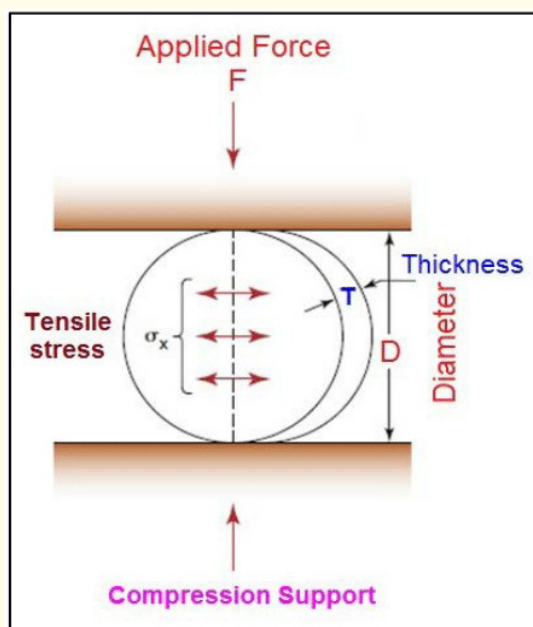


*Figure 2: Schematic illustration of Diametral Tensile Strength.*

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The specimens were made at room temperature of 23 ± 2ºC and relative air humidity of 50 ± 10%, as recommended by ADA specification # 66 [19]. After mixing, the materials were inserted with a Centrix syringe (Centrix, Shelton, USA) into metallic matrices, which were previously coated with a thin layer of petroleum jelly (Sidepal, Guarulhos, Brazil). The insertion was done slowly to adapt the material into the matrix and avoid bubble formation. The matrices were slightly overfilled with the GIC; a polyester strip (Proben, Catanduva, Brazil) covered with a thin layer of petroleum jelly was placed on the material and a cover slip was placed on top of it. Hand pressure was then applied for 20 seconds while excess material was extruded from the top of the matrices for DTS test which considers specimens with 8 mm in diameter and 2 mm in diameter.

**Compressive strength**

Compressive strength (CS) of specimens was performed by the universal testing machine at cross speed of 1 mm/min (MPa), as shown in figure 3 where the specimens were placed in vertical position, with force incident on the long axis. The CS was calculated by the following formula:

$$CS = P/\pi\, r^2.$$

Where: P= load at fracture; r = the radius of sample cylinder; and π = (constant) 3.14.
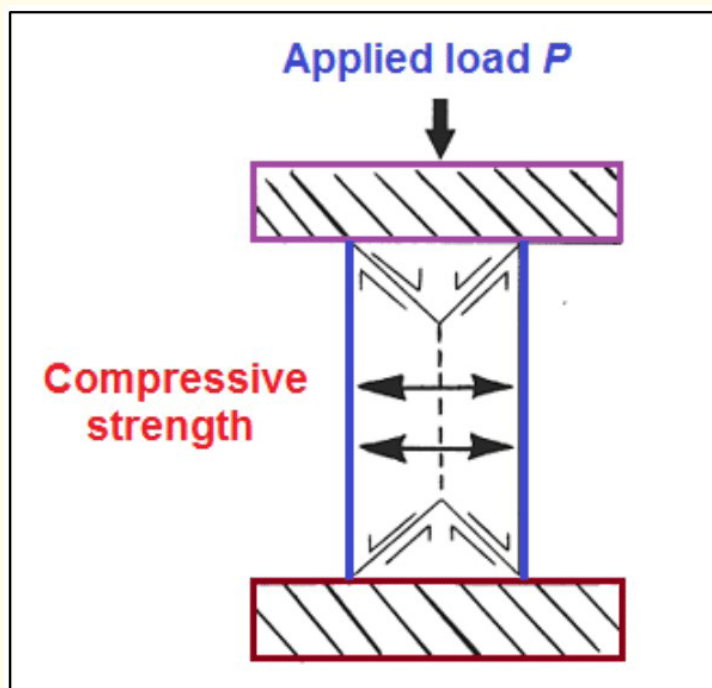


*Figure 3: Schematic Illustration of Compressive Strength.*

For CS test matrices were compressed in a device. Two minutes after the start of the mix, the matrices were placed in an oven at 37 ± 1ºC and 95 ± 5% relative humidity, for 15 minutes. Then, the specimens were ejected from the matrices and the excess material was removed with a carver and petroleum jelly was applied to protect the GIC during the initial setting reaction. This considers specimens with 4 mm in diameter and 6 mm in height [20].

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Finally, One-way analysis of variance (ANOVA) was used for the statistical analysis of compressive strength between the samples. Mean values and standard deviations were estimated. A (P) values < 0.05 were considered to be statistically significant.

**Setting time**

The setting time as defined by the IS0 standard is taken as being the time after mixing at which a standard flat-ended indenter fails to make a complete indentation in the cement at 37°C. A penetrometer was constructed that would fit into a laboratory cabinet in order to the measurements could be carried out at 37°C and at least 90% humidity. Each capsule of cement was mixed at room temperature (23°C + 1) for 10.0 s in the Silamat mixing device and then extruded into a circular metal mould of 10.0 mm diameter and 2.0 mm depth; this size of cavity was adequately tilled by one capsule of each cements. Once a level surface had been produced the mould was placed on to the platform of the penetrometer in the humidity cabinet. One and a half minutes after the start of mixing the indenter (1.0 mm in diameter) was gently lowered onto the surface of the cement and allowed to remain there (under a load of 400g) for 5s. It was then lifted and wiped if necessary. For the first capsule of cement this was repeated every 30s until no indentation was created in the surface of the material-in this way an approximate setting time was calculated. Three further capsules of cement were then tested in the same manner except that in the region of the setting time the penetrometer was lowered onto the cement every 10 seconds [21].

## Results

### Thermal analysis

Differential scanning calorimetry and thermogravimetric analysis (DSC-TGA) were used to determine the transformation temperatures and the mass losses of the samples. DSC-TGA curves of the gel-derived control sample before stabilization up to 1400°C. The DSC curve was found to contain both endothermic and exothermic peaks. The first endothermic peak is at around 60°C - 180°C and the TGA graph shows around 21% weight loss around this peak; therefore it can be concluded that this peak corresponds to the removal of the absorbed water and the silanols in the dried gel. The other peak at 380°C with 15.8% weight loss was due to the water removed from further condensation of precursors and catalysts.

Endothermic peaks approximately at 1300°C correspond to melting of the glass around that temperature. Also exhibits two endothermic and two exothermic peaks in the 600°C - 1264°C range, which plays a significant role in the crystallization of the gel-derived. The TGA curve shows low amount of weight loss (~12%) around this range of temperature are shown in figure 4.
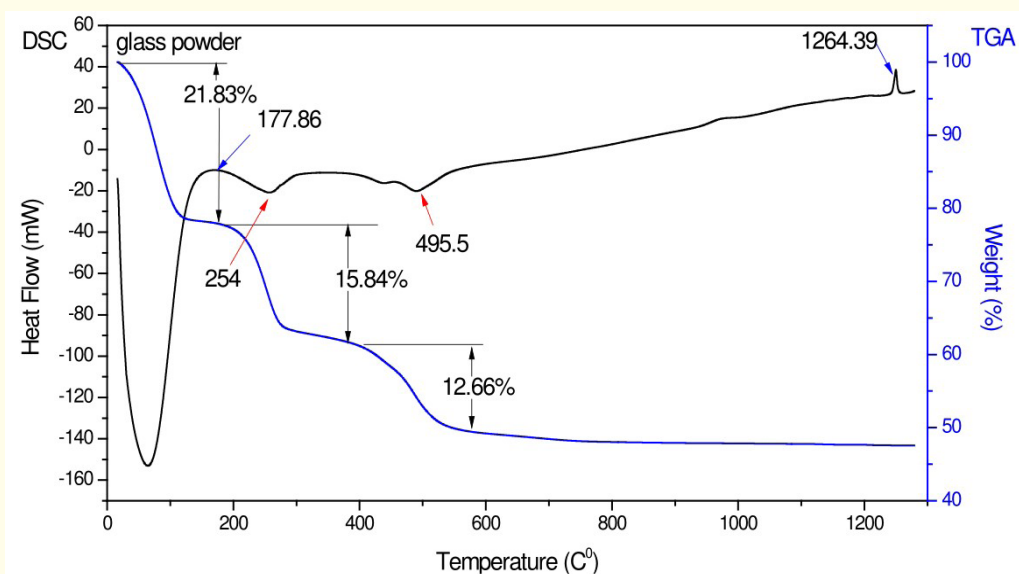


***Figure 4:*** *Differential scanning calorimetry and thermogravimetric analysis (DSC-TGA) curves of non-stabilized control sample prepared by sol–gel method.*

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On the other hand, exothermic peaks represent phase transformation and crystal formation occurrences shows that stabilization at higher than 500°C temperatures is needed to remove all nitrogen-rich compounds out of the sample.

### X-ray Diffraction Analysis

**Phase Analysis (XRD):** The pattern of X-ray diffraction from glass ionomer powder sample is shown in figure 5 (before compounding with the polymer liquid). As observed from the figure, no apparent peak was detected in the pattern of X-ray diffraction. This was an indication of am orphic and glassy structure of the produced glass ionomer powder [22,23].
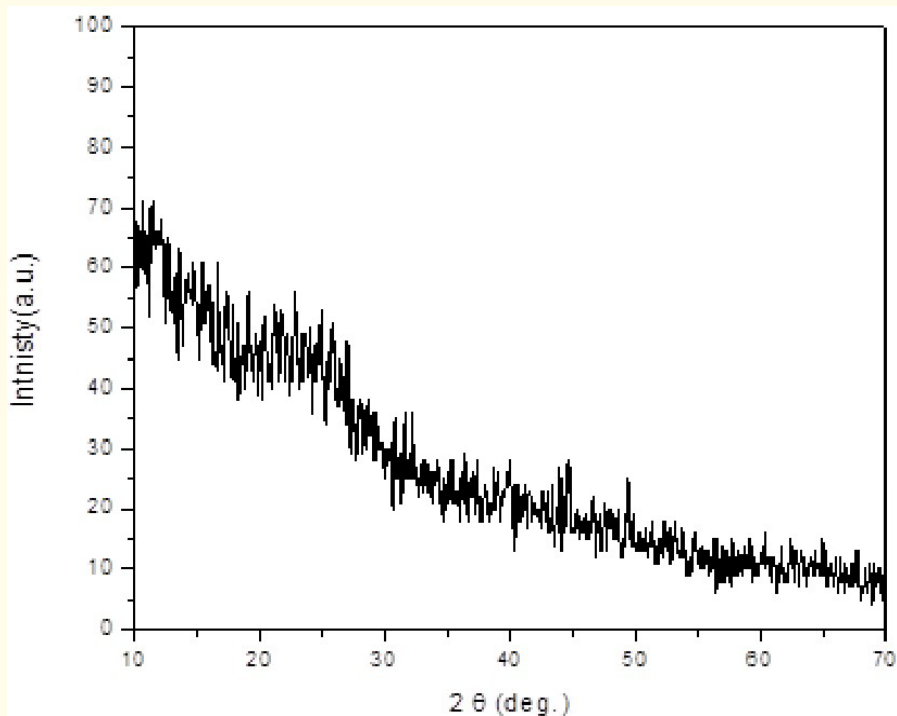


***Figure 5:*** *Powder X-ray diffraction (XRD) patterns of the sol–gel derived for control sample.*

### FTIR Analysis

The FTIR spectra of the glass powder were used to characterize chemical structure of silicate network for the sample shown in figure 6. Sample characterization is possible as specific chemical bonds will absorb in varying intensities and at varying frequencies.
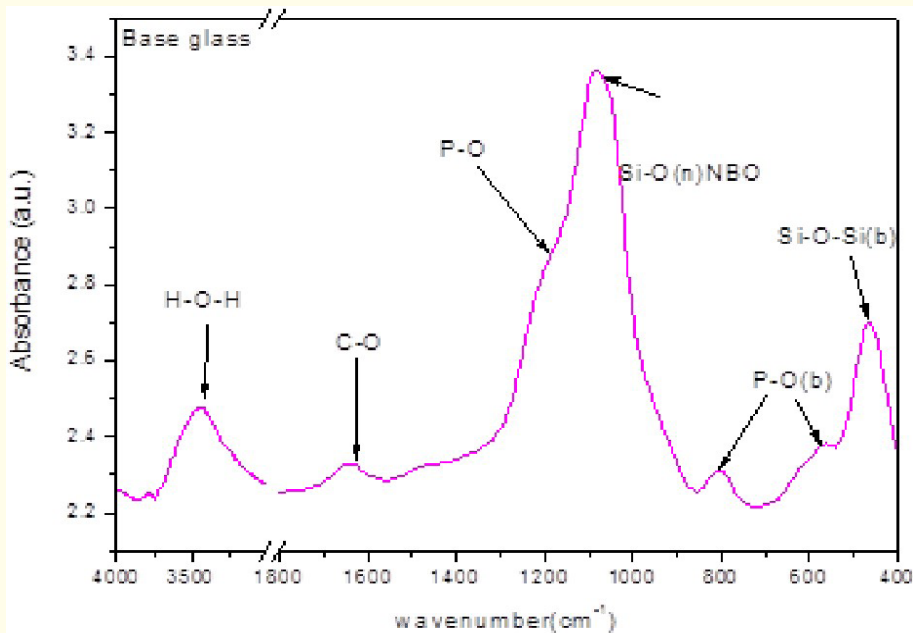
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*Figure 6: FTIR spectra of control sample after calcined at 580°C.*

As for the sample before adding PAA, it was found that the vibration bands occurred at 465 cm$^{-1}$ assigned at Si–O–Si bending mode, and the shoulder at 1064 cm$^{-1}$ is related to the Si–O–Ca vibration bend [24]. The vibration band at 1180 cm$^{-1}$ and a double at 807,575 cm$^{-1}$ is associated with the stretching vibration of phosphate groups [25,26].

**Diametral Tensile Strength (DTS)**

In this test, a compressive force is applied to a cylindrical specimen across the diameter by compression plates. While the stresses in the contact regions are indeterminate, there is evidence of a compressive component that hinders the propagation of the tensile crack. Large shear stresses that exist locally under the contact area may also induce a shear failure before tensile failure at the center of the specimen.

The Diametral Tensile Strength of the GIC was measured. Statistical analysis showed that DTS of glass ionomer cements with and without NPs addition, before and after plasma treatment were significantly different ($P < 0.05$), as shown in table 2.

*Table 2: Mean Diametral Tensile Strength (DTS) of GIC's in MPa and standard deviations (SD).*

| Glass-Ionomer Cements | Before Plasma Treatment | After Plasma Treatment |
|---|---|---|
| Control | 5.123768 (0.97) | 6.659324 (0.81) |
| TiO$_2$ 5% | 6.092432 (0.64) | 8.654782 (0.67) |
| TiO$_2$ 10% | 7.043794 (0.76) | 10.025758 (0.86) |
| Zn O 5% | 7.0650344 (1.42) | 9.325415 (0.92) |
| Zn O 10% | 8.914782 (0.97) | 13.067122 (0.95) |

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The cements based on the plasma treatment had higher strengths in both modalities. The specific surface area of the GIC caused difference [27], because the PAA used to fabricate the cements was of the same molecular weight and concentration, and the particle size of the two glasses was almost identical. The interfacial surface area between glass particles and PAA per unit volume increases using plasma treatment glass, because the plasma treatment has produced a GIC that exhibits higher specific surface area due to increases surface roughness than the untreated plasma one, [28] as shown in figure 7 (a, b).
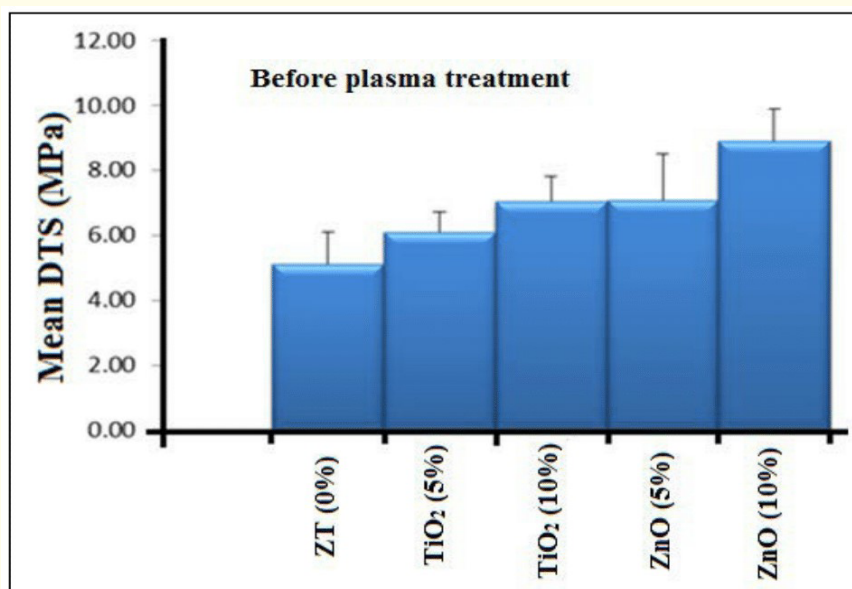


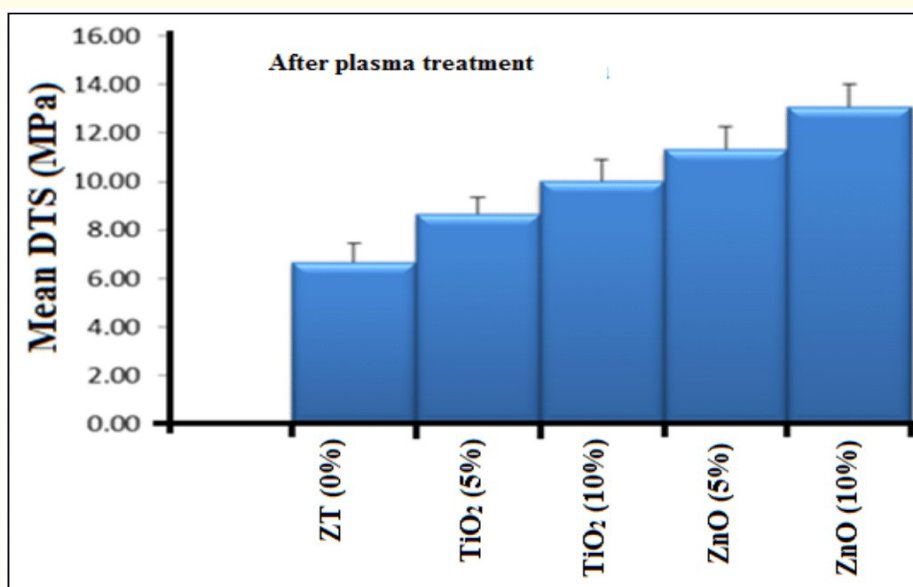***Figure 7a:*** *Change in DTS of GICs before treated plasma.*



***Figure 7b:*** *Change in DTS of GICs after treated plasma.*

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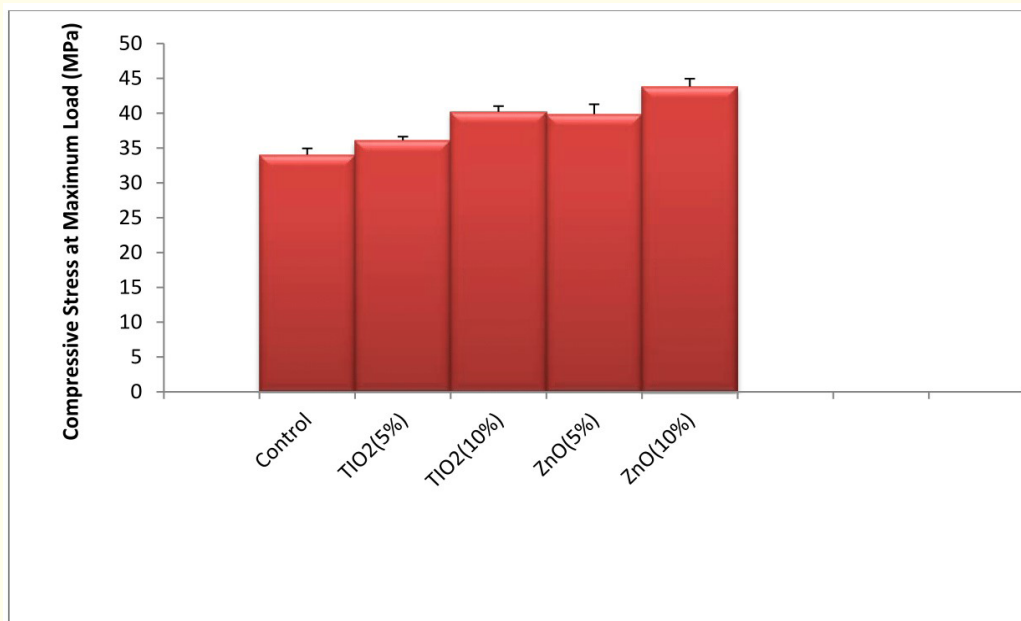
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**Compressive Strength (CS)**

Table 3 shows the compressive strength (CS) of the GIC with and without TiO$_2$ or ZnO NPs addition and also before and after DBD plasma treatment. Statistical analysis showed that CS of all types of GIC were significantly different (P < 0.05). As it can be observed, compressive strength increases in GIC containing 5% and 10% (w/w) TiO$_2$ and ZnO NPs than zero wt%. It may be attributed to the small sizes of the TiO$_2$ and ZnO particles supplemented into the glass powder and the presence of the NPs can occupy the empty spaces between the larger GIC glass particles and act as additional bonding sites for the polyacrylic polymer; this means that the base cement did not incorporate particles because of the small size particles and greater surface of TiO$_2$ and ZnO NPs compared to those of the glass [17].

*Table 3:* Mean Compressive Strength (CS) of GIC's in MPa and standard deviations (SD).

| Glass-Ionomer Cements | Before Plasma Treatment | After Plasma Treatment |
|---|---|---|
| Control | 34.03264 (0.91) | 39.241324 (1.53) |
| TiO$_2$ 5% | 36.092432 (0.54) | 41.354252 (0.67) |
| TiO$_2$ 10% | 40.197127 (0.82) | 45.025758 (0.48) |
| ZnO 5% | 39.865034 (1.42) | 49.645421 (0.92) |
| ZnO 10% | 43.807122 (1.14) | 56.217142 (1.70) |

The cements based on the plasma treatment had higher compressive strengths in both modalities, the difference might be caused due to increasing in degree of cross linking of the polymer molecules within the TiO$_2$ and ZnO-GIC composites after plasma treatment [27]. as shown in figure 8 (a, b).



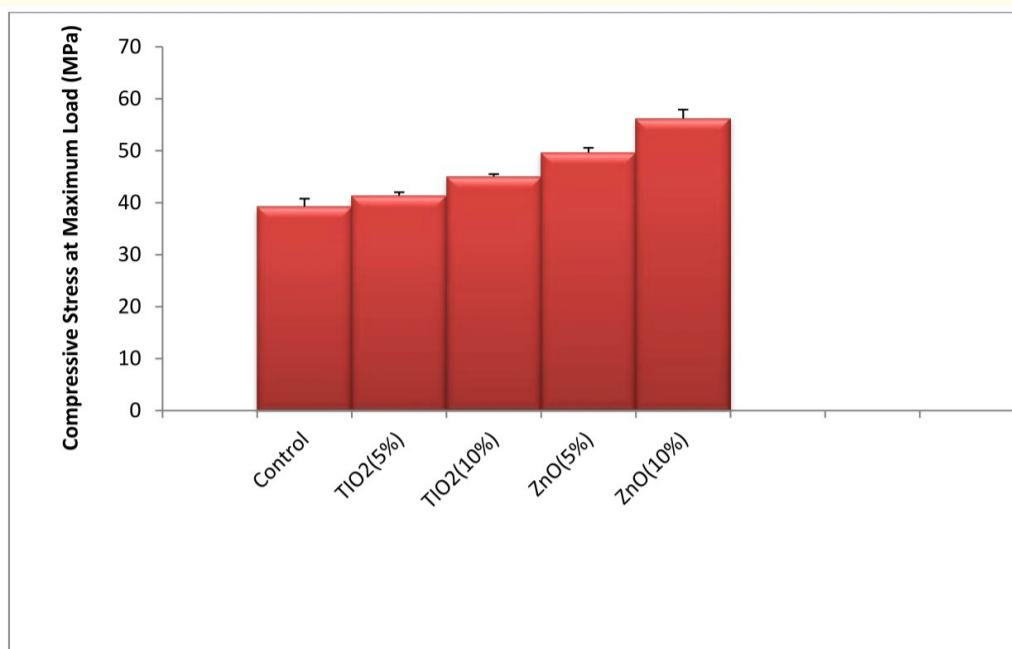*Figure 8a:* Compressive strength of GICs before plasma treatment.

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**Figure 8b:** *Compressive strength of GICs after plasma treatment.*

**Setting time**

The results of the setting time are shown in table 4 and figure 6. The latter is a comparison of the two cases treated by plasma and untreated. A steady increase in the setting time of the untreated GICs is demonstrated (Figure 9).

**Table 4:** *Setting time of the resulting GICs with treated by plasma and untreated.*

| GICs samples | Before plasma treatment | After plasma treatment |
| --- | --- | --- |
| TZ 0% | 8.032642 (0.69) | 5.99552 (1.034) |
| ZnO 5% | 9.97127 (0.90) | 7.010864 (1.15) |
| TiO$_2$ 5% | 7.010864 (0.54) | 5.643794 (0.15) |
| ZnO 10% | 10.80714 (1.81) | 7.860814 (0.68) |
| TiO$_2$ 10% | 6.425758 (0.82) | 4.723768 (0.58) |
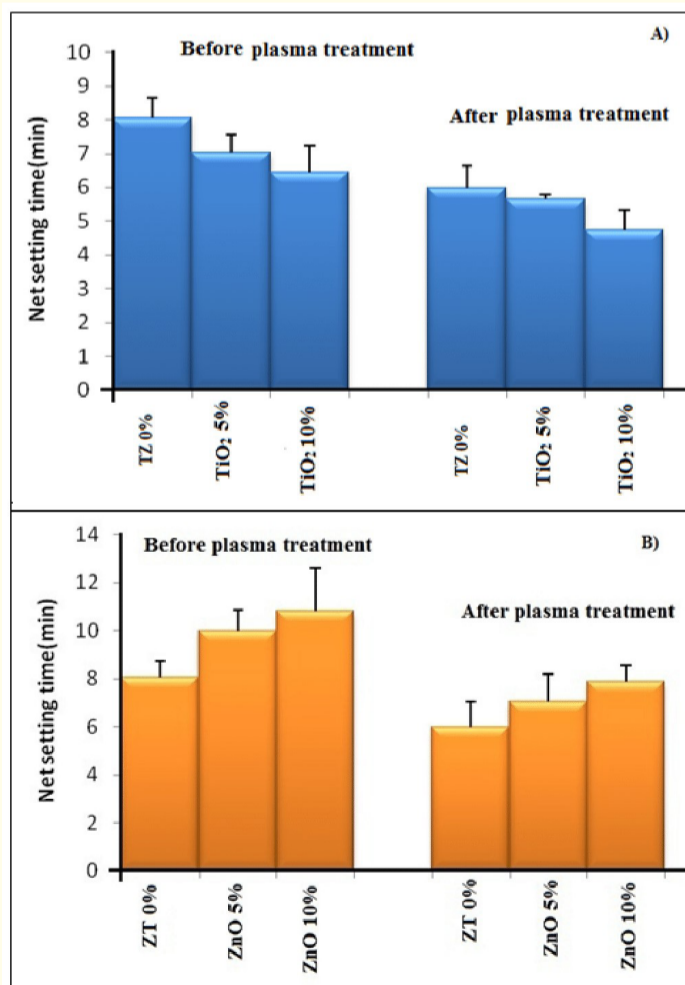


**Figure 9:** *A comparison of setting time for TiO2 (A) and ZnO (B) before and after plasma treatment.*

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Generally, the setting time for all three GIC compositions with untreated much higher than GICs treated by plasma. With a constant amount of polymeric solution a decrease in the setting time for all three GIC compositions was observed (Figure 9). This variation in setting time may be due to the room temperature during the experiments.

## Discussion

In this work the primary aim was to investigate the effect of Plasma Treatment on the Physical Properties of ZnO and TiO$_2$ Containing glass ionomer Cement Prepared by a Quick Alkali-mediated sol–gel Method.

### Thermal analysis

Thermogravimetric analysis (TGA) and differential scanning calorimetric (DSC), analysis curves for sample (Zn$_0$Ti$_0$) are shown in figure 4. The TGA curves of the sample showed three main stages of weight losses as the heating process proceeded from room temperature up to 1200°C. Those weight losses appeared at the temperature intervals of 20 - 180, 180 - 380, 380 - 575, and 575 - 1200°C for control sample. The first weight loss was attributed to the removal of water in the form of (humidity and physically adsorbed water) from the surface and any the residual alcohol in the pores of the dried gel. The second weight loss was reflected in the exothermic peaks centered on at 380°C on the DSC curve of glass sample. The third weight loss occurred from the end of the second drop in mass around at 575°C.

### X-ray Diffraction Analysis

According the Phase Analysis (XRD) for glass ionomer powder, the results indicate the formation of a glass structure n glass cement amorphous state. The obtained results were in conformity with the reported results by [22,23].

### FTIR Analysis

The FTIR spectra of the base glass sample after drayed at 580°C. The broad band sat 3550 cm$^{-1}$ are due to stretching vibrations involving H-O-H groups. The peaks at 1160, 1070 and 795 cm$^{-1}$ are characteristic of a silica network; the band sat 1180 $cm^{-1}$ is a scribed Si-O (s) components of the a symmetric stretch of the SiO$_4$ unit, and the feature at 635 cm$^{-1}$ is due to the symmetric stretch of the SiO$_4$ unit. The assignment of the wave length at 568 and 807 cm$^{-1}$ region of the phosphorus bonded P-O (b), also band at 1101c$^{-1}$ for P=O(s). The sharp weak bands at 486 and 455 cm$^{-1}$ are due to Si-O-Si (b)(r) Symmetric bending vibrations, additionally, for silicate glasses modified by the incorporation of alkali or/and alkali-earth elements, the shoulder at $\sim$ 870 - 1040 cm$^{-1}$ related to the stretching modes Si–O with non-bridging oxygen per SiO$_4$ tetrahedron (Si–O–NBO) can be observed [29].

### Diametral Tensile Strength (DTS)

There are several clinical indications for the use of glass ionomer cements, such as chemical bonding to the dental substrate, biocompatibility, and fluoride release. However, some of these indications are limited by their mechanical strength. Several ionomer materials have been developed in an attempt to enhance their mechanical properties, a fact that justifies the constant research effort that has been made to assess the intended improvements. Therefore studying the effect of plasma treatment of the prepared glass ionomer cement by the sol-gel technique upon its mechanical properties was performed in the current work.

The reason of determining the diametral tensile strength was that glass ionomer is a brittle material i.e. have high compressive strength but with low tensile strength. Rupture under low tension characterizes brittle materials, susceptible to brittle fracture. In these cases, tensile strength is not indicated to evaluate material reaction to tensile stresses, because of the low cohesive condition. An alternative method of tensile strength is calculated indirectly by compressive testing. It is a relative simple and reproducible test. It is defined as diametral compression test for tension or indirect tension [30-32].

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The cements based on the plasma treatment had higher strengths in both modalities. The specific surface area and particle size of the glasses caused difference because the PAA used to fabricate the cements was of the same molecular weight and concentration, and the particle size of the two glasses was almost identical. It means that the effect of surface areas greater than that of particle size [33].

This surface roughness results from the impact of ions and free electrons generated from the plasma to the surface of GIC which in turn can form, cross-link, or in the presence of oxygen, react rapidly to form various chemical functional groups on the GIC surface. Polar functional groups that can form and enhance bond ability include carbonyl (C=O), carboxyl (HOOC), hydro peroxide (HOO-), and hydroxyl (HO-) groups. Even small amounts of reactive functional groups incorporated into polymers can be highly beneficial to improving surface characteristics and physical properties [34].

### Compressive Strength (CS)

Cylindrical-shaped specimen dimensions tested in this work were 6 mm height and 4 mm diameter which was within the dimensions allowed by ISO 991711. This ISO stated that dimensions of the samples should have a relation of length to diameter not more than 2:1. This because if this proportion is exceeded, it can result in undesirable bending of the specimen [32]. The GICs powder treated with plasma had higher Cs than untreated plasma. This is due to an increase in the number of cross-linking that have improved the properties of the matrix phase, therefore mechanical and physical properties of the final composite also improved [35]. The higher intrinsic mechanical properties such as diametral tensile strength and compressive strength by higher cross-linking density were also reported by several other authors [36,37].

### Setting time

The oxygen plasma have the capacity to bind or create –COOH and –OH groups on the inert surface of GIC The formation of these groups increases the surface energy and decrease the contact angle values at the same time [38]. This may be refer to increasing diffusion, wettability and may be also causing changes in chemical structure of polymer surfaces which leads to the formation of new bonds on the surface or attachment of oxygen molecules. We suggest that plasma treatment reduces setting time.

## Conclusion

Within the limitations of the present study.it was found that:

1. GICs with and without zinc and titanium oxide NPs were successfully prepared by quick alkali mediated sol-gel method.

2. Dielectric barrier discharge was used to improve the mechanical properties of GIC.

3. Using plasma treatment has a positive effect on improving the physical and mechanical properties of GICs. In addition to improving their mechanical properties by adding oxide nanoparticles such as ZnO or TiO$_2$ NPs, as alternating oxide instead of alumina that has hazard effect on human health.

## Conflict of Interests

The authors of this paper certify that they have no proprietary, financial, or other personal interests of any nature or kind in any product, service, and/or company that is presented in this work.

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