EFFECT OF ADDITIONS SILVER NANOPARTICLES ON RESIN MODIFIED GLASS–IONOMER MECHANICAL PROPERTIES.

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It is desirable to improve the mechanical strength of resin-modified glass-ionomer cement material while maintaining its favorable clinical properties such as fluoride release, tooth bonding and biocompatibility. In this study, nanoparticles of silver powder (NaAg) of 0.1 and 1% by weight percent were added into the resin modified glass ionomer powder. The resin-modified glass-ionomer cement (RMGIC) was used as a control without the addition of silver nanoparticles (NaAg). The effects of silver powder nanoparticles (NaAg) on shear bond strength, flexural strength, compression strength, tensile strength and fracture toughness were measured using a universal testing machine from Lloyd. Recorded values of shear bond strength, bending strength, compression strength, tensile strength in (MPa) and fracture toughness in (MPa.m1/2) were collected, tabulated and statistically analysed. For testing the significance between the means of tested properties of all tested materials, which are statistically significant when the P value ≤ 0.05, one way analysis of variance (ANOVA) and Tukey’s tests were used. The addition of silver nanoparticles (NaAg) to the glass ionomer modified by the resin resulted in significantly higher shear bond strength, flexural strength, compression strength, tensile strength and fracture toughness. These results show that silver nanoparticles (NaAg) added to the resin-modified glass-ionomer cement can be used as a dependable restorative material with improved shear bond strength, flexural strength, compression strength, tensile strength and fracture toughness. The addition of spherical nanoparticles of silver powder (NaAg) to RMGIC powder has been concluded that the shear bond strength, flexural strength, compression strength, tensile strength and fracture toughness increase.

Introduction:
In recent years, the use of amalgam as a restorative has decreased and this can be attributed to public concern regarding the safety controversy. There is now a tendency towards the use of tooth-colored restorative materials especially composite resins.

However, resin composites still have their own significant deficiencies such as relative complicated manipulation technique, non-adhesion to tooth, shrinkage, water absorption and relatively high thermal expansion coefficients.

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Efforts have been made to find an alternative that can eliminate or reduce the disadvantages of amalgams and resin composites\(^5\). Glass–ionomer cements (GICs) have so far been considered a leading candidate\(^9\). GICs have shown numerous unique characteristics that both resin composites and amalgams do not have\(^5,7\). Due to the ability to link with calcium ions in the tooth, these unique properties include direct adhesion to the tooth\(^8\), anticariogenic properties due to release of fluoride\(^9\), due to low thermal expansion coefficients similar to the tooth, thermal compatibility with tooth enamel and dentin\(^3\), minimized microleakage due to low shrinkage at the tooth-enamel interface\(^3\) and low cytotoxicity due to low monomer content incorporated\(^10,11\). Regardless of numerous advantages of GICs, low mechanical strength, brittleness and poor wear resistance have restricted the current GICs for use mainly at certain low stress-bearing sites such as Class III and Class V cavities\(^5,7\). Recent improvements have resulted in materials with sufficient strength to withstand biting forces in both deciduous and permanent teeth in cavities of class I and class II. They are the materials of choice for use with the Atraumatic Restorative Treatment (ART) technique\(^12\). More recently introduced, resin-modified glass ionomer cements (RM-GIC) claim to improve the mechanical properties of GICs\(^13\).

The light-cured resin modified glass-ionomer cements are hybrid materials prepared by the incorporation of photopolymerizable components into a conventional acid-base mixture. They consist of a complex mixture of components, and may include: Poly acrylic acid or a modified poly acrylic acid, a photocurable monomer such as hydroxyethyl methacrylate, HEMA, or a photocurable side chain grafted onto the photopolymerizable components into a conventional acid-base mixture. They consist of a complex mixture of components, and may include: Poly acrylic acid or a modified poly acrylic acid, a photocurable monomer such as hydroxyethyl methacrylate, HEMA, or a photocurable side chain grafted onto the photopolymerizable components into a conventional acid-base mixture. These nanoparticles release silver ions, damaging transmembrane electron transportation and preventing DNA replication\(^21-23\). On the other hand, several studies have demonstrated that silver nanoparticles are non-toxic to both animals and humans cells\(^24-26\). Indeed, nowadays silver nanoparticles are being applied as antimicrobials incorporated in biomaterials and food packaging, and in coatings for water filters, washing machines, refrigerator and air purifiers, in order to reduce surface microbial biofilm formation\(^21-23\).

Silver is a metal known for its broad spectrum of activity against bacteria, fungi and certain viruses and in the form of nanoparticles its properties are considerably enhanced\(^19,20\). Silver nanoparticles have affinity with molecular groups that contain sulfur and phosphorus, which are found in bacterial membranes and within bacterial cells. These nanoparticles release silver ions, damaging transmembrane electron transportation and preventing DNA replication\(^21-23\). On the other hand, several studies have demonstrated that silver nanoparticles are non-toxic to both animals and humans cells\(^24-26\). Indeed, nowadays silver nanoparticles are being applied as antimicrobials incorporated in biomaterials and food packaging, and in coatings for water filters, washing machines, refrigerator and air purifiers, in order to reduce surface microbial biofilm formation\(^21-23\).

The objective of this study was to evaluate the shear bond strength, flexural strength, compression strength, tensile strength and fracture toughness of nanoparticles of silver (NAg) added to resin-modified glass ionomer cements (RM-GIC).

**Material and Method:**

Resin-modified glass–ionomer cement (RMGIC) (3M Dental Products, St. Paul, MN, USA) was used as the control. The powder was 100% fluoro-alumo-silicate glass; the liquid was composed of polyalkenoic acid and a mixture of other acrylates and light sensitive catalyst. The resin-modified glass ionomer powder was added nanoparticles of silver powder (NAg) of 0.1, 0.5 and 1 by weight percent.

**Grouping of Specimens:**

The specimens were divided into 4 main groups of 30 specimens for each laboratory test (bond strength, flexural strength, compression strength, tensile strength and fracture toughness). Each group was subdivided into 3 subgroups of 10 each specimens as following: Group A: RMGIC powder without any additives (control group), Group B: RMGIC powder with 0.1 wt % NAg, Group C: RMGIC powder with 1 wt % NAg.

**Shear bond strength:**

**Mold construction:**

A specially designed split Teflon mould was manufactured to form the 3 mm diameter and 2 mm thick RMGI disk sample. The mold consisted of a metal ring that held two halves of Teflon together. These dimensions have been
Specimen preparation:
Non carious human molars were selected freshly extracted. The study included only sound enamel free of cracks or staining. Saline solution used to store the extracted teeth to obtain buccal and lingual surfaces, they were sectioned mesio distally. Each surface was embedded in self curing acrylic resin using a 2 cm long Teflon mold, 1 cm wide and 1 cm thick, so that the external surface could be exposed. The blocks were stored in normal saline until they were used after polymerization was completed before application of RMGIC, each surface was treated with a cavity conditioner for 10 seconds according to the manufacturer's instructions, rinsed with water, air sprayed and then the mold was centralized into the surface and Resin modified glass ionomer cement (RMGIC) is packed in the mold using a plastic spatula, a celluloid matrix has been applied to the material in order to obtain a smooth finished surface. The Curing time for RMGIC materials was 40 seconds using visible light Curing unit (light emitting diodes) (LED) according to the manufacturer's instructions. The celluloid matrix was removed after Curing and the Teflon mold was carefully split and removed. After the mold was removed, side curing was carried out to ensure proper polymerization. The acrylic blocks were stored at 37°C for 24 hours in distilled water. Radiometer Curing equipment used to ensure stable light intensity during the polymerization of all specimen.

Shear bond strength testing:
Shear bond testing was done using a universal testing machine (Lloyd mechanical testing machine) at a cross head speed of 0.5mm/min as shown in figure 1.

A specially designed upper attachment for the machine was fabricated with the same dimensions of the RMGIC disc (3mm in diameter and 2mm in thickness) to fit into it.

The blocks were then mounted on the lower attachment and the upper attachment held the RMGIC disc on the testing machine. They were then tested in shear at a crosshead speed of 0.5mm/min until fracture.

Maximum load needed to fracture the bond was obtained in Newton (N).

The shear bond strength in Kg/Cm² was calculated from the equation:

\[ \sigma_s = \frac{P}{\pi r^2} \]

Where:
- \( \sigma_s \): shear bond strength in Kg/Cm²
- \( P \): is the shear load in Kg
- \( \pi = 3.14 \)
- \( r \): is the radius of the specimen in Cm

The shear bond strength was converted to MPa by multiplying the results by 0.09807.

Specimens' preparation for mechanical testing:
Each group was mixed with glass ionomer cement liquid on a glass slab using plastic cement spatula. The mixed cement was condensed in the Teflon mold which was placed on glass plate. Celluloid strips covered the specimens and pressed with another glass plate. All specimens were exposed for 2 minutes to light emitting diodes (LED) (BG-light-LTD, 4002 Plovdiv, 430-490 nm, Bulgaria). After setting the specimens were removed from the mold and stored in distilled water 24 hours Prior testing. Radiometer Curing equipment used to ensure steady light intensity during the polymerization of all specimens (LI-189.Li-Cor Inc, Lincoln, NE68504, USA). Lloyd mechanical testing machine (model LRX plus II. Fareham, England) was used to measure all specimens.
Flexural strength:
Mold construction:
A specially designed Teflon mold was manufactured to form a rectangular specimen with 25 mm length, 5 mm width and 3 mm height dimensions. These dimensions were determined in accordance with the International Organization for Standards ISO No.4049 (2000) recommendation

Flexural strength testing:
Flexural strength was quantified by a three point loading test using Lloyd mechanical testing machine as shown in figure 2. Each specimen was adjusted to the bending attachment consisting of two parallel supports 20 mm apart and the load was applied with a third rod placed centrally between the two support. The specimens were loaded to fracture at a speed of 0.75 mm / min. The maximum force required to fracture the specimens from the stress strain curve was recorded in Newtons. The flexural strength was calculated in (MPa) for all specimens from the following equation:

\[ Q = \frac{3Pl}{2bd^2} \]

Where Q the flexural strength in MPa, (P) is the maximum load at the point of fracture in (N), l is the distance between the supports (mm), b is the width of the specimen and d is the thickness of the specimen.

Compressive strength:
Mold construction:
In accordance with International Standards Organization (ISO) No 9917 (2000), a specially designed Teflon mold of 4 mm in diameter and 6 mm in height was manufactured.
Compressive strength testing:
The specimens were loaded on done using a universal testing machine at a cross head speed of 0.5mm/min as shown in figure (3).

The specimens were placed with flat end vertically between the two metal plates.

The load was applied until the specimen was crushed and the peak force required to fracture each specimen was recorded in Newton from stress strain curve. The compressive strength was calculated in (MPa) using the following equation:

Compressive strength = 4P/πd
Where (P) is the load at the fracture point (N) and d is the diameter (mm) of the specimen.

Fig 3:- A photograph of compressive strength testing

Diametral tensile strength:
Resin-modified glass–ionomer cement is a brittle material that cannot withstand tensile stresses; therefore, an indirect tensile test is used to measure the tensile strength of resin - modified glass - ionomer cement A compressive load is placed on a cylindrical sample diameter in this test. The compressive stress induces tensile stress in the force application plan. The tensile stress is directly proportional to the compressive load in such a situation..

Mold construction:
According to International Standards Organization (ISO) No 7489 (2000), a specially designed Teflon mold with a diameter of 6 mm and a height of 4 mm was manufactured.

Diametral tensile strength testing:
In this test, the disc specimen was mounted on the Lloyd mechanical testing machine and the load was applied to the specimen using a cross head speed of 0.5 mm/min applying a compressive force on the specimen until fracture. The diametral tensile strength was calculated in (MPa) using the following equation:

DTS = 2P/πdt
Where (DTS) is the diametral tensile strength (MPa), (P) is the load (N) at the fracture point, d is the diameter of the specimen t is the thickness of the specimen.

Fracture toughness:
Mold construction:
A specially designed Teflon mold was manufactured to form a rectangular specimen with a length of 25 mm, a width of 5 mm and height of 3 mm. These dimensions were determined by the American Society for Tests and Materials (ASTM Designation) recommendation (E182005, 2005).
Fracture toughness testing:
In the middle of each specimen a notch was made on one edge with 2.5 lengths using a sandpaper disk. The samples were tested by three bending points in a Lloyd mechanical testing machine at a crosshead speed of 2 mm / min, with the notch on the tensile side and the loading pin aligned with the notch. Fracture toughness was determined calculated in (MPa.m^{1/2}) from the following equation:

\[ KIC = \frac{(PQ \cdot S/BW^{1.5})}{f(a/w)} \]

Where KIC is stress intensity factor, PQ is peak load at fracture, S is the distance between supports, a is the notch depth, w is the width of the specimen and B is the thickness of the specimen and \( F(a/w) \) is calculated from the following equation:

\[ F(a/w) = \frac{2 + a/w}{(0.886 + a/w - 13.32 a^2/w^2 + a^3/w^3 - 5.6 a^4/w^4)} \]

The recorded values have been collected, tabulated and analyzed statistically. For testing the meaning between the means of tested properties of all tested materials, which are statistically significant when the P value 0.05, one way analysis of variance (ANOVA) and Tukey's tests were used.

Results:
The comparison between the mean shear bond strength in (MPa) in table (1) and figure (4), the mean flexural strength in (MPa) in table (1) and figure (5), the mean compression strength in (MPa) in table (1) and figure (6), the mean tensile strength in (MPa) in table (1) and figure (7) and the mean of fracture toughness in (MPa.m^{1/2}) in table (1) and figure (8) of RMGIC groups.

RMGIC powder with 1 wt % nanoparticles of silver (NAg) (group C) showed the significantly highest mean shear bond strength, highest mean flexural strength, highest mean compression strength, tensile strength and highest mean fracture toughness followed by RMGIC powder with 0.1 wt % NAg (group B) then RMGIC powder without any additives (control group) showed significantly lowest mean strength.

Table (1): Comparison between the means of shear bond strength in (MPa), flexural strength in (MPa) and fracture toughness in (MPa.m^{1/2}) of (Nag) tested groups.

| Groups                  | Group A | Group B | Group C | P-value |
|-------------------------|---------|---------|---------|---------|
| Shear bond strength (MPa) | Mean  | SD     | Mean  | SD     | Mean  | SD |
|                         | 12.78\(^\text{d}\) | 0.53 | 16.52\(^\text{b}\) | 0.47 | 22.21\(^\text{a}\) | 0.2 |
| Flexural strength (MPa) | 12.31\(^\text{c}\) | 0.83 | 17.75\(^\text{b}\) | 0.87 | 25.75 \(^\text{a}\) | 1.1 |
| Compressive strength (MPa) | 48.52\(^\text{c}\) | 2.18 | 59.08\(^\text{b}\) | 3.55 | 67.78\(^\text{a}\) | 3.76 |
| Tensile strength (MPa) | 4.85\(^\text{c}\) | 0.45 | 5.08\(^\text{b}\) | 0.69 | 7.78\(^\text{a}\) | 0.61 |
| Fracture toughness (MPa.m\(^{1/2}\)) | 0.17\(^\text{c}\) | 0.02 | 0.369\(^\text{b}\) | 0.05 | 0.566\(^\text{a}\) | 0.08 |

* Significant at P ≤ 0.05, Means with different letters are significantly different according to Tukey’s test.

**Fig. 4:** Bar chart of means of shear bond strength in (MPa) of the tested groups.
Fig. 5:- Bar chart of means of Flexural strength in (MPa) of the tested groups.

Fig. 6:- Bar chart of means of Compressive strength in (MPa) of the tested groups.

Fig. 7:- Bar chart of means of tensile strength in (MPa) of the tested groups.

Fig. 8:- Bar chart of means of Fracture toughness (MPa.m$^{1/2}$) of the tested groups.
Discussion:-
Glass ionomer cement is one of the most important dental cements popular for its biocompatibility, bacteriostatic properties and double bonding mechanisms (ionic and micromechanical bonds)\textsuperscript{28}. A contemporary idea to increase the strength and improve the mechanical properties of glass ionomer cements is added to the cement matrix by different nanoparticles.

In this study, instead of conventional glass powder, we added silver nanoparticles (NAg) to RMGI. Nanoparticles were higher in crystallinity and colloidal stability is more effective and easier to apply. Results of this study demonstrated that the incorporation of silver nanoparticles (NAg) to resin glass ionomer cement increased bond strength, flexural strength, compressive strength, tensile strength and fracture toughness compared to resin modified glass ionomer cement.

The results also showed that the increase in the percentage of NAg added to the glass ionomer cement modified by resin from 0.1 percent to 1 percent wt also resulted in an increase in all these mechanical properties tested. In the present study silver nanoparticles (NAg) were incorporated into the resin modified glass ionomer powder with concentrations of 0.1 and 1 by weight percent.

Silver nanoparticles (NAg) have powerful antibacterial properties\textsuperscript{29}. Their small particle size and large surface area could enable them to release more Ag ions at a low filler level, thereby reducing Ag particle concentration necessary for efficacy\textsuperscript{30}. The Ag ions in the resin agglomerated to form nanoparticles that became part of the resin\textsuperscript{31}.

The addition of nanoparticles of silver (NAg) was increased compressive strength, diametral tensile strength and flexural strength. These fillers were able to bond to the matrix by chemical bonding\textsuperscript{32}.

Because the majority of shear bond failures were cohesive failures, the interfacial bond at the interface was stronger than cohesive bond within the material itself.

Flexural forces are generated under clinical situations, and the dental materials need to withstand the repeated flexing, bending, and twisting forces\textsuperscript{33}.

The compressive strength of resin-modified glass ionomer cement containing nanoparticles of silver is much higher. It was due to strong covalent bonds of resin matrix\textsuperscript{34}.

Fracture toughness is a measurement of a material’s ability to resist catastrophic failure and is a better indicator of clinical strength than average stress-based tests\textsuperscript{35}.

Mitchell et al. compared the fracture toughness of conventional, resin-modified glass ionomer and composite luting cements and made the same suggestion stating that fracture toughness is a material property and may be a more reliable parameter to predict clinical performance than compressive or diametral strength measurements\textsuperscript{36}.

Another factor that may influence mechanical properties is the uptake of water. The hydrophobic characteristic of filler might influence surface and grain boundary diffusion, and then increase the resistance of the cement to water uptake\textsuperscript{37}.

Following post-set water uptake, the acidic monomers of RMGIC ionize then react with the filler to initiate an acid-base reaction, producing ionic cross-linking\textsuperscript{38}. In these dual setting systems, resin reinforcement produces higher bond strengths to dental tissues as well as enhanced mechanical strength\textsuperscript{39,40}.

Conclusion:-
This study shows that incorporation of nanoparticles of silver (NAg) to resin glass ionomer cement increased the bond strength, flexural strength, compressive strength, tensile strength and fracture toughness as compared to resin modified glass ionomer cement.

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