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# MECHANICAL PROPERTIES OF THE NEW KETAC™ UNIVERSAL GLASS IONOMER RESTORATIVE MATERIAL: EFFECT OF RESIN COATING

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#### ABSTRACT

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Statement of the problem: Clinical performance and survival rates of posterior high strength GICs are questionable. A new generation of conventional glass-ionomer restorative material (Ketac<sup>TM</sup> universal) without coating has been launched recently for restoring class I and II cavities without investigations.

**Objectives**: This study was conducted to evaluate the mechanical properties of a new generation of conventional glass ionomer (Ketac<sup>™</sup> universal) for restoring posterior stress bearing areas. Furthermore, the effectiveness of resin coating on Ketac<sup>™</sup> universal GIC was evaluated.

Materials and Methods: Three types of conventional glass ionomer restorative materials were selected for this study (Ketac<sup>™</sup> Universal; KU, Ketac<sup>™</sup> Molar; KM and Fuji IX GpFast; FIXF). Each GICs material specimens were prepared according to manufacturer' instructions and distributed randomly into two groups (N=30) (uncoated and coated groups) and then conditioned in distilled waterat 37°C for 24 hours. The flexural strength, compressive strength, and hardness tests for each material were evaluated using a universal testing machine. Furthermore, additional representative un-coated and coated specimens for each material were prepared for their characterization under scanning electron microscope. Data were analysed by multivariate ANOVA. Bonferroni post hoc test was used for multiple comparisons. Paired t-test was used to detect significant differences between un-coated and coated GICs. Interactions between GICs materials and coating were also performed. P-value is significant if it was less than .05.

**Results:** The highest significant flexural strength, compressive strength and hardness values were noticed with Ketac universal and the lowest was noted with FIXF. Also, only significant improvement in the flexural and compressive strength of FIXF when resin coating was applied.

**Conclusions:** KU represents an encouraging line of higher clinical longevity of GICs' filling material in stress bearing areas. Also the resin coating has no significant effect on the tested mechanical properties of KU GICs.

KEY WORDS: Coating- Ketac<sup>™</sup> universal- Mechanical properties

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# **INTRODUCTION**

The recent way to control dental caries involves dental materials which retain both restorative and prophylactic characteristic combining with a satisfactory mechanical property. Conventional GICs although, it has questionable clinical longevity regarding their mechanical properties such as low fracture toughness, hardness and flexural strength which limits their use to the non-stress bearing areas, but it highly recommended because of their fluoride release, rechargability, biocompatibility, and chemical adhesion.<sup>1-6</sup>

Due to the beneficial GICs properties, developments have been carried out to improve their shortcomings by changes in glass particle size and their distribution, improved surface reactivity, and setting reaction. The faster-setting, highviscosity conventional GICs, which called high strength conventional glass ionomer is one of these developments.<sup>7-9</sup> Further developments have been carried out, a self-adhesive coating was known to be one of the suggested method for enhancing the mechanical properties of the highly viscous GICs.<sup>10</sup> In order for saving procedural steps during GICs manipulation, a high-viscosity conventional GIC (Ketac<sup>TM</sup> Molar) with improved the mechanical properties without the need for a coating.<sup>11</sup>

Recently Ketac<sup>™</sup> Universal GIC is the latest progress in glass ionomer technology in the field of restorative dentistry. Saving steps like coating for a faster procedure was the aim of this material evolution. Also, it can provide higher mechanical properties than other glass ionomers which necessitate a coating. Moreover, according to the manufacturer, "it can be used in high stress bearing are as due to the special improved filler composition leading to high mechanical properties even with lower viscosity compared to Ketac<sup>™</sup> Molar glass ionomer restorative".<sup>12</sup>

Compressive strength, flexural strength, and surface micro-hardness are appropriate mechanical tests for predicting clinical performance of glass ionomer cements inside the oral cavity.<sup>8,13-15</sup> For investigation of filler size, distribution and porosity inside the materials, scanning electron microscopic analysis is considered an effective and adequate method.<sup>16,17</sup>

Glass ionomer	Composition	Manufacturer	
Ketac <sup>™</sup> Universal (aplicap). Lot :	Oxide glass, water, copolymer of acrylic acid-malic acid,	3M ESPE Deutschland GmbH	
Ketac <sup>TM</sup> molar (Quick aplicap) Lot	Al-Ca-La fluorosilicate glass 5% copolymer	3M ESPE Deutschland GmbH	
:490576	acid (acrylic and maleic acid), Polyalkenoic acid,		
	tartaric acid, water		
Fuji IX GP Fast(capsule)	Aluminofluorosilicate glass, polyacrylicacid, distilled	GC Corporation	
Lot :1602061	water, poly carboxylic acid	Tokyo, Japan	
Equia Coat	Methyl methacrylate, colloidal silica, camphorquinone.	GC Corporation	
Lot:1501061	(nanofilled self-adhesive light-cure)	Tokyo, Japan	
Single bond universal adhesive	MDP phosphate monomer, dimethacrylate resins,	3M ESPE Deutschland GmbH	
Lot:587885	HEMA, polyalkenoic acid copolymer, filler, ethanol,		
	water, initiators, silane		

TABLE (1) Materials used in the study

Therfore, this study was aimed to evaluate the flexural strength, compressive strength, and hardness of Ketac<sup>™</sup> Universal glass ionomer and to compare the findings with two other types of GICs. Furthermore, the effect of resin coating on Ketac<sup>™</sup> Universal GIC was assessed. The null-hypotheses tested were that, there was no significant difference in flexural strength, compressive strength, and hardness between (1) Un-coated Ketac<sup>™</sup> Universal and uncoated Fuji IX GP fast. (2) Un-coated Ketac<sup>™</sup> Universal and uncoated Ketac<sup>™</sup> Molar. (3)Coated and uncoated Ketac<sup>™</sup> Universal GIC.

## MATERIALS AND METHODS

Two conventional highly viscousGICs; Fuji IX Gp fast (FIXF) and Ketac<sup>TM</sup> Molar (KM) and the new generation of conventional GICs Ketac<sup>TM</sup> universal (KU) were selected for this study. For each material, the corresponding light cured resin coating supplied or recommended by the same manufacturer was chosen. It should be noted that, for Ketac<sup>TM</sup> Universal and Ketac<sup>TM</sup> molar GICs, coating is optional step. The materials used in this study are listed in (Table1).

# Study design

Sixty specimens were prepared for each GICs material according to manufactures' instructions. Each GICs material specimens were distributed randomly into two groups un-coated (control group) and coated groups and then conditioned in distilled waterat 37 °C for 24 hours. Then flexural strength, compressive strength, and hardness tests were evaluated for each material. Furthermore, one representative specimen from each group for each material (un-coated and coated) were prepared for scanning electron microscopic analyses to study the effects of microstructure and resin coating on their mechanical properties.

## **Specimen preparation**

The GICs caspules were inserted in the Aplicap Activator (3M ESPE, St Paul MN, USA). The activator levers were firmly depressed and held down for 2 to 4 second. The capsule was mixed at 4,300 rpm in a Dentomax compact (Degussa -Hul SAG, Germany) for 8 sec. The material was applied with the Aplicap Applier (3M ESPE, St Paul MN, USA), into mold and the excess was removed using sharp #12 bard barker scalpel blade followed by the application of another polyester strip and another glass slab on the mold top with the application of constant load of 200g. After the removal of the glass slab, the material was left to set at room temperature. Polyester strip were removed about 2.5 min after mixing. After setting, the specimens were detached from their mould and a wet 600-grit silicon carbide (SiC) abrasive paper was used to remove the residues then the speciments were kept in distilled water at 37 °C for 24 hours before testing.

As for specimens receiving resin coating, after three minutes of GICs capsule activation, resin coating was applied then light cured for 20s using a LED light (Elipar Free ligh 2, 3M ESPE, 1,226 mW/cm<sup>2</sup>) by three overlapping irradiations.

## **Flexural strength**

Ten bar-shaped specimens (N=10) for each material (25 mm length x 2 mm thickness x 2 mm width) were prepared in rectangular-shaped stainless-steel split mould.

Three-point bending test was used to measure flexural strength in a universal testing machine (LLOYD LRX, LLOYD instruments Ltd., Fareharn, Hampshire, UK) at acrosshead speed of 0.5 mm/ min based on ISO standard.<sup>18</sup> Flexural strength (MPa), was calculated using this formula : FS (ó) =  $3F (L)/2wh^2$  Where F is the maximum load at the point of fracture, L is the distance between the two supports (mm), w is the specimen width (mm) and h is the thickness of specimens (mm).<sup>14</sup>

## **Compressive strength**

According to ISO standard<sup>19</sup>, stainless steel split mould 4 mm in diameter and 6 mm in height was used for cylindrical specimens preparation (N=10). The universal testing machine at a crosshead speed of 0.5 mm/min was used to measure compressive strength according to the following equation<sup>17,20</sup>:  $Cs=P/\pi r^2$  where P is the load (N) at fracture, ( $\pi$ =3.14 constant) and r is the radius of specimen cylinder (mm).

## Surface microhardness

A split Teflon mould 6 mm in diameter and 3 mm in thickness was used to prepare disc-shaped specimens (N=10) for each material. A digital microhardness tester (Micro Met 6040 Wilson Microhardness; BUEHLER, U.S.A.) was used to measure surface micro-hardness utilizing a diamond indenter with 50 g load and a dwell time of 10 s. The Vickers hardness (VHN) for each specimen was calculated using the following equation: VHN = 1854.4 x L/d<sup>2</sup> Where, L is the applied indentation load (kg) and d is the mean indentation diagonal length (mm).<sup>21</sup>

# Scanning electron microscopic (SEM) analysis

One representative specimen from each group for each material (un-coated and coated) was observed under field emission scanning electron microscope (Quanta FEG 450, Amsterdam, Netherland) with original magnification range X100-1000 to study the microstructure and resin coating effects on the mechanical properties of tested GICs. The specimens were vacuum sputter-coated with gold, and a high vacuum (JFC-1600, JEOL, and Japan) was used for the specimens dehydration before SEM analysis.

#### Statistical analyses

Shapiro-Wilk test was used to test the normal distribution of data. The data was parametric and met the normal distribution. Multivariate ANOVA was used to test significant differences of flexural strength, compressive strength and hardness between groups.Post hoc Bonferroni test was used for multiple comparisons. Paired t-test was used to detect significant differences between coated and uncoated GICs conditions. Interactions between GICs materials and resin coating were also performed. P-value is significant if it was less than 0.05.

## RESULTS

Means and standard deviations of flexural strength, compressive strength and hardness of all tested GICs with or without coatare presented in table 2.

## **Flexural strength**

For uncoated GICs, there was a significant difference in flexural strength (MPa) between all GICs (p < 0.001). Multiple comparisons with Bonferroni post hoc test revealed a significant difference between (KU and FIXF) and between (KM and FIXF). However, no significant difference between KU and KM (P=0.73). For coated GICs, significant improvement was noticed for FIXF (p < 0.05), while no significant difference was noted with KM and KU (P=0.057, P= 0.061) respectively. The results of multivariate analysis showed that, influence of interaction between coating and GICs was proven to be significant on flexural strength (F(2,54)= 176.90, p<.001\*).

## **Compressive strength**

There was a significant difference in compressive strength (MPa) between all uncoated GICs (p < 0.001) except between KU and KM (P=0.062). For coated GICs, significant improvement was noticed for FIXF (p < 0.05), while no significant difference was noted with KM and KU (P=0.072, P= 0.082) respectively. The results of multivariate analysis showed that, influence of interaction between coating and GICs was proven to be significant on compressive strength (F(2,54)= 287.50, p<.001\*).

# (1031)

## Hardness

There was a significant difference in hardness values (VHN) between all uncoated GICs (p < 0.001) except between KU and KM (P=0.075). For coated GICs, no significant improvement was noticed for FIXF (P=0.11), KM (P=0.092), and KU (P=0.12). The results of multivariate analysis showed that, influence of interaction between coating and GICs was proven to be non significant on hardness (F (2,54) = .187, p=.83).

## Scanning electron microscopic analysis

SEM images showed: Fuji IX Gp fast has larger diameter of glass particles (Fig.1.A), However, homogenous small glass particles size representing a greater particle surface area was noticed in Ketac<sup>™</sup> Molar and Ketac<sup>™</sup> Universal (Fig. 2. and 3 A). Moreover, nearly similar SEM images between Ketac<sup>™</sup> Molar and Ketac<sup>™</sup> Universal. The SEM images revealed a micro-mechanical interlocking between coating and FIXF (Fig.1.B), while no interaction with KMand KU.(Fig. 2 and 3 B).

TABLE (2) Means and standard deviations of flexural strength (MPa), compressive strength (MPa) and hardness of all tested GICs with or without coat

	Flexural strength		Compressive strength		Hardness	
	Un-coated	Coated	Un-coated	Coated	Un-coated	Coated
FIXF	21.78±.89 <sup>bB</sup>	33.46±1.93 <sup>cbA</sup>	141.58±5.31 <sup>bB</sup>	190.80±4.19 <sup>bA</sup>	55.74±3.7 <sup>bA</sup>	53.00±4.19 <sup>bA</sup>
КМ	37.96±2.19 <sup>aA</sup>	35.1±1.32 <sup>abA</sup>	242.5±1.78 <sup>aA</sup>	243.49±3.17ªA	84.94±3.88ªA	82.35±2.03ªA
KU	39.52±2.16 <sup>aA</sup>	38.06±1.42ªA	243.58±1.78ªA	244.50±3.1ªA	86.94±3.6 <sup>aA</sup>	84.35±2.0ªA

Means with different small letters in the same column per each test indicate statistically significant different at p<0.05. Means with different capital letters in the same raw per each test indicate statistically significant different at p<0.05.



Fig. (1) SEM image of Fuji IX Gp fast showing: A: Microstructure, B: Coating layeron its surface. White arrow showing void inside the material. Black arrows showing mechanical interlocking between coating and Fujie IX Gp fast surface.



Fig. (2) SEM image of Ketac<sup>TM</sup> Molar showing: A: Microstructure, B: Coating layer on its surface.



Fig. (3) SEM image of Ketac<sup>™</sup> Universal showing: A: Microstructure, B: Coating layer on its surface.

## DISCUSSION

The High viscous glass ionomer cements might be the selected material to be used as a posterior restorative material not only due their enhanced mechanical properties but also their chemical adhesion to tooth structures, biocompatibility, fluoride release and uptake.<sup>22</sup> However, their clinical performance and survival rates are questionable.<sup>23,24</sup> In clinical studies, these types of GICs are related to Fuji IX (GC Corporation, Japan) or Ketac<sup>TM</sup> Molar (3 M ESPE, Germany) products.<sup>25</sup> KU is a new generation of conventional glass ionomer with enhanced mechanical properties even with lower viscosity compared to KM according to the manufacturer claims.<sup>12</sup> Accordingly, these three types of conventional GICs (FIXF, KM, and KU) were used in this study.

For standardization, (1) light cured resin coating supplied or recommended by the same manufacturer was chosen for each material. The manufacturer claims that, for KU and KM GICs, coating is an optional step since performance of the material will remain unchanged. (2) FIXF was choosed in this study to harmonize its setting time (3.35 minutes)<sup>26</sup> with other two types KM<sup>1</sup> and KU<sup>12</sup> setting time (3.30 minutes and 3.40 minutes) respectively which is lower than Fuji IX GP Extra<sup>27</sup> (2.30 m).

GICs are a brittle material which characterized by lower tensile strength and might be failed by crack propagation. Flexural strength is appropriate measure of GICs strength as it represents the clinical situation of opposing tooth exerts forces onto the restoration.<sup>28-30</sup> Moreover,the compressive strength is an essential property in restorative materials; particularly in mastication process. These tests are more suitable to compare brittle materials with low tensile strength.<sup>31</sup> Accordingly, two common mechanical strength (Flexural strength and compressive strength) tests were assessed in this study.<sup>29</sup>

Up to the knowledge of the authors, till now the mechanical properties of the new KU GIC have not been studied previously. Therefore, direct comparison of the mechanical properties results of KU with outcomes of other studies was not possible. Therefore, this study was conducted to evaluate the mechanical properties of the new generation of conventional glass ionomer (KU) for restoring posterior stress bearing areas versus two conventional posterior glass ionomer cement (KM and FIXF). Furthermore, the effectiveness of resin coating on Ketac<sup>™</sup> universal GIC was assessed.

The results of this study revealed that, KU and KM GICs recorded significant highest flexural and compressive strength values than FIXF. Powderliquid ratio, setting reaction, integrity of internal texture, presence of voids inside the material and small glass particles are considerd factors affecting flexural and compressive strength of the material.<sup>17,32</sup> All the tested GICs have nearly similar powder-liquid ratios, fast setting reaction (3.6-4/1)(3.35-3.40 minutes) respectively. <sup>11,12,26</sup> Thus, the strength of these tested materials was affected by integrity of internal microstructure, presence of voids and small glass particles. Accordingly, it was not surprising to have significant higher flexural and compressive strength for KU and KM as they have more dense internal microstructure, fewer and smaller voids, and smaller glass particles in comparison to the larger glass particle and larger voids seen in FIXF. These results are in accordance

with Prentice et al.<sup>32</sup> and Xie et al.<sup>17</sup>, they found that, the more decrease in glass particle size, the more increase in the strength of GICs as this will increase surface area of glass particles. Moreover, the material strength increased with more integrated microstructure. In contrast, there was nonsignificant difference between KU and KMGICs regarding flexural and compressive strength. This may be due to the nearly similar microstructure. This justification might agree to the SEM imaging results in this study.

Hardness is the resistance of the material to permanent surface indentation. It is the suitable test for estimating the materials degradation, durability and assessing their hardening process. Accordingly,it is considered essential property to expect the clinical performance of a restorative material inside the oral cavity.<sup>2,28</sup> It was reported by Xie et al.<sup>17</sup> that, small glass particles and lower porosity inside the material structure interrelated with hardness values.

Accordingly, the presence of well-dispersed, numerous small tightly packed glass particles inside the matrix may be a reason for higher hardness values for both KU and KM than FIXF. Moreover, a highly integrated glass particle–polymer matrix which was clear in SEM imaging resulted in the highest hardness values for KM and KU respectively. Also, the presence of cracks between glass particles and polymer matrix t in FIXF may be responsible for the lower hardness values.

Considering the effect of coating on the mechanical properties of all tested GICs, the flexural and compressive strength of FIXF was only enhanced. This could be attributed to two reason; First, the penetration of a self-adhesive coating inside the material which could fill the voids, counteract crack initiation and so strengthen the material. Second, coating may delay the water exposure of the material until complete maturation of the GIC reaction.<sup>10</sup> This finding might be due to the interaction between resin coating and FIXF

GIC since no interaction was found between resin coating and KU or KM as seen in SEM imaging. In line with findings, other studies which stated that the flexural strength of Fuji IX and Fuji IX GP Extra for coated GICs were significantly higher than uncoated specimens.<sup>33-35</sup> Regarding surface hardness for FIXF, coating has no significant improvement since hardness is a surface mechanical property, while flexural strength and compressive strength is an interinsic characteristic of the material.<sup>34,36</sup>

Although no significant improvement were detected for the effect of coating on KU and KM GICs, they have higher mechanical properties than FIXF. The justification for this could be detected in the internal microstructure and greater integrity of glass particle with polymer matrix seen in microstructure for KU and KM GICs. These findings in aggrement with Bonifacio et al.<sup>35</sup> who stated that, although there was no significant improvement for the effect of coating on Ketac Molar, it has higher strength thanFuji IX GP Extra.

Accordingly, from the results of this study, the first null hyposthesis was rejected and the second and third was accepted.

## CONCLUSION

Within the limitations of this study, Although KU has higher insignificant difference in the tested mechanical properties than KM, but it represent an encouraging line of higher clinical longevity of GICs'filling material in stress bearing areas. Also, savingstep for faster procedure is important issue in KU as the coating has no significant effect on the tested mechanical properties.

This study has a limitation, testing was done without simulations of in-vivo conditions such as saliva which may influence the mechanical propertiesas the water storage leads to only material degradation, whilst storage in saliva can increase the mineral content of glass-ionomer which in turn may affect mechanical properties of GICs.

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