SURFACE ROUGHNESS AND SOLUBILITY OF A NANO-FILLED RESIN MODIFIED GLASS-IONOMER (INVITRO STUDY)

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ABSTRACT

INTRODUCTION: Nanotechnology was used in the development of glass ionomer cements to provide some value added features not typically associated with this type of restorative materials.
OBJECTIVES: This study was conducted to evaluate the surface roughness and solubility of a nano filled resin modified glass ionomer cement and to compare it with a conventional type of glass-ionomer cement.
MATERIALS AND METHODS: Forty disc shaped specimens were prepared using Teflon split mold according to manufacturer instructions. Specimens were divided into two group :Group **A (20** specimens prepared from Ketac Nano) and **Group B (20** specimens prepared from Ketac Molar). Each group was subdivided into two subgroups of 10 specimens each. Twenty specimens from different subgroups were subjected to surface roughness test. The other twenty specimens from different subgroups were subjected to solubility test. Data were collected, tabulated and statistically analyzed.
RESULTS: Concerning surface roughness; Ketac Nano showed statistically significantly lower surface roughness than Ketac Molar where the mean values were 0.27 ± 0.10 µm and 0.48 ± 0.14µm respectively. Concerning solubilty; Ketac Nano showed statistically significant lower solubility than Ketac molar where the mean values were 4.25 ± 1.87 and 12.16 ± 2.89 µg/mm^3 respectively.
CONCLUSIONS: It was concluded that the addition of nano-fillers to RMGI seemed to decrease its surface roughness and to improve but without completely eliminating the solubility of the nano-glass ionomers.
KEY WORDS: surface roughness, solubility, nano glass ionomers.

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INTRODUCTION

Glass ionomer cements (GICs) were developed in 1972 by Wilson and Kent and presented unique restorative materials with many uses in clinical practice (1). What differentiates GIC from other restoratives is their chemistry, which allows them to be self-adhesive to enamel and dentin and provide for caries-protective fluoride release at the margins of restorations, as well as their ability to have the fluoride within their chemical matrix recharged by outside exposure to other fluoride-containing materials (2,3).

However, their poor mechanical properties, such as low fracture strength, toughness and wear limit their extensive use in dentistry as a filling material in stress-bearing areas (4). The requirement to strengthen those cements has led to an increasing research effort into reinforcement concepts (5).

Although all GICs share the same generic properties, differences between commercial products may occur. They have a wide range of uses such as lining, bonding, sealing, luting or restoring a tooth. Restorative types of glass ionomer is useful in situations where there is adequate tooth structure to support the material and where they are not subjected to heavy occlusal loading. Their use in posterior teeth has been limited by their physical properties. However, the need for a tooth-coloured material with relatively easy handling properties prompted the development of the high viscosity GICs (6).

However, alterations to their composition and the powder/liquid ratio affected their mechanical properties, handling and setting times, consistence and wear, improving their usage in clinical practice. The highly viscous conventional glass ionomers are particularly effective in the atraumatic restorative treatment (ART) and in places lacking the conventional infrastructure needed for clinical treatment (7-11).

In order to expand the clinical uses of GIC, resin was added to the formulation. When compared to conventional GIC, resin modified glass ionomers provide improved physicomechanical properties and resistance to early contamination by moisture, less microleakage, and improved adhesion to enamel and dentin combined with significant improvement in esthetic properties (2, 12).

Nanotechnology was used in their development to provide some value added features not typically associated with glass- ionomer restorative materials. Generally, glass-ionomer restoratives can contain a broad range of particle size. Filler particle size can influence strength, optical properties, and abrasion resistance (13). By using bonded nanofillers and nanocluster fillers, along with fluoroaluminosilicate glass Ketac Nano restorative has improved esthetics, yet still provides the benefits of glass ionomer chemistry, such as fluoride release. The nano-filled resin-modified glass ionomer cement, or “nano-ionomer” (Ketac N100) was developed by 3M ESPE (14-16).

Studies suggest that the commercially available nano-filled Resin Modified Glass Ionomer Cement (RMGIC) does not hold any significant advantage over conventional RMGICs as far as the mechanical and bonding properties are concerned. Conversely, other studies suggest that incorporation of nano-sized apatite crystals not only increases the mechanical properties of conventional GICs, but also can enhance fluoride release and bioactivity. By increasing the crystallinity of the set matrix, apatites can make the set cement chemically more stable, insoluble, and

improve the surface roughness and the bond strength with tooth structure. However, due to a lack of long-term clinical studies, the use of nano-modified glass ionomers is still limited in daily clinical dentistry (17).

Therefore, it would be of interest to evaluate surface roughness and solubilty of a nano-filled resin modified glass ionomer restorative material and to compare it to a conventional glass ionomer restorative material. The null hypothesis in this study was that the nano filled resin modified glass ionomer restorative material would show the same surface roughness and solubility as the conventional glass ionomer restorative material.

MATERIALS AND METHODS

Restorative materials that were used in this study: (Table 1)

- Nano-filled resin modified glass ionomer cement (Ketac Nano).
- Conventional glass ionomer (Ketac Molar).

A total number of 40 disc shaped specimens of 10 mm diameter and 2 mm thickness were prepared into specially designed teflon split mold according to manufacturer's instructions. Specimens were divided into two groups:

Group A

Twenty disc shaped specimens of 10 mm diameter and 2 mm thickness were prepared from ketac Nano light curing restorative capsules (3M-ESPE, St. Paul, MN, USA). Capsules were activated by lifting their nozzle upwards and inserted into an applier to inject Capsule content directly into the teflon mold. The mold was filled and then covered with a transparent matrix strip and pressed flat with a glass slide against the bottom of the mold. Then light cured from its upper surface for 20 seconds using led curing unit (Woodpecker, LED P, china) with light intensity 600 mW/cm^2.

Group B

Twenty disc shaped specimens of 10 mm diameter and 2 mm thickness were prepared from Ketac Molar capsules (3M-ESPE, Seefeld, Germany). Capsules were activated for 2 seconds using an activator then triturated in an amalgamator for 8 seconds and inserted into an applier to inject capsule content directly into the Teflon mold through the capsule nozzle. The mold was filled and then covered with a transparent matrix strip and pressed flat with a glass slide. After 5 minutes according to manufacturer's instructions to assure complete polymerization matrix was discarded and glass slide was removed. Discs were removed from mold by pushing the Teflon condenser against the bottom in an upward direction.

Group A and group B were both subdivided into two subgroups of 10 specimens each. For surface roughness test twenty specimens from different subgroups were examined using surface roughness measuring instrument for roughness Ra value measurement. The surface roughness is defined as the average of the height of the surface profile above and below a centerline throughout a prescribed sampling length. Specimens were stabilized and the surface roughness of each specimen was measured in 5 different positions using a surface profilometer(Surftest 211, Mitutoyo, Tokyo, Japan). Surface profilometer consists of a pointed stylus to trace the profile of the surface irregularities. The cut-off value for surface roughness was 0.8 mm, and the traversing distance of the stylus was 4.0 mm. The radius of the tracing diamond tip was 5 µm, the measuring force and speed were 4 mN (0.4 gf) and 0.5 m s−1, respectively. The average roughness value (Ra, µm) of an individual disc was taken as the mean of the Ra (average of peaks and valleys of a surface) values.

For solubility test twenty specimens were taken from different subgroups and were placed into pre-numbered glasses and were deccicated using calcium chloride crystals and stored in an incubator (Helena- laboratories, U.S.A) at 37±1°C for 1 hour to eliminate moisture. Then each sample was taken from its glass and weighed in an electronic balance analyzer (M1 values) (RADWAG, balances and scales, Poland). 10 ml of deionized water was added to the specimens into the pre-numbered glasses and the samples were stored in an incubator at 37±1 C for 7 days. Then samples were removed from deionized water, blotted dry with absorbent paper, dried with gentle air spray for 1 minute and were weighed (M values). Then the samples were deccicated and placed in incubator for 1 hour and were weighed again (M2 values). The M value was not used to determine the solubility value of the materials but needed to be recorded for it is a transition value between M1 and M2 values according to the ISO 4049 standardization (18).

- Solubility was calculated according to the formula:
  - $\mathbf{Wsol} = \frac{\mathbf{M1-M2}}{\mathbf{V}}$
  - Wsol: Solubility of test material (µg/mm^3)
  - V: Volume of test material (mm^3) $V = [(\pi*D^2)/4]*L$ where V= volume, π; 3.1416, D= diameter of specimen in mm, and L= thickness of specimen in mm
  - M1, M, and M2: Weighed value of test material (µg)

Statistical analysis: Data were collected, tabulated and statistically analyzed. Differences in surface roughness and solubility values were assessed using student t-test; $p \leq 0.05$ was considered as statistically significant.

RESULTS

In the present study the mean value of the surface roughness of Ketac Nano was 0.27 ± 0.10 µm and the mean value of the surface roughness of Ketac Molar was 0.48 ± 0.14µm. When both groups were compared using the Student t test, Ketac Nano showed statistically significant lower surface roughness than Ketac Molar ($p < 0.05$) (Table 2 & Fig. 1).

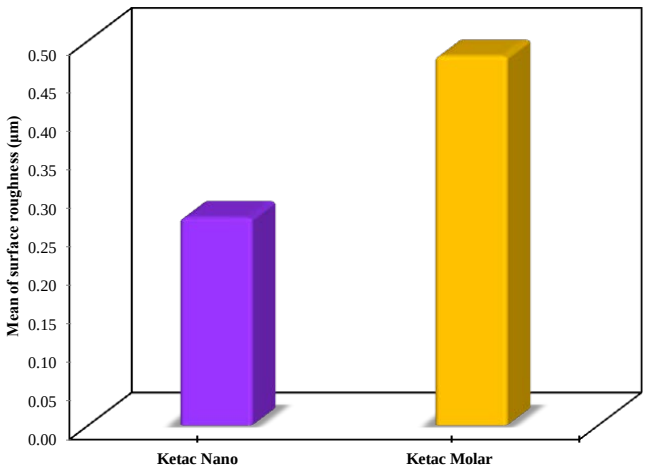

Figure (1): Comparison between the two studied groups according to surface roughness.

Regarding the solubility, the mean value of solubility of Ketac Nano (W) was 4.25 ± 1.87 and the mean value of solubility of Ketac Molar (W) was 12.16 ± 2.89 µg/mm^3. When both groups were compared using the Student t test,

Ketac Nano showed statistically significant lower solubility value than Ketac molar ($p < 0.05$) (Table 3 &Fig. 2).

Pearson correlation coefficient test showed that there is no correlation between surface roughness and solubility of the two tested GICs ($r = 0.6$ & 0.2 for Ketac Nano and Ketac Molar respectively) ($P > 0.05$).

Table (1): Characteristics of the used materials

Materials	Type	Filer size	Composition	Manufacturer
Ketac™ Nano Light-Curing Glass Ionomer Restorative	Light-curing nano-ionomer restorative	0.005-0.025 µm	Deionized water, hydroxymethyl methacrylate (HEMA), acrylic/itaconic acid-copolymer photoinitiators fluoroaluminosilicate (FAS), methacrylate modified polyalkenoic acid, nanomers and nanoclusters.	3M-ESPE, St. Paul, MN, USA
Ketac™ Molar Easymix	High viscosity conventional glass ionomer	1-9 µm	Powder: Aluminium-calcium-lanthanum fluorosilicate glass, liquid: polycarboxylic acid	3M-ESPE, Seefeld, Germany

Table (2): Comparison between the two studied groups according to surface roughness.

Groups	Surface roughness (µm)				
	Mean	SD	Min	Max	Median
Ketac Nano	**0.27**	± 0.10	0.17	0.45	0.21
Ketac Molar	**0.48**	± 0.14	0.31	0.73	0.44
p	**0.001***				

p: p values for Student t-test for comparing between the two groups.
*: Statistically significant at $p \leq 0.05$.

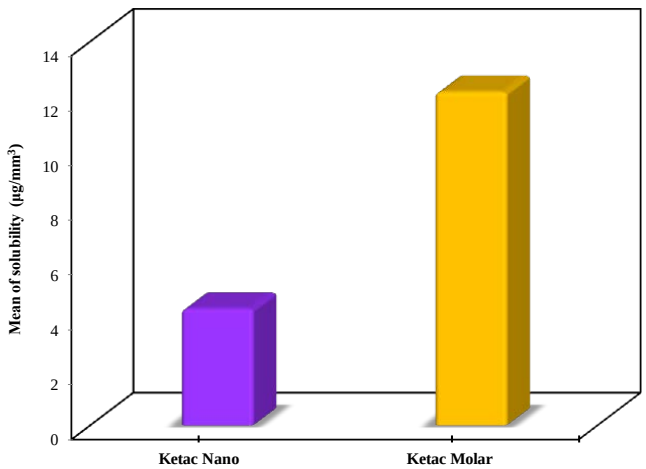

Figure (2): Comparison between the two studied groups according to solubility.

Table (3): Comparison between the two studied groups according to solubility.

Groups	Solubility W (µg/mm³)				
	Mean	SD	Min	Max	Median
Ketac Nano	**4.25**	± **1.87**	0.64	6.36	4.73
Ketac Molar	**12.16**	**2.89**	8.28	17.19	11.46
p	**< 0.001***				

p: p values for Student t-test for comparing between the two groups.
*: Statistically significant at $p \leq 0.05$.

DISCUSSION

The study of surface roughness is important due to the fact that this property affects light reflection, color fading appearance of cracks and aesthetics, in addition to favoring biofilm accumulation which could aggravate the risk of carious lesion and periodontal disease (19- 21).

Profilometers have been widely used to measure surface roughness in vitro. It provides two-dimensional information and arithmetically calculates average roughness offering various treatment choices (22).

In the current study Mylar strip was used during specimen preparation as recommended by Sidhu et al (23), that the cover or finishing used in clinical procedures may veil the characteristics of the material in laboratory experimentations. The best evenness of the surface was attained when the materials were cured in contact with the polyester strip (24, 25).

Several authors have shown the smoothest surfaces of GICs are obtained with the Mylar strip. However, it was noted that the correct anatomic contour of the restoration is rarely achieved by using only a Mylar strip (25, 26).

In the current study no finishing or polishing was done to specimens as Finishing or polishing of esthetic restorative materials always pose a difficulty because particles and matrix differ in hardness and thus cannot be abraded uniformly. For a finishing system to be effective, the cutting particles must be harder than the filler materials. Otherwise, the polishing agent will only remove the matrix and leave the particles protruding from the surface (27-29).

The value of the surface roughness (Ra) considered critical when the retention and adherence of microorganisms is equal to 0.2 µm (20).

Also Leitão and Hegdahl (30), reported that the surface is considered rough when it bears peaks and valleys of great amplitude with reduced undulation.

Preliminary studies have shown that highly viscous conventional GICs has enhanced mechanical properties by their improved chemistry (31). On the other hand, it presented higher roughness mean values in other studies suggesting that this behavior may be related to the size and shape of glass particles on its surface. Particle size has been shown to play an important role in surface roughness of material. Some studies have been recorded the highest values of surface roughness for the materials with larger particle size (4).

In the present study; Ketac Nano showed significant lower surface roughness than Ketac Molar where the mean values were 0.27 ± 0.10 µm and 0.48 ± 0.14µm respectively. This may be due to addition of nano-fillers to RMGI.

Similar to the present study, Hussein et al (32), found significant difference in the surface roughness between Ketac Nano and Ketac Molar. The Ra values of Ketac™ N100 were the lowest among all GICs tested in their study.

Also Ozdemir-Ozenen et al (33), found the mean initial surface roughness of Ketac N100 was significantly lower than the other tested conventional glass ionomer (Fuji IX).

Soares et al (34), studied conventional glass ionomer cement, resin modified glass ionomer and nanofilled glass ionomer cement (Ketac Nano, 3M-Espe). Similar to the present study Soares et al, found that nanofilled glass ionomer resin cement had significantly lower surface roughness than the both tested glass ionomer cements.

Also Mohamad, et al (35), found that the microfilled GIC showed the highest surface roughness, followed by nanofilled GIC which is in agreement with present study.

Furthermore, Bala et al (22), evaluated surface roughness of a nanofiller GIC, resin-modified GIC, conventional GICs, and a silver-reinforced GIC. They showed that the mean initial Ra value of Ketac N100 showed smoother surfaces than the other tested GICs both before and after polishing.

Similar to the present study, in Ragab et al (36), and Singh et al (37), the Ketac N100 showed the lowest Ra value among the other tested materials. While Momesso et al (38), studied the conventional high-viscosity GIC other than Ketac Molar, it showed high roughness mean values as in the present study.

In the current study it was found that Ketac Nano showed significant lower solubility than Ketac Molar where the mean values of solubility were 4.25 ± 1.87 and 12.16 ± 2.89 $\mu g/mm^3$respectively. This may be due to addition of nano fillers which improves without completely eliminating the solubilty of Ketac Nano.

Zaazou et al (39), evaluated the solubility of nano-glass ionomer cement restoration when used with and without unfilled surface resin sealant coating. Similar to the present study it was found that nano-glass ionomer showed low solubility values. Moreover, coating did not show statistically significant lower solubility than nano-glass ionomer uncoated group.

However, Dinakaran (40), studied the sorption and solubility of compomer, conventional glass-ionomer and resin modified glass ionomer cements in different media. Conventional glass-ionomer (Fuji II) cement showed the high values of water sorption and solubility in all the different immersion media than other tested materials which was similar to the present study.

The current study revealed that surface roughness and solubility of nano filled resin modified glass ionomers were significantly lower than conventional glass ionomers and thus, the null hypothesis was rejected.

CONCLUSIONS

Within the limits of this study, it can be concluded that, Ketac Nano showed lower surface roughness and lower solubility values than Ketac Molar. The addition of nano-fillers to RMGI seemed to decrease its surface roughness and improve but without completely eliminating the solubility of the nano-glass ionomers.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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