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# Bioactivity of Gutta flow II versus Modified Gutta Percha based Silicon Endodontic Sealers by Nanobioactive Fillers

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## Abstract

The success of endodontic therapy is relied on radicular system cleaning, shaping, elimination of micro-organisms, and three dimensional filling of the radicular complex.This study was conducted to develop and assess new root canal sealer incorporating nano-sized bioactive glass into Gutta Flow II. The following concentration was used depend on a pilot study included adding (3%) of 45S5 bioactive glass into the Gutta Flow II. These materials were tested through assessment bioactivity. bioactivity test was undertaken after immersion of the tested samples into PBS for three days, seven days, fourteen days, and twenty eight days using FTIR too. study was found that it's peaks was appear at level 800-1000 $cm^{-1}$. The results showed that GFII group revealed no peak at the 910 $cm^{-1}$, while, while BG3% revealed 179.85 pixel height, in conclusion the newly developed sealers exhibited apparent apatite and apatite precursor forming ability significantly with BG 3% while GuttaFlow II sealer showed no apatite layer forming ability.

***Key words:*** *bioactivity, chemical analysis, 45S5 bioactive glass*

## Introduction

Although gutta-percha based silicon sealers like Gutta Flow II is characterized by many advantages like simplicity of handling, setting expansion that may improve its sealing ability, in addition to certain extent of anti-microbial activity due to the effect of silver particles which were added as fillers [1] but generally gutta percha still has shortcomings , involving the inability to re-enforce the root canal treated tooth [2]. Furthermore, the ability of gutta-percha to provide hermetic seal is still controversial as it does not capable to bond with root dentine [3]. Specifically, Gutta Flow II still has considerable cytotoxicity, insufficient antibacterial activity, no bioactivity [4]. Calcium silicate–based materials are classified as bioactive substances due to their ability to stimulate hard tissue remineralization and regeneration in both the dental pulp and bone [5]. Silicone-based endodontic sealers showed relatively good biological features [6]. The silicone-based sealer GuttaFlow II (Coltene Whaledent, GmBH + Co KG) constitutes a blend of polydimethylsiloxane and gutta-percha powder with nanometer-sized silver particles incorporated as a preservative. GuttaFlow II, a development of its predecessor GuttaFlow, is also a cold thixotropic flowable system. Both GuttaFlow and GuttaFlow2 are silicone-based root canal sealants that have variable forms of the silver particles used [7]. A novel formula of polydimethylsiloxane with gutta-percha powder joined with calcium silicate particles was introduced in late 2015 and named GuttaFlow Bioseal (Coltene/Whaledent AG, Altstatten, Switzerland). In the present study nano-sized bioactive fillers of 3% were incorporated and distributed mechanically into gutta flow II sealer in order to develop bioactivity. Nano-sized 45S5 bioactive glass were chosen to be used in this study to lower the percentage of the incorporated powder in order to keep the physical characteristics of the original sealer regarding solubility and flow ability. In addition, to keep the newly developed material clinically applicable.

## Materials and Method

**Sample grouping:**

**Group 1 (control group):** ten samples were prepared from Gutta flow II sealer.

**Group 2:** ten samples were prepared from Gutta flow II incorporated with nano sized 45S5 bioactive glass particles of optimum percent by weight according

to the optimization results of the pilot study.

**Preparation of bioactive glass containing Gutta Flow II 3%:**

The sample was prepared by mixing 0.3 mg of 45S5 bioactive glass with 7.7 mg of gutta flow II base. The pistons of both base and catalyst were separated by diamond disc that mounted on engine driven straight hand piece at their connected base. The gutta flow II was extruded on sterile screw cup, then 0.3mg was removed from the base to be replaced by 0.3 mg of bioactive glass. The mixing procedure was accomplished by adding the 0.3 bioactive glass carefully, and the mixture was mixed well by using disposable plastic spatula which was installed into INGCO cordless drill till complete homogenization of the mixture. The mixing time was 2 minutes and the speed was 750 R.P.M. Then the newly mixed material was reloaded in the base tube of the syringe carefully by using plastic spatula. The base piston was re-inserted into the base tube and pushed down to the level of the catalyst tube and rejoined at their base by using adhesive liquid.

**Assesment of apatite forming ability (bioactivity) using Fourier Transform**

**Infrared Spectroscopy**

Disk samples were impregnated into phosphate buffered saline solution (PBS) to mimic tissue fluid immersion and examined at 3, 7, 14, 28 days post immersion. At each period, samples were displaced from storage solution. The pH of the saline solution was usually checked by pH meter to keep its pH at 7.2 [8]. 0.1 mg of the each disc was taken from its surface and tested by FTIR according to the manufacturers instructions to detect the development of carbonated hydroxyappetite layer at each sample surface.

## Results and Discusion

**Chemical analysis using FTIR**

According to the chemical structure of the gutta percha figure (5) and it's other included materials gutta flow II, the FTIR spectra range was 400 – 4000 cm $^{-1}$. The spectra of 50 Nano meter sized particles of 45S5 was 910 cm$^{-1}$, in which the primary peaks in correspond to the Si-O vibrational modes of the bonds in the glass network, the stretching vibration at 1090 cm$^{-1}$ and the rocking vibration at 450 cm$^{-1}$. The broad peaks in the 750-950 cm$^{-1}$ region correspond to Si-O-Si vibrational modes associated with the $Ca^{+2}$ and $Na^{+}$ ions in the glass network as shown in the figures (2).The chemical groups in the FTIR spectrum of HA are $PO4^{-3}$, $OH^{-}$, $CO3^{-2}$, as well as $HPO4^{-2}$ that are characteristic of non- stoichiometric HA. $PO4^{-3}$ group forms intensive IR absorption bands at 1000 –1100 cm$^{-1}$. $CO3^{-2}$ intensive peaks between 1460 and 1530 cm$^{-1}$ which represent HA as shown in the figure (2).

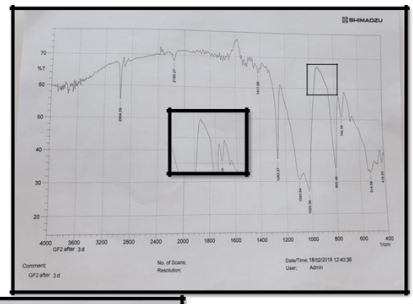

**Fig (1) shows FTIR chart of GF II which appears no peak at 910 cm$^{-1}$ level**

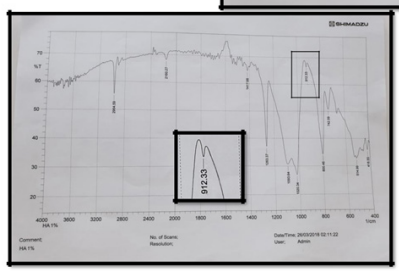


**Fig (2) shows FTIR chart BG3% which appears a peak of 63.95 pixel at 910 cm-1 level**

**Bioactivity test:**

According to the structure of the carbonated layer figure (4) the FTIR study was found that it's peaks was appear at level 800-1000 cm$^{-1}$

According to the results of the bpresent study, GFII showed no development of carbonated hydroxyapatite layer

The peak of carbonated material start to appear in the BG 3% group at the third day with obvious increment of it's length figures (3).

Apparently BG 3% group reach to the maximum concentration of the carbonated HA layer which was indicated by increase the length of the peak at day 28,


**Fig (3) shows FTIR chart of BG3% after 14 days which appears a peak of carbonated hydroxyapatite at 900 $cm^{-1}$ level**

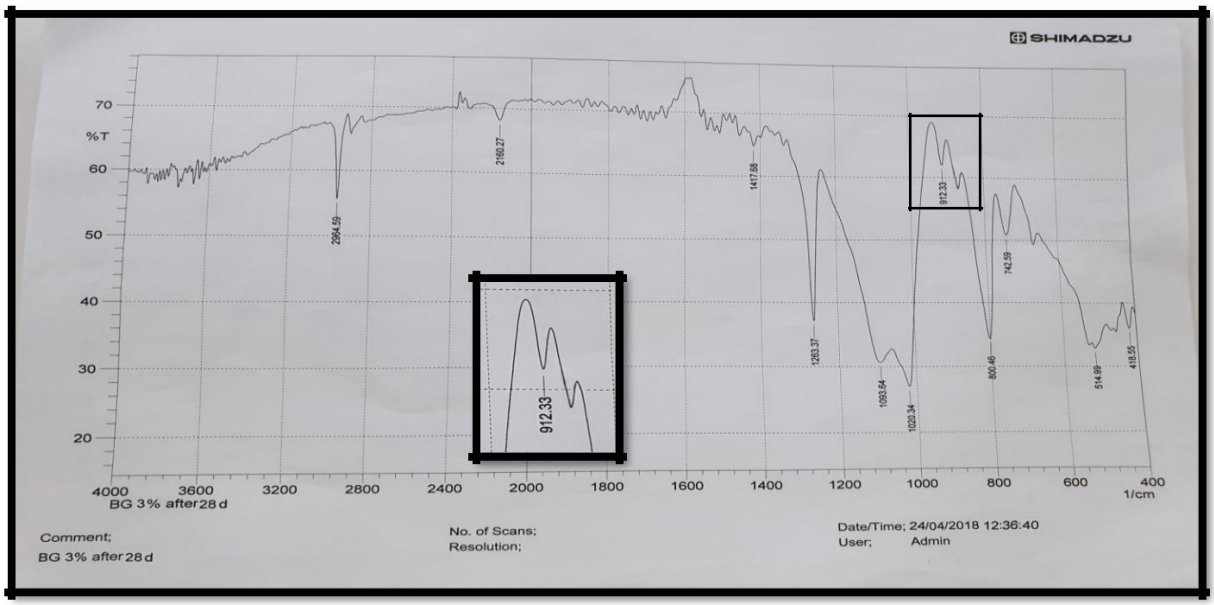


**Fig (4) shows FTIR chart of BG3% after 28 days which appears a peak of carbonated hydroxyapatite at 900 $cm^{-1}$ level**

## Discussion

Through out the obturation step of endodontic remedy, periradicular tissues may contact the root canal sealents via extrusion through the apical foramen [9]. When sealers are in intimate contact with the periapical tissues for extended periods of time, their breakdown toxic products may hamper the periapical healing process by inhibiting the proliferative capability of the periradicular cell population [10].

Therefore, apart from good physical and chemical characteristics, endodontic sealers should be biologically compatible [11]. Several methods have been recommended for filling of root canals. Use of gutta-percha, a semi-solid root filling material, in combination with sealer is the most commonly performed method of root filling [12]. Low dose of bio-active material is needed because the nano- particles allowed more surface area that may provide better activity, this may prevent adverse effect on the sealer flow characteristics. Incorporation of nano-

sized bioactive materials enhance bioactivity of already available endodontic sealer this is agreed with [13].

Particulate size has been proved to have effect on biomineralization; nanoparticles showed better performance than microparticles in induction of biomineralization [14]. Paying attension to this fact, there might be still potential for enhancement of the performance of bioactive features.

there is no specific information on the osteogenic and cementogenic potential of calcium silicate-based sealers on mesenchymal progenitor cells through out the repair of periodontal ligament and relative surrounding tissue, and the molecular mechanisms underlying the osteogenesis and cementogenesis induced by various dental substances are unclear [15]. This report is disagree with the present study. While

[16] stated that calcium silicate-based sealers are new root canal sealers that have demonstrated apatite forming ability. This studies are agree with the present study.

Biomineralization and formation of hydroxyapatite (HA) begin with release of ions, especially calcium ions from the inorganic calcium-silicate particles and continue with formation of Si-OH groups at the material's surface. The Si-OH groups on the surface function as ideal site for nucleation of HA. HA, i.e. $Ca10(PO4)_6(OH)_2$ precipitates first as an amorphous layer with the stoichiometric ratio Ca/P 1.67 and later crystallized into carbonated hydroxyapatite (CHA). HA precipitation is favored by an increase in the pH of the surrounding solution. The increase of the pH of the interfacial solution to around 7.9 and above around the dissolving BG has been shown. The combination of adequate physical charateristics containing silicone-based sealer and bio-interactive bioactive glass-ceramic (BG) particles is a prospective option to conventional root canal sealers. Silicone-based endodontic sealers also showed advantages against mineral trioxide aggregate (MTA) cements in manipulation and clinical application. This study explores in detail the potential of GB, a new endodontic sealer containing BG particles to induce biomineralization. The aim was to demonstrate apatite forming ability, the influence of the BGC on the surface structure and examine ion dissolution patterns of the endodontic sealer GB more widely that is currently available in the scientific literature. Most of the data in similar studies are now limited to concern only calcium ion release. In addition, change of pH in the surrounding media. This is agreed with [17].

BGs gradually dissolve when exposed to aqueous media. The degree of dissolution depends on the ratio of the surface area of the glass in contact with a certain volume of solution. However, the Ca/P ratio levelled off rather slow. This is agree with [18]. The rather weak intensities of the HA reflections are indicative of a thin HA layer, which also results in strong interfering signals from the crystalline filler materials inside the underlying matrix [17]. Biomineralization requires excess of mineral forming ions to be present in the solution and in this case, the ions originated from the dissolution of BG particles and from the SBF. In this study, the carbonated HA was measured by using FTIR analysis. Although Ca ion release is strongly related to bioactivity most of the studies in current scientific literature ignores the release of other ions which also have biological interest. Different BG compositions used as bioactive fillers in composites are known to have different dissolution behaviours. The newly developed materials showed low number of BG particles in the PDMS matrix, but despite of that, biomineralization, and more importantly increase of pH to the bacteriostatic level was seen. Therefore, it can be assumed that the sealer may have beneficial effects also in vivo conditions where ratio of surface area/volume favors the emerging local alkalinity. This is agreed with [17]. FTIR has been intensively used in the study of the surface reaction of bioactive materials after immersion in simulated body fluid (SBF) solution [19] and after implantation [20]. Both diffuse reflectance and specular reflectance can be used for monitoring the formation of the HCA layer at the surface of bioactive materials. The HCA layer is characterized by the P–O bending vibration peaks at 560 and 604 $cm^{-1}$ and the P–O asymmetric stretching vibration bands between 1000 and 1150 $cm^{-1}$ [21]. The peaks corresponding to the bending vibration are most used to discriminate between HCA and bioactive material since the P–O stretching band is superimposed on the Si–O stretching band corresponding to bioactive glasses while the absorption band corresponding to Si–O bending is between 400 and 500 $cm^{-1}$ [21], at lower frequencies compared with P–O bending in HCA. The magnitude of HCA peaks at 560 and 605 $cm^{-1}$ increases relative to the foam Si–O peaks at 450 $cm^{-1}$ as the immersion time is longer. This indicates that the HCA layer is thicker for the samples soaked in simulated body fluid (SBF) solution for longer times. However, the signal-to-noise ratio of the reflection spectra is poor due to the porous surface,

600 interferograms being needed to obtain the spectra. Therefore, the easiest application of FTIR is the study of bioactive powders with a KBr diffuse reflectance device. In this case, the signal is much stronger and the preparation time is less. carbonate peak at 875 $cm^{-1}$ developed at 0.005 g $ml^{-1}$and increased in intensity with prolonging the time of immersion into SBF. In particular, the band around 500 $cm^{-1}$ can be related to the longitudinal optical (LO) Si–O–Si vibration, indicating a strong distortion of the SiO4 tetrahedra at high content of glass network modifiers (i.e. $Ca^{+2}$ and/or $Na^{+}$ ) [22]. The next band at 600 $cm^{-1}$ can be attributed to either the P–O bending vibrations of $PO4^{-3}$ groups present in the amorphous phosphate, or pseudolattice vibrations of the PO4 tetrahedra with at least one bridging oxygen [23]. The bands in the 732–805 $cm^{-1}$ range were attributed in the literature to the Si–O–Si bending mode. The band in the 1000–1050 $cm^{-1}$ range could be related to stretching vibrations of the Si–O–P [24] . A higher intensity of the bands at around 930 and 854 $cm^{-1}$ for the 45S5 glass indicates an increased degree of depolimerization of the silicate network. The FTIR spectra of the bio active glass confirm the beginning of crystallization, while the band at around 930 $cm^{-1}$ becomes more intense and sharper, indicating the crystallization of the major $Na_2Ca_2Si_3O_9$ phase (combeite). Additionally. A weak band at 696 $cm^{-1}$ can be assigned to the symmetric stretching vibrations of the Si–O–Si in the crystalline silicate [25]. The group of bands at 526, 574 and 619 $cm^{-1}$ indicates the presence of crystalline phosphate-rich phase, i.e. $Na_2Ca_4(PO_4)_2SiO_4$ (silicorhenanite). The two latter bands are characteristic of the P–O bending vibrations.this is agreed with [25]. The band at 696 $cm^{-1}$ can be ascribed not only to Si–O–Si stretching mode, but also to P–O bending. This is agreed with [26] .

## Conclusion

**Financial Disclosure:** There is no financial disclosure.

**Conflict of Interest:** None to declare.

**Ethical Clearance:** All experimental protocols were approved under the College of dentistry- Dept. of Oral diagnosis, Iraq and all experiments were carried out in accordance with approved guidelines.

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