



# Effect of forsterite nanoparticles on mechanical properties of glass ionomer cements

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## Abstract

The aim of this study was to assess the effect of forsterite nanoparticles on the mechanical properties of glass ionomer cements (GICs). So, forsterite nanoparticles were produced by a sol–gel process and added to the ceramic component of a commercial glass ionomer cement at 1–4 wt%. An X-ray diffraction (XRD) analysis technique was used to characterize the phase composition and the grain size of forsterite nanoparticles. Compressive strength (CS), three-point flexural strength (FS), and diametral tensile strength (DTS) of the prepared glass ionomer-forsterite nanocomposites were measured. Analysis of variance (ANOVA) was used to compare the obtained results. Scanning electron microscopy (SEM) imaging technique was used to study the morphology of the fractured surface after performing the mechanical tests. XRD analysis confirmed the synthesis of pure nanocrystalline forsterite particles. Statistical analysis showed a significant difference between the results of mechanical properties of the control specimens and the glass ionomer-forsterite nanocomposites. The highest compressive strength, flexural strength and diametral tensile strength were obtained using 3, 1, and 1 wt% of forsterite nanoparticles, respectively. However, at 1 wt% forsterite nanoparticles, all three measures of strength exhibited a significant increase compared to the commercial GIC. Thus, addition of 1 wt% forsterite nanoparticles to the ceramic component of GIC is desired for dental restorations and orthopedic implants applications, where the maximum strength in all three modes of loading would be beneficial.

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## 1. Introduction

Glass ionomer cements (GICs) are direct tooth-colored restoration materials with many advantages including; anticariogenic properties due to fluoride ion release, thermal compatibility with tooth enamel due to the low thermal expansion coefficient similar to those of tooth structure, the module coefficient similar to dentin, ability of chemical bonding with dentin, biocompatibility, and low cytotoxicity. Nevertheless, weak mechanical properties limit their high-stress applications, such as class I and II restoration [1,2].

Many efforts have been done in order to improve the mechanical properties of GICs over the past few years with their advantages and limitations. The incorporation of amalgam, silver, and metal powders as reinforcements into GIC powder was reported by Irie et al. with inferior esthetic appearance and low bonding strength to the enamel [3]. Mitra and co-workers investigated the light-cured GIC, where resin and a light-curing catalyst were added to stimulate the setting and enhance the mechanical strength of GIC. Light-cured GIC is widely used due to its significant high flexural strength [4]. However, it shows lower compressive strength compared to the conventional GIC [5]. In addition, resin-modified GIC may cause dental pulp irritations, biological side-effects, cytotoxicity, and secondary caries, whereas these negative effects are

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not caused by conventional GIC [6,7]. Incorporation of SiC whiskers/short fibers into GIC enhances mechanical properties. However, there are health concerns regarding the release of the small fibers and their cytotoxicity to vital organs, similar to the cytotoxicity caused by asbestos fibers [8,9].

Unlike forsterite microparticles, forsterite nanoparticles demonstrate bioactive characteristics due to their high surface energy and reaction tendency that lead to integration with adjacent tissue. They also present higher mechanical properties than forsterite microparticles and hydroxyapatite (HA) [10]. Thus, forsterite nanoparticles might be a good candidate for replacement of HA ceramic in loaded-applications.

Mechanical properties and bioactivity of forsterite nanoparticles and also the possibility of controlled magnesium and silicon ions release that can repair and regenerate bone tissue, make them a desirable candidate to be used as a secondary phase in ceramic matrix. There are several methods to synthesize forsterite nanoparticles including; solid reactions, high temperature and sol–gel. Among them, the sol–gel process provides low working temperature, small size of powder crystals, a narrow particle size distribution, and chemical homogeneity [11].

Kharaziha et al. synthesized forsterite nanoparticles calcined at 800 °C. Fig. 1 shows the transmission electron microscopy image of the nanoparticles with the average size of 25–45 nm [12].

Our previous study showed that adding forsterite ( $Mg_2SiO_4$ ) nanoparticles to the ceramic component of GIC increases the bioactivity of the nanocomposite and decreases fluoride release [13]. In the present study, the aim is to synthesize glass ionomer–forsterite nanocomposites with different amounts of forsterite nanoparticles composition and assesses their mechanical properties including; compressive strength (CS), three-point flexural strength (FS), and diametral tensile strength (DTS).

## 2. Materials and methods

### 2.1. Forsterite nanoparticles

Forsterite nanopowder was synthesized by the sol–gel process according to Kharaziha et al. [12]. Magnesium nitrate hexahydrate

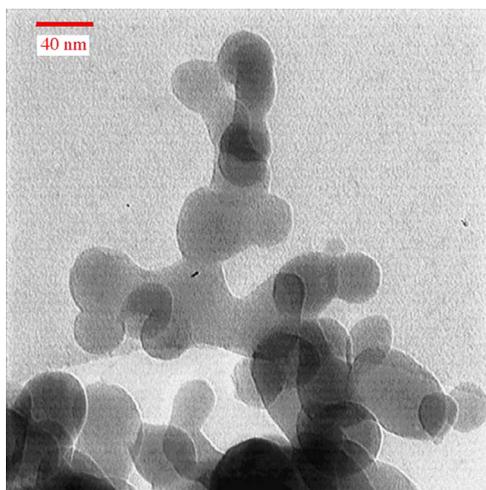


Fig. 1. TEM micrograph of  $Mg_2SiO_4$  nanopowder calcined at 800 °C [12].

( $Mg(NO_3)_2 \cdot 6H_2O$ , Merck, 99.99% purity), colloidal silica ( $SiO_2$ , 34 wt% solid fraction, Sigma), polyvinyl alcohol polymer (PVA) (Merck, molecular weight=72,000), sucrose (Merck, 99.9% purity) and nitric acid were the starting materials. Fuel system combined PVA and sucrose (sucrose-to-metal molar ratio=4:1) was applied to prepare forsterite. Water-based solutions of magnesium salts and colloidal silica were prepared with the stoichiometric molar ratio of forsterite ( $Mg:Si=2:1$  mol) by pouring colloidal silica into the aqueous solution of magnesium nitrate (0.0142 mol magnesium nitrate) dissolved in 50 cc deionized water. The aqueous solution of sucrose (sucrose-to-metal ratio=4:1 mol) in 100 cc deionized water was added dropwise to the precursor solution and two solutions were homogenized together on a warming plate under 2 h continuous stirring. PVA (PVA monomer-to-metal molar ratio=0.8:1) dissolved in 20 cc deionized water was added and the pH was adjusted at 1 using nitric acid and the solution was mixed homogeneously by constant stirring for 2 h with a magnetic stirrer. Subsequent heating at 80 °C for 2 h on a hot plate stirrer, the prepared gel was aged for 24 h. The resulting gel was then heated on a hot plate at 100 °C in air for enough time to complete the dehydration process and change into a voluminous, black, and fluffy gel. To increase the purity of the forsterite nanoparticles, the dried gel was calcined in a furnace at 800 °C for 2 h.

Phase composition analysis of nanoparticles was performed by an X-ray diffractometer (XRD, Philips Xpert) using Ni filtered Cu Ka ( $ICu\ Ka=0.154$  nm, radiation at 40 kV and 30 mA) over the  $2\theta$  range of 20°–80° (time per step: 1.25 s and step size: 0.05°). XRD spectra were compared to standards compiled by the Joint Committee on Powder Diffraction and Standards (JCDPS) [14]. The crystallite size of the forsterite nanoparticles was calculated using the modified Scherrer equation [15].

### 2.2. Glass ionomer–forsterite nanocomposite

Glass ionomer–forsterite nanocomposite was produced by adding forsterite nanoparticles at 1, 2, 3, and 4 wt% to the ceramic component of the GIC, where GIC and forsterite nanoparticles were mixed in the amalgamator for 30 s according to the manufacturer's instruction. Powder/liquid ratio was set at 2.7/1. Then, the specimens were transferred into an aluminum mold. Specimens were removed from the mold after 1 h and prepared for following tests. Commercial GIC (Fuji II, GC International, Tokyo, Japan) was used as the control.

### 2.3. Mechanical properties measurements

Mechanical tests were performed on a screw-driven testing machine (Hounsfield, Model H25KS, England) with a cross-head speed of 0.5 mm/min.

For compressive strength test, cylindrical specimens ( $4 \pm 0.1$  mm diameter and  $6 \pm 0.1$  mm height) were prepared according to ISO 9917-1 standard. The compressive strength was calculated by the equation  $C=4P/\pi d^2$ ; where  $C$  is the compressive strength (MPa),  $P$  is the load at fracture (N), and  $d$  is the diameter of the cylindrical specimen (mm).

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