Evaluation of Nano-hydroxyapatite-silica-added Glass Ionomer Cement

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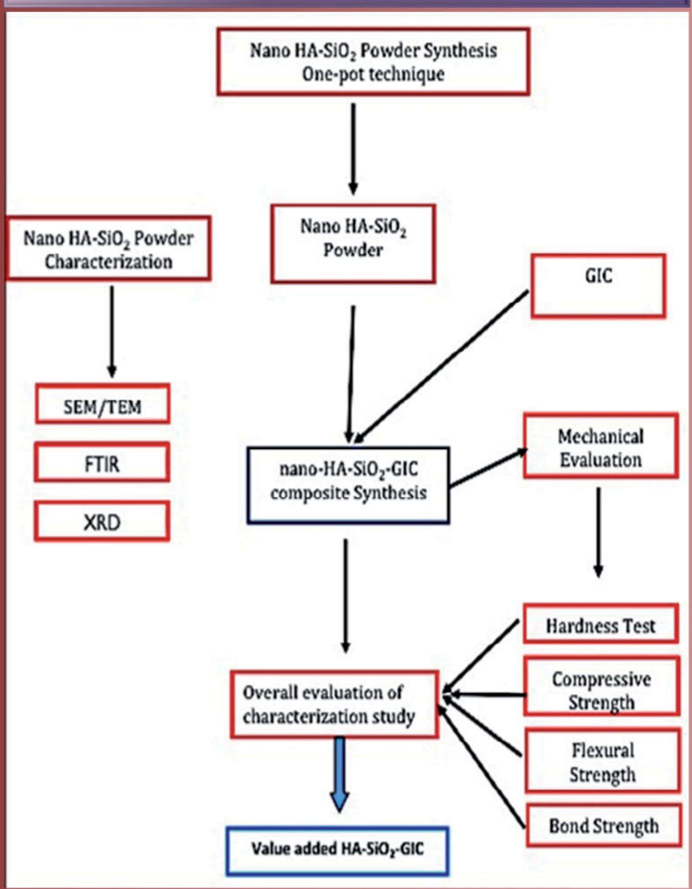
Introduction

- Glass ionomer cement has various uses in dentistry. They are used as restorative materials in paediatric dentistry, as a lining and base, fissure sealants, and atraumatic restorative treatment (ART) materials [1]. Most of the commercially available GIC fulfil the minimum criteria of ISO standard and are categorised as clinical grade GIC [2]. However, they are still mechanically weak and are not recommended for use under heavy occlusal load such as on the posterior teeth. It is predicted that the addition of nano-HA-Si to GIC matrix could produce a material with better mechanical properties. Therefore, a complete evaluation of nano-HA-silica-added GIC is crucial before any recommendations can be made .
- To the best of our knowledge, mechanical, physical, and chemical properties of nano-HA-silica-GIC are yet to be determined. Therefore, the aim of this study was to synthesise nano-HA-Si and to evaluate and compare the effect of the addition of nano-HA-Si as filler on the mechanical properties of nano-HA-Si-GIC in comparison to conventional GIC, in terms of its surface hardness, compressive strength, flexural strength, and shear bond strength.

Methods and Materials



Characterisation Studies

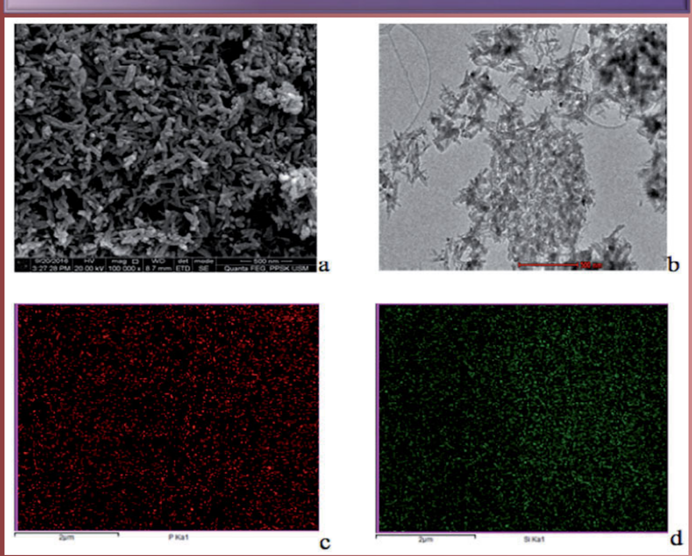


Figure 1. Morphology and dot mapping for HA-35SiO$_2$ (a) SEM Micrograph, (b) TEM Micrograph, (c) SEM dot mapping of phosphorus and d) SEM dot mapping of silicon

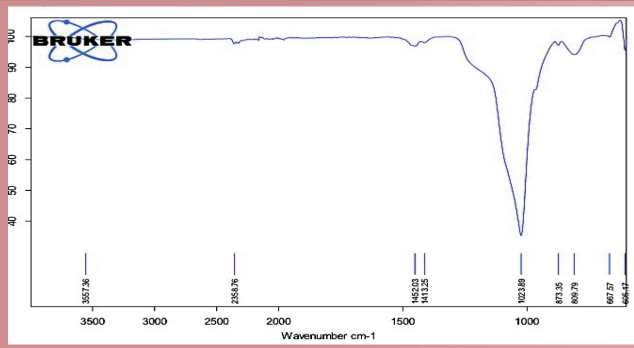


Figure 2. FTIR spectra of nano-hydroxyapatite-silica powder sintered at 600 C

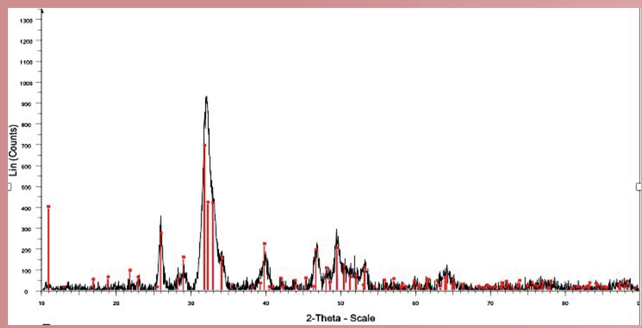


Figure 3. XRD traces of SiO$_2$ and hydroxyapatite after calcination at 600 C

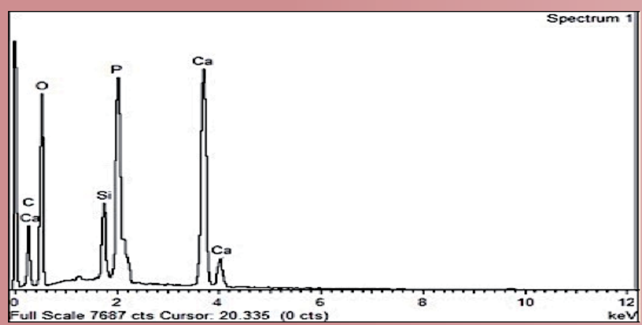


Figure 4. EDX spectrum of nano-hydroxyapatite-silica powder

Results

- It was found that the nano-powder consisted of a mixture of spherical silica particles (~50 nm) and elongated hydroxyapatite particles in the range between 100-200 nm.
- The results for the mechanical testing for various percentages of nano-HA-Si-GIC (Table 1) showed superior values as compared to the control. The maximum hardness value was achieved with the addition of 10% nano-HA-Si to GIC for all groups, with a decrease of hardness value at higher percentages, Fig. 5(a). A similar trend was seen for compressive and flexural strength, Fig. 5(b and c). 10% nano-HA-35SiO$_2$-GIC reported statistically higher mechanical properties as compared to 10% nano-HA-11SiO$_2$-GIC, nano-HA-21SiO$_2$-GIC, and the control (Table 1). Nano-HA-35SiO$_2$-GIC also exhibited statistically higher shear bond strength as compared to the conventional GIC (Table 2).

Table 1. The means and standard deviations of Vickers hardness, compressive strength, and flexural strength of nano-HA-Si-GIC

Vickers Hardness (HV)			
Percentage added	Nano- HA–11SiO$_2$-GIC	Nano- HA–21SiO$_2$-GIC	Nano- HA–35SiO$_2$-GIC
	Mean (SD)	Mean (SD)	Mean (SD)
0 (control)	47.59 (±6.82)^{a,1}	47.59 (±6.82)^{a,b,1}	47.59 (±6.82)^{a,b,1}
5	48.43 (±2.86)^{a,1}	51.9 (±5.36)^{a,b,1}	55 (±7.69)^{b,2}
10	50.66 (±3.32)^{a,1}	57.49 (±5.19)^{b,2}	64.77 (±6.18)^{c,3}
15	48.39 (±5.54)^{a,1}	49.92 (±3.61)^{a,b,1}	61.52 (±1.87)^{c,4}
20	38.96 (±4.78)^{a,2}	42.51 (±4.64)^{a,b,1}	57.33 (±4.51)^{c,5}
Compressive Strength (MPa)			
Percentage added	Nano- HA–11SiO$_2$-GIC	Nano- HA–21SiO$_2$-GIC	Nano- HA–35SiO$_2$-GIC
	Mean (SD)	Mean (SD)	Mean (SD)
0 (control)	119.82 (±20.36)^{a,1}	119.82 (±20.36)^{a,b,1}	119.82 (±20.36)^{a,b,1}
5	120.16 (±10.42)^{a,1}	123.67 (±23.1)^{a,b,1}	127.84 (±15.39)^{a,b,1}
10	125.36 (±17.56)^{a,1}	134.64 (±10.97)^{a,b,1}	143.42 (±13.94)^{b,c,2}
15	118.08 (±17.09)^{a,1}	122.82 (±13.16)^{a,b,1}	138.33 (±16.36)^{c,3}
20	90.21 (±20.33)^{a,2}	105.59 (±10.65)^{b,1}	136.19 (±6.4)^{c,4}
Flexural Strength (MPa)			
Percentage added	Nano- HA–11SiO$_2$-GIC	Nano- HA–21SiO$_2$-GIC	Nano- HA–35SiO$_2$-GIC
	Mean (SD)	Mean (SD)	Mean (SD)
0 (control)	11.53 (±1.63)^{a,1}	11.53 (±1.63)^{a,b,1}	11.53 (±1.63)^{a,b,1}
5	11.82 (±2.05)^{a,1}	12.16 (±1.64)^{a,b,1}	16.11 (±2.11)^{c,2}
10	12.03 (±1.13)^{a,1}	12.63 (±0.37)^{a,b,1}	17.68 (±1.81)^{c,3}
15	10.25 (±1.67)^{a,1}	12.21 (±1.68)^{b,1}	14.93 (±1.58)^{c,4}
20	8.39 (±1.09)^{a,2}	10.22 (±1.55)^{b,1}	13.58 (±1.6)^{c,5}

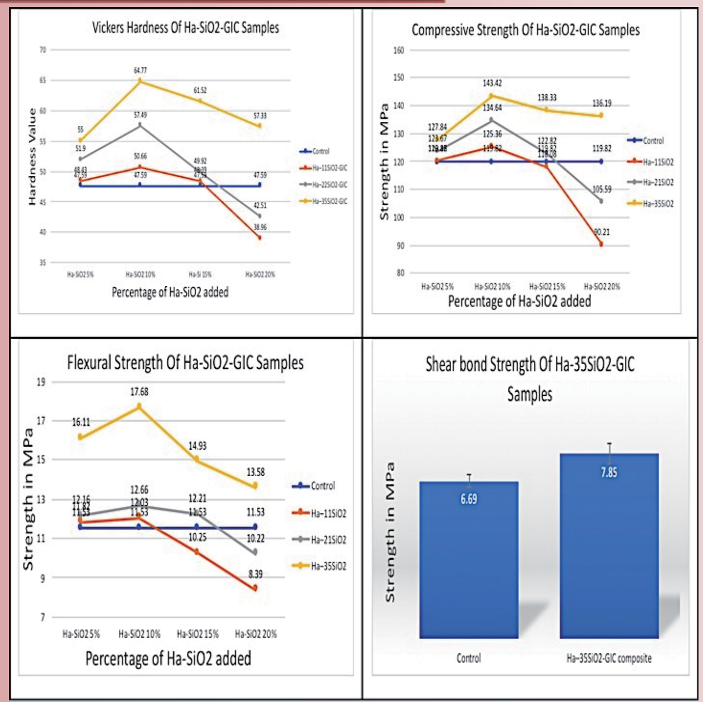


Figure 5. Mechanical properties. (a) Vickers hardness, (b) compressive strength, (c) flexural strength and (d) shear bond strength for nano-HA-35SiO$_2$-GIC

Discussion

- The increase in mechanical properties of the reinforced GIC could be related to the fact that the round silica particles fill the voids in elongated-shaped HA particles, Fig. 1(a) and 1(b), and by occupying the empty spaces in the GIC matrix. This leads to a better crack prevention mechanism, as the internal defects and pores of the GIC matrix are reduced, which prevents crack initiation. The results for mechanical testing reported in the current study are better than the earlier published work for HA-added GIC [3-6].
- The mechanical properties of all the tested material decreased with the addition of higher percentages (15 and 20%) of nano-HA-Si to GIC. This could be due to over crowding of filler particles as well as high powder/liquid ratio leading to dry mix. The increase in nano particles can also result in a decreased intersection between the ionomer network and nano-particles, thus weakening the tested material.

Table 2. Shear bond strength of 10% nano-HA-35SiO2 added GIC

Shear Bond Strength (MPa)			
Materials	Mean	SD	P-value
Control (conventional GIC)	6.69	1.06	0.030
10% Nano-HA–35SiO2-GIC	7.85	1.65	

Conclusions

- The characterisation of nano-hydroxyapatite-silica powder from SEM and TEM shows that the morphology of this nano-powder consists of elongated particles of HA surrounded by spherical silica particles. The mechanical properties improved with addition of 10% HA-35SiO$_2$ to the GIC. There was a ~36%, ~19.7%, and ~53.4% increase in the Vickers hardness, compressive strength, and flexural strength, respectively., Shear bond strength was also enhanced by ~17.34 % in comparison to conventional GIC.
- Enhanced mechanical properties can be attributed to denser packing of the hydroxyapatite-silica-glass ionomer cement matrix. The addition of nano-HA-silica to conventional GIC significantly enhanced the mechanical properties of the material. Hence, it can be suggested as a potential dental restorative material in dentistry.

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