

# Comparison between differently synthesized hydroxyapatite composites for dentistry applications

H.K.G.K.D.K. Hapuhinna\*, R.D. Gunaratne\*, H.M.J.C. Pitawala\*\*

\* Department of Engineering Technology, University of Sri Jayewardenepura, Sri Lanka

\*\* Department of Science & Technology, Uva Wellassa University, Sri Lanka

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**Abstract-** Hydroxyapatite and its composites have applied in several biomedical applications as a bioceramic. This research used to examine chemical and structural suitability of newly synthesized Eppawala hydroxyapatite composite varieties as dental filling material, by comparing and contrasting them with human tooth as well as commercially available glass ionomer cement (GIC), used in dentistry applications. Commercially available 2-hydroxyethyl methacrylate monomer used to reinforce solid state sintered and sol gel synthesized hydroxyapatite ceramic to prepare its composites and their physical, chemical properties were compared. Results show there is a close similarity between synthesized products and human tooth while crediting high thermal stability, good crystalline, and porous properties than the commercial product. Finally, study concluded newly synthesized composites can be applied directly as a substitution for human tooth while having different properties from each other.

**Index Terms-** Hydroxyapatite, Dental fillings, 2-hydroxyethyl methacrylate, Human tooth

## I. INTRODUCTION

Hydroxyapatite is a widely used bioceramic which has a close chemical and structural similarity with human hard tissues and performs several outstanding properties biocompatibility, non-inflammatory in nature, osteoconductivity, non-toxicity, bioactivity etc. [1-11]. As a result it has a range of biomedical applications mainly in the fields of orthopedics and dentistry. [12-20]

Here in this study we have synthesized hydroxyapatite by converting chlorapatite using solid state sintering method and sol gel technique considering its ability to replace chlorine with other groups due to the increase of reactivity as its chlorine positions are under strain in the structural framework. [21,22] Chlorapatite were collected from Sri Lankan Eppawala apatite deposit, which usually contains 34-40% total phosphorus expressed as percentage of Phosphorus pentoxide ( $P_2O_5$ ). [21-25] Apart from that, for sol gel technique, ethanol and dil. nitric acid were used and for solid state sintering technique Calcium hydroxide was used. Further synthesized hydroxyapatite is reinforced with a reactive resin, 2-hydroxyethyl methacrylate. It is a hydroxyester and a monomer resin widely used as a desensitizing agent for dentistry applications, as its ability to

stimuli pain by get sealing into sensitive areas in the teeth and blocking the dentinal tubules at the dentin surface. [26]

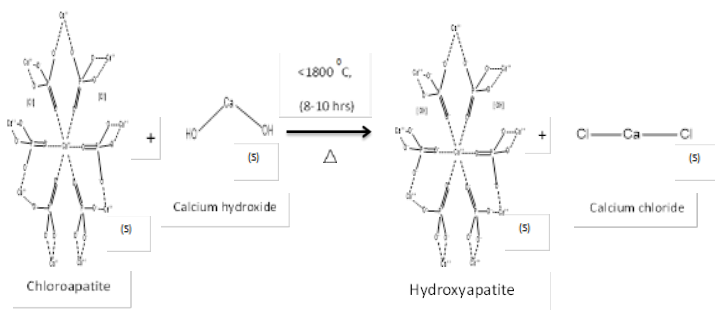
This study mainly focused to find out structural suitability of synthesized hydroxyapatite composites as dental filling material, by comparing and contrasting it with human tooth as well as commercially available glass ionomer cement, which is currently used for dental applications in Sri Lanka. Selected commercial product containing two main parts as GIC majorly consists of fluoroaluminosilicate glass and 2-hydroxyethyl methacrylate polymer, is currently used self-curing conventional glass ionomer restorative material in Sri Lankan government hospitals which indicates for non-stress bearing restorations, deciduous teeth restorations, geriatric restorations, intermediate restorative and base material for cavities using sandwich technique, cervical restorations, cold build ups, temporary fillings and dentine replacement etc. It is prepared directly before use by mixing its powder component with polymer component clinically and resulted paste is cured within a few seconds. [27]

## II. EXPERIMENTAL PROCEDURE

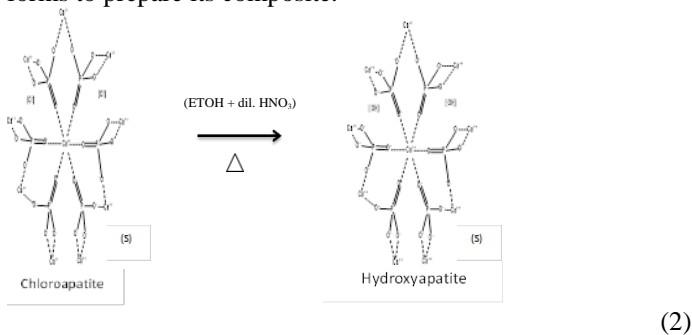
### A. Sample preparation

As the first step, natural raw apatite mineral were collected from the Eppawala apatite site. Then they were sorted as high grade rock phosphate (HERP) by the visual appearance of less coated apatite. After removing mud, collection of apatite rocks were dried under sunlight, crushed using a jaw crusher (Serial no: 1720011, China) into small crystals /powder, grind further into micron/Nano level HERP powder using a planetary Ball Mill (XQM – 4.0A) and sieved using sieve set (A060\_01AC/0219, Scotland). Less than 63 micron range particle size powder were collected and oven dried at a temperature around 120 °C for 5 hrs to prepare moisture removed HERP powder (MHERP).

Solid state sintered composite sample was prepared as mentioned bellow. MHERP powder was added with needed weight of  $Ca(OH)_2$  powder, after well mixing, sieving and high temperature heat treating solid state sintered Eppawala hydroxyapatite powder (SSHAp) was synthesized according to equation (1). [12,22] Then the synthesized ceramic powder was mixed with commercial methyl methacrylate (MMA) liquid monomer, until the ductile dough forms to obtain its composite.



Sol gel synthesized Eppawala hydroxyapatite (SGHAp) was prepared under sol gel technique acidified route using MHERP, absolute ethanol, and diluted acid as the raw materials as mentioned in equation (2). Mixture of MHERP and dil. Acid (1:1 ratio) was stirred well in absolute ethanol medium for 4-6 hrs until the formation of gel. Then it was oven dried to a temperature less than 120 °C for 15 hrs and again two stage heat sintering were done starting from 400 °C to 750 °C for 8hrs. [12,22] Finally the synthesized ceramic powder was mixed with commercial 2-hydroxyethyl methacrylate polymer until a paste forms to prepare its composite.



As the fourth step, commercial GIC powder and 2-hydroxyethyl methacrylate were mixed together until a paste forms, that sample was used to compare synthesized ceramic composite.

### B. Sample characterization

Before mixing with the polymer, commercial GIC powder and Eppawala hydroxyapatite was examined under X-ray fluorescence spectroscopy (Rigaku XRF Spectrometer) to find out its elementary composition and presence of impurities. The polymer was identified using Fourier transform infrared spectroscopy (Bruker – Alpha FTIR Spectroscopy) under ATR technique. Then the mixtures of newly synthesized product and commercial product along with the human tooth were characterized using XRD, FTIR, TGA, and SEM with EDS techniques. The crystallographic phases of samples were determined by X-ray diffractometer (Rigaku – Ultima. IV diffractometer) in reflection mode with Cu K $\alpha$ 1: 0.154 nm radiation. 1.5<sup>0</sup> min<sup>-1</sup> scanned speed was used to collect data within 2 $\theta$  range from 15<sup>0</sup> to 80<sup>0</sup> angles. The presence of functional groups was confirmed by FTIR over the region 400-4000 cm<sup>-1</sup> using KBr pellet technique. The resolution of the spectrometer was 4 cm<sup>-1</sup>. The surface morphology and microstructural features of samples were studied using Hitachi SU6600 analytical variable pressure field emission scanning electron microscope

(FE-SEM) and oxford instruments EDX with AZtec software. Furthermore, thermogravimetric analysis (TGA) was done using a thermal analyzer (SDT Q600) with N environment, 10 °C min<sup>-1</sup> heating rate, and 1450 °C maximum temperature to find out the thermal stability of samples.

### III. RESULTS AND DISCUSSION

Both solid state sintered Eppawala hydroxyapatite (SSHAp) and sol gel synthesized Eppawala hydroxyapatite (SGHAp) powder contain Ca, P and O include in higher weight percentages and Fe, Al and Si as the impurities with hexagonal crystal structure showing a close similarity with mammalian tooth and consists of many correlated, microcrystalline structures/particles/ spherulites while credenting good thermal stability. Specially, SSHAp powder interprets porous properties and SGHAp powder interprets highly crystalline structure. [12,22]

Table 1. XRF results for Commercial Glass Inomer Cement

Element	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5	Spot 6
	Mass %	Mass %	Mass %	Mass %	Mass %	Mass %
13 Al	49.46	42.39	43.03	45.82	43.29	43.13
16 S	0.30	-	-	-	-	0.25
20 Ca	0.48	0.35	0.39	0.32	0.31	0.30
26 Fe	0.23	0.17	0.20	0.17	0.18	0.20
38 Sr	49.34	56.79	55.36	53.29	55.94	55.87
40 Zr	0.19	0.20	0.27	0.28	0.27	0.16
56 Ba	-	0.10	0.74	0.12	-	0.08

Table 1 shows that sample contains Al and Sr in higher amounts and Zr, S, Fe, Ca and Ba in less amounts. [26]

Table 2. SEM with EDS results for Solid state product with mixture, results for Sol gel product with polymer mixture, commercial GIC with polymer mixture and Human tooth

Element	Solid state sintered product with polymer mixture			Sol gel product with polymer mixture			Commercial GIC with polymer mixture			Human Tooth		
	Spo t 1	Spo t 2	Spo t 3	Spo t 1	Spo t 2	Spo t 3	Spo t 1	Spo t 2	Spo t 3	Spo t 1	Spo t 2	Spo t 3
	Wt %	Wt %	Wt %	Wt %	Wt %	Wt %	Wt %	Wt %	Wt %	Wt %	Wt %	Wt %
O	58.4	60.0	60.1	61.4	58.9	55.4	53.5	57.4	57.0	63.7	58.3	49.2
C	16.0	17.0	17.2	17.7	16.5	20.5	13.8	17.3	17.1	19.4	14.9	7.4
Ca	18.1	15.8	15.9	13.6	15.8	12.6	-	-	-	10.2	17.3	28.9
P	6.7	6.4	6.2	6.8	7.6	10.5	0.8	0.5	0.5	5.2	8.8	13.8
Cl	0.7	0.7	0.6	0.6	0.8	0.9	-	-	-	-	-	-
Al	-	-	-	-	0.3	-	7.3	5.1	5.2	0.3	-	-
Fe	-	-	-	-	0.1	-	-	9.8	-	0.2	-	-
Sr	-	-	-	-	-	-	7.2	4.8	4.9	-	-	-
Si	-	-	-	-	-	-	6.4	4.3	4.4	0.5	-	-
Na	-	-	-	-	-	-	1.0	0.8	0.7	0.3	0.3	0.7
F	-	-	-	-	-	-	9.8	-	10.1	-	-	-
Co	-	-	-	-	-	-	0.1	-	-	-	-	-
Mg	-	-	-	-	-	-	-	-	-	0.2	0.4	-

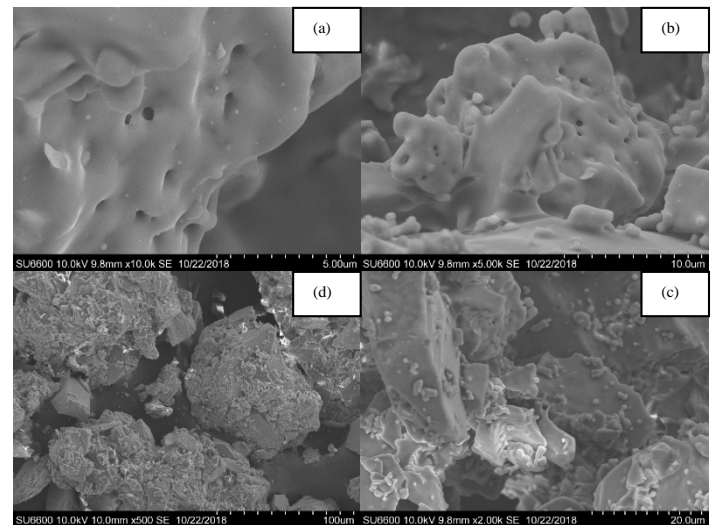


Figure 1. SEM images for SSHAp with polymer mixture, 10.0 kv, a) 10K b) 5KX c) 2KX d) 500X

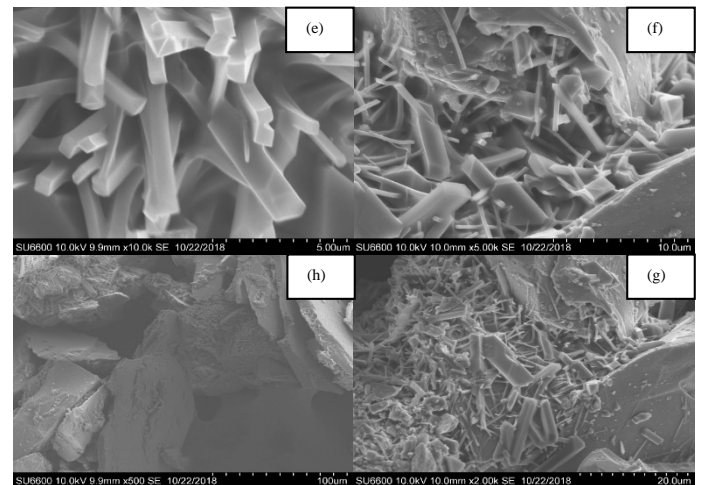


Figure 2. SEM images for SGHAp with polymer mixture, 10.0 kv, e) 10K f) 5KX g) 2.2KX h) 500X

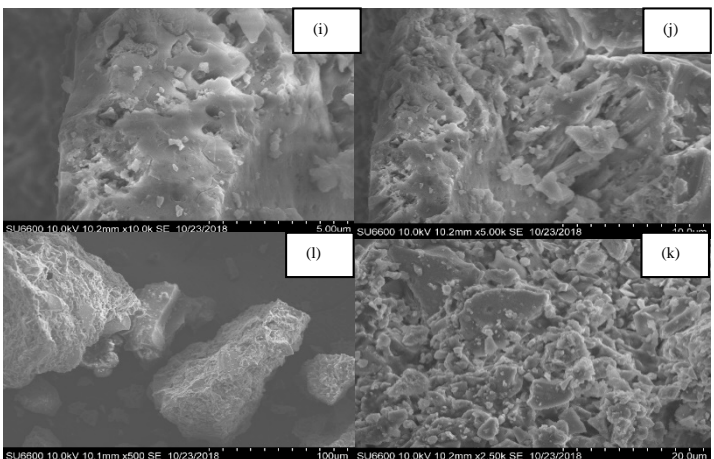


Figure 3. SEM images for Commercial GIC with polymer, 10.0 kv, i) 10KX j) 5KX k) 2.2KX l) 500X

Results for SEM with EDS analysis of solid state product with polymer mixture, sol gel product with polymer mixture, commercial GIC with polymer mixture and human tooth are given in the Table 2. According to that; SSHAp with polymer sample contains O, Ca in higher amounts and then P, C, Cl in order. SSHAp with polymer sample contains O in higher amounts and then C, Ca, P, Cl in order and also Fe in very less amount as an impurity. Commercial product mixture contains O, C presence in higher amounts of the products and in order F, Al, Sr, Si, N, P and negligible amount of Ca. Human tooth contains O presence as the highest amount and then C, Ca, P and Si in order. Na, Al, Fe, Mg presence in very fewer amounts. When comparing results, it can be stated that both SGHAp with polymer and SSHAp with polymer mixtures have similarity with human tooth in composition. Among synthesized composites SSHAp with polymer sample contains more Ca percentage, which may occur due to the addition of Calcium hydroxide at the beginning for preparation of ceramic.



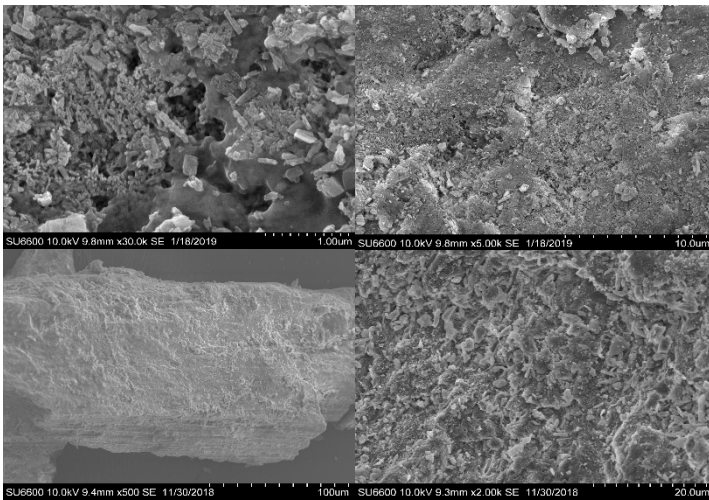


Figure 4. SEM images for Human tooth, 10.0 kv, i) 30KX j) 5KX k) 2KX l) 500X

Considering Figure 1-4; SEM images of all mixtures and human tooth show that there are good correlations of particles. SSHAp with polymer mixture and human bone only carried out micropores as shown in the Figure 1. Porosity would be helpful for tooth ingrowth as well as for good blood circulation to the tooth. Figure 2 carries good microcrystalline needle shapes particles for SGHAp product mixture. Commercial GIC mixture shows microporous structure as mentioned in Figure 3. According to Figure 1, 2 & 4, some crystalline property can be found in human tooth, both SSHAp with polymer and SGHAp with polymer mixtures. Among them crystallinity is higher in SGHAp mixture.

Figure 5 shows that the resulted graph for polymer it has coincided with the FTIR characteristic graph for 2-hydroxyethyl methacrylate, therefore polymer in the commercial product was confirmed as the 2-hydroxyethyl methacrylate polymer. It interprets several peaks related to stretching vibrations including a sharp intense peak at  $1731\text{ cm}^{-1}$  related to the presence of ester carbonyl group, broad peak nearly  $1150\text{ cm}^{-1}$  due to the C-O (ester bond) and a peak nearly  $1250\text{ cm}^{-1}$  is due to the vibrations of C-C bond. Also literature shows the broad peak ranging from  $(3000-3500)\text{ cm}^{-1}$  is owing to the presence of stretching vibration.<sup>[27]</sup> As shown in the Figure 6, all peaks for phosphate groups in the  $560\text{ cm}^{-1}$ ,  $640\text{ cm}^{-1}$ ,  $963\text{ cm}^{-1}$ ,  $1028\text{ cm}^{-1}$  and  $1110\text{ cm}^{-1}$  wave no range and characteristic peak for OH/ Hydroxyapatite nearly  $3572\text{ cm}^{-1}$  wave no appeared in the Human Tooth as well as the SSHAp with polymer and SGHAp with polymer mixtures.<sup>[12,22]</sup> It confirms that even after the mixing, the presence of hydroxyapatite in both SSHAp product and SGHAp product. When considering commercial product mixture, it shows peak nearly  $3572\text{ cm}^{-1}$  wave no range, which may due to the presence of OH<sup>-</sup> group, but that couldn't be identified as hydroxyapatite characteristic peak, as no peaks found related to phosphate groups. Apart from that, some peaks can be found commonly in both commercial product mixture, SSHAp mixture and SGHAp mixture except in human tooth nearly  $750\text{ cm}^{-1}$ -  $2000\text{ cm}^{-1}$  and  $3000\text{ cm}^{-1}$  wave no range in Figure 6, they are the related peaks for 2-hydroxyethyl methacrylate. As a result it can be concluded that both commercial product, SSHAp and SGHAp mixed well with the polymer.

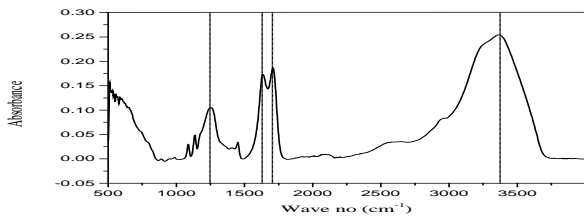


Figure 5. FTIR graph for polymer

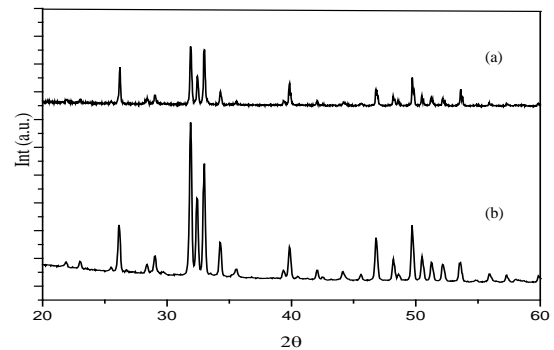


Figure 7. XRD pattern for (a) SSHAp with polymer mixture (b) SGHAp with polymer mixture

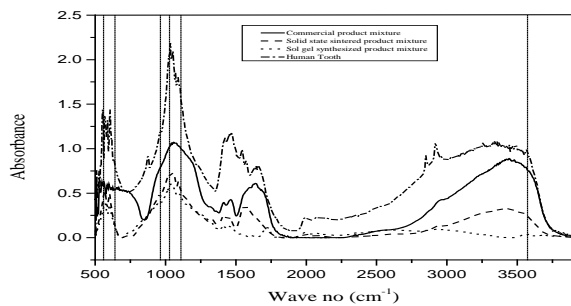


Figure 6. FTIR comparison for Commercial GIC with polymer mixture, SSHAp with polymer mixture, SGHAp with polymer and Human tooth

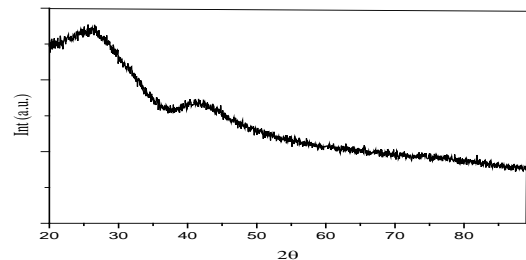


Figure 8. XRD pattern for Commercial GIC with polymer mixture

Figure 7 (a) & (b) explains even after mixing 2-hydroxyethyl methacrylate polymer, XRD results of both SSHAp and SGHAp mixtures have shown all characteristic peaks related to the crystallographic phases 002, 210, 211, 112, 300, 202, 310, 222, 213 and 004 of hexagonal hydroxyapatite, which shows similarity to human tooth. [12,22,28,29,30,31,32,33] Comparing those results with Figure 8, XRD pattern for commercial GIC with polymer it has carried out an amorphous structure without crystalline properties.

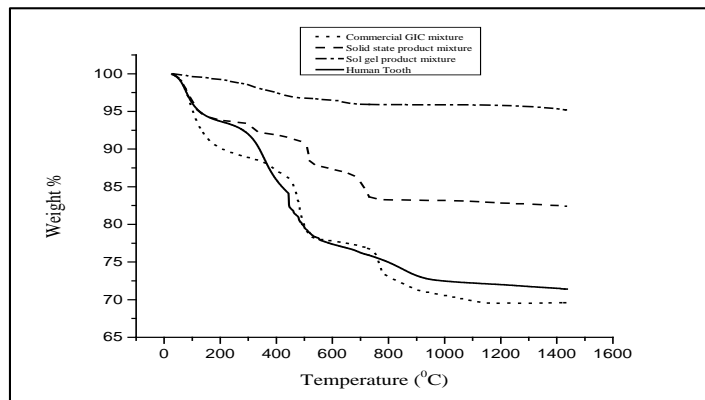


Figure 9. Comparison between TGA results of Human Tooth, Commercial product mixture, Sol gel product mixture and Solid state product mixture

When comparing TGA results for human tooth, commercial GIC with polymer mixture, SSHAp with polymer mixture and SGHAp with polymer mixture according to Figure 8, human tooth, SSHAp and SGHAp mixtures have shown the same pattern of weight loss, which was slightly different from commercial product mixture. At the beginning they show weight losses nearly 100 °C to 400 °C which may associate with the dehydration of samples, following that again samples have reduced their weight nearly 600 °C to 700 °C which may occur due to the gas elimination. Then again from 700 °C up to 1400 °C weight losses have occurred due to the incipient transformation of produced hydroxyapatite in  $\beta$ -TCP. [12,22,28,30,31,32,33]

Therefore, it indicates the presence of hydroxyapatite in products as well as in human tooth. Those results confirm the composition similarity of human tooth and synthesized composite mixtures, as they were containing hydroxyapatite.

Also due to the least amount of weight loss in synthesized composite mixtures than human tooth and commercial product mixtures, it can be concluded that the synthesized SSHAp with polymer and SGHAp with polymer mixtures perform high thermal stability and good material stability in nature and application.

#### IV. CONCLUSION

The study concludes that SSHAp with 2-hydroxyethyl methacrylate and SGHAp with 2-hydroxyethyl methacrylate composites have chemical and structural similarity with human tooth and perform high thermal stability and good material stability in nature. Also SGHAp composite interprets microporous structure with less than 50 micron range particles and SSHAp composite consists highly crystalline particles.

Therefore, both resulted composites can be used as a direct substitution for human tooth.

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#### AUTHORS

**First Author** – H.K.G.K.D.K. Hapuhinna is with Department of Engineering Technology, University of Sri Jayewardenepura, Sri Lanka, [khapuhinna@gmail.com](mailto:khapuhinna@gmail.com)

**Second Author** – R.D. Gunaratne is with Department of Engineering Technology, University of Sri Jayewardenepura, Sri Lanka, [rajitha@sjp.ac.lk](mailto:rajitha@sjp.ac.lk)

**Third Author** – H.M.J.C. Pitawala is with Department of Science & Technology, Uva Wellassa University, Sri Lanka

**Correspondence Author** – R.D. Gunaratne (Rajitha Deshapriya Gunaratne), [rajitha@sjp.ac.lk](mailto:rajitha@sjp.ac.lk), +94705758877