

Preparation and Characterization of Phosphate Based Glasses

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Abstract - A novel magnetic phosphate based glass material was developed by Fe ions. The phosphate glass (PG) and magnetic phosphate glass (MPG) were prepared employing melt - quenching technique. The effect of heat treatment was analyzed for both PG and MPG at 400°C and 600°C. The crystalline phase, functional group analysis, surface morphology and elemental composition of the synthesized samples were evaluated using XRD, FT - IR, FE - SEM and EDX respectively.

Key Words: phosphate glass, magnetic phosphate glass, met- quenching.

1. INTRODUCTION

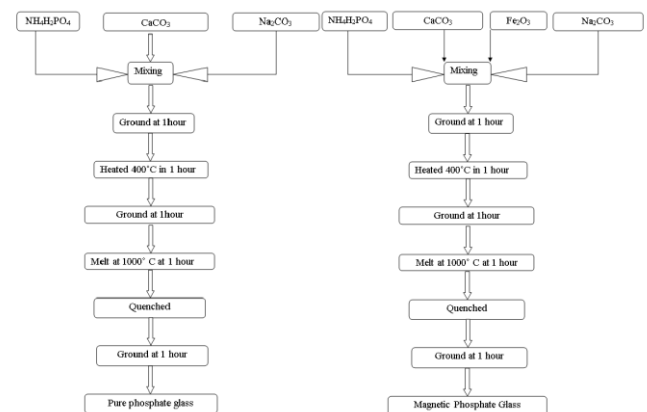
The materials which can withstand and perform their task properly within the alive body without any harmful effects on surroundings are known as biomaterials. Bio-materials are divided into four categories Bio inert materials, Bioactive materials, Biodegradable materials and Porous materials [1]. Since Hench et al. First discovered Bio - glass in 1970s, large number of studies have focused on bioactive glasses and glass ceramics [2]. The development of glass- ceramics with good bioactivity and magnetic property has attracted much attention in recent decades [3]. Magnetic bioactive glass ceramics are specially designed to restore bone tissue after tumour extirpation [4].

2. Materials and Methods

2.1. Preparation of Glass Sample

Phosphate glasses $45P_2O_5-34CaO-21Na_2O$ [PG] and $45P_2O_5-34CaO-16Na_2O-5Fe_2O_3$ [MPG] (mol. %) was prepared for composition using $NH_4H_2PO_4$ (99.999%), $CaCO_3$ (99.995%), Na_2CO_3 (99.9%), Fe_2O_3 (99.99%) chemicals with melt-quenching technique. The amounts of precursors were weighed using digital electronic balance. The chemicals were then mixed and grinded in a mortal pestle [5]. The mixed powder of these precursors were placed in silica crucible and melted in electric resistance furnace. The powder of

precursors were initially kept at 400°C for 1 hour for calcination to release water from the starting materials then they were reheated at 1000°C and kept at this temperature for 1 hour in order to achieve the homogeneity. The melt was poured on the flat stainless steel plate in air to obtain glass. The steps involved in this process was summarized in flow chart 1.



Flow Chart -1: (PG) (MPG) glass preparation

3. Results and discussion

3.1. X-RAY diffraction analysis

The XRD data was collected for the 2θ range $10^\circ-60^\circ$ for a step size of 0.02° . The XRD pattern of as prepared PG and MPG were shown in Figure1. The absence of sharp peaks and presence of broad humps confirms amorphous nature of glasses and the characteristic 'amorphous hump' was detected between $20^\circ - 35^\circ$ in the produced glasses [6].

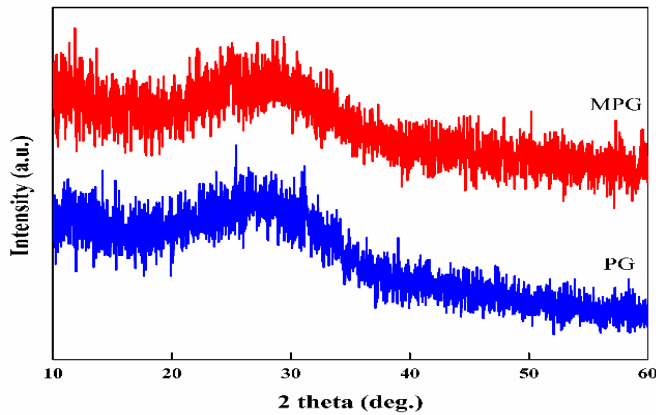


Fig -1: XRD pattern of PG and MPG samples

The PG and MPG samples heat treated at 400 and 600 °C were shown in Fig.2 and 3 respectively. Heat treatment resulted in the emergence crystalline phases in the PG and MPG samples. The PG sample sintered at 400 °C exhibited β -calcium pyrophosphate (β - $\text{Ca}_2\text{P}_2\text{O}_7$) (JCPDS No: 81-2257) as the major phase [7]. In the case MPG, phases like β - $\text{Ca}_2\text{P}_2\text{O}_7$ and iron oxide (Fe_2O_3) (JCPDS No: 89-7047) were observed [8].

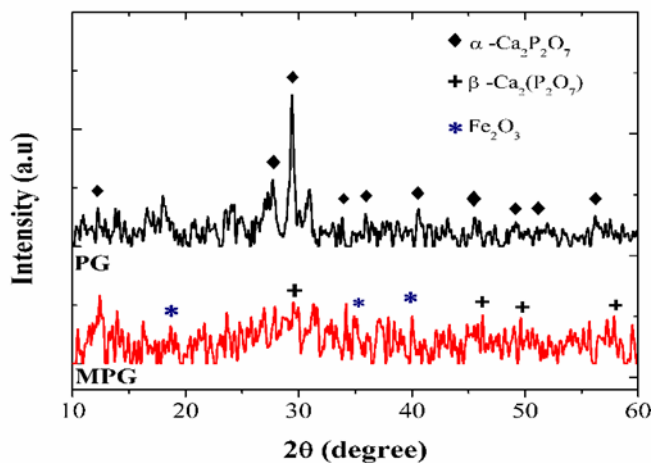


Fig -2: XRD pattern of PG and MPG samples sintered at 400°C.

When the sintering temperature was increased to 600 °C, phases like - calcium sodium phosphate (NaCaPO_4) (JCPDS No: 76-1456) and (β - NaCaPO_4) (JCPDS No: 29-1193) became the major components in the sintered PG sample [9]. Additionally iron oxide (Fe_2O_3) (JCPDS No: 89-7047) phase along with NaCaPO_4 (JCPDS No: 76-1456) were observed in the MPG sample [10]. These results suggest that the prepared samples having multi-phase microcomposite behavior.

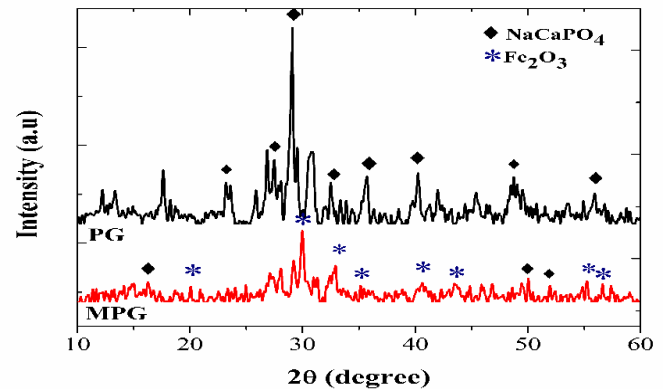


Fig -3: XRD pattern of PG and MPG samples sintered at 600°C.

3.2. Fourier Transform Infrared Spectroscopy

FTIR spectra of PG and MPG were shown in Figure 4. The frequency of the predominant characteristic bands observed along with assignments in PG and MPG. The pattern that is observed indicates the presence of phosphate groups like P-O-P, PO_2 and PO_3^{2-} in the samples [11].

The bands at 535 cm^{-1} and 750 cm^{-1} were attributed to the symmetric stretching vibrations of PO_3^{2-} of the phosphate group in the PG and MPG samples [12].

The PO_2 asymmetric characteristic vibration is observed at 1269 cm^{-1} and 1526 cm^{-1} . The bands at 727 cm^{-1} , 775 cm^{-1} and 1097 cm^{-1} were ascribed to the symmetric stretching vibrations of P-O-P of the phosphate group in the glass samples. The wave number 893 cm^{-1} corresponds to the asymmetric stretching vibrations of P-O-P of the phosphate group. The O-H groups were observed between $3400\text{--}3800\text{ cm}^{-1}$ wave number regions [13].

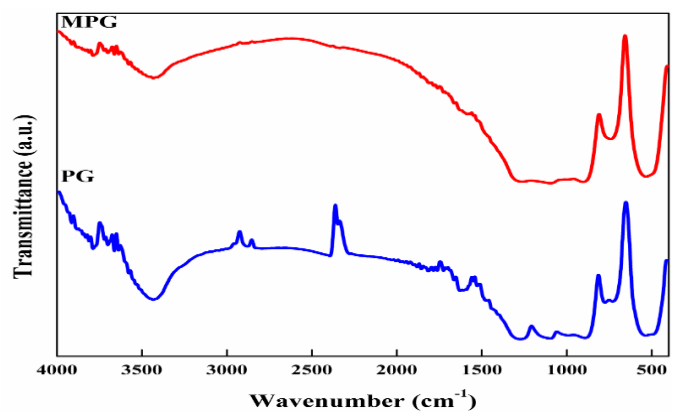


Fig -4: FTIR spectra of PG and MPG glass.

3.3. Scanning Electron Microscope and EDX Analysis

The Field Emission Scanning Electron Microscopy (FE-SEM) is a powerful tool to carry out the morphology of the materials such as crystals, glass, powder and so on. Figure 5 shows the morphology of the PG and MPG samples. FE-SEM images revealed that PG and MPG samples possessed non-spherical, irregular and angular morphology nature. The particle size varied between 1 – 5 μm for the PG and MPG [14].

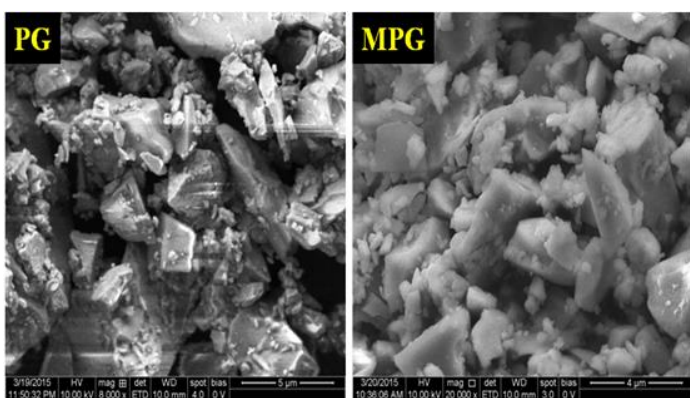


Fig -5: FE-SEM images of PG and MPG samples.

Elemental analysis of glass samples has been done using EDX. Existence of elements which are expected in structure of glass is confirmed. The EDX spectra of the glass samples were shown in Figure 6. The presence of P, O, Ca and Na in the PG sample is depicted in the EDX spectra. The P, O, Ca, Na and Fe elements were observed in the MPG sample. The atomic and weight percentage of the materials present in the PG and MPG samples were listed in Table [15].

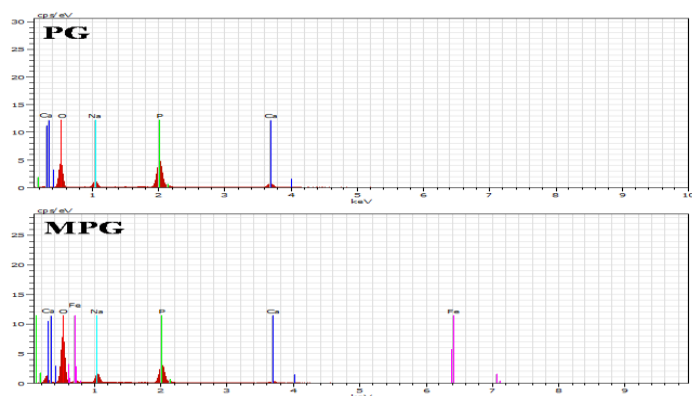


Fig -6: EDX spectrum of PG and MPG samples.

Table -1: Elemental analysis of PG MPG samples

Element	Weight %		Atomic %	
	PG	MPG	PG	MPG
O	49.64	60.44	66.29	75.72
P	29.86	19.00	20.60	12.30
Ca	15.00	8.89	8.00	4.45
Na	5.50	6.53	5.11	5.69
Fe	-	5.14	-	1.84
Total	100.00	100.00	100.00	100.00

4. CONCLUSIONS

We have successfully synthesized Phosphate based glasses [PG] and [MPG] were prepared by melt-quenching technique. The effect of heat treatment has been analyzed for both PG and MPG samples at 400°C and 600°C respectively. From X-ray diffraction (XRD) analysis the amorphous nature of the as prepared material is confirmed. The absence of sharp peaks and presence of broad humps confirmed the amorphous nature. After heat treatment, the XRD pattern exhibits multi-phase micro composite contains α -calcium phosphate, β -calcium phosphate, calcium sodium phosphate and iron oxide for PG and MPG samples. FT-IR spectra indicated the presence of phosphate groups such as P-O-P, PO₂ and PO₃²⁻ in the samples and that showed a strong band at 535cm⁻¹ and 750cm⁻¹ which is characteristic of PO₃²⁻ vibrational band. FESEM analysis revealed that the PG and MPG samples possessed non-spherical, irregular and angular morphology nature. EDX confirms the presence of chemical constituents, like P, O, Ca, Na and Fe elements.

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One Day International Seminar on Materials Science & Technology (ISMST 2017)**4th August 2017****Organized by****Department of Physics, Mother Teresa Women's University, Kodaikanal, Tamilnadu, India**

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