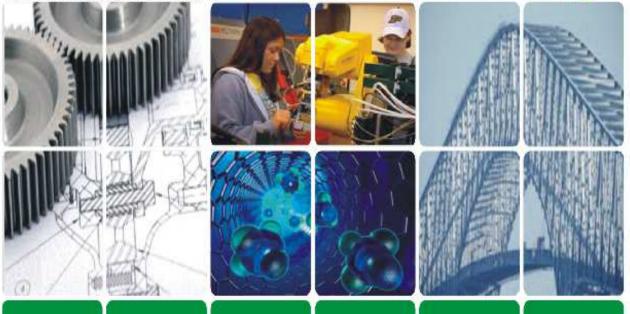
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Message

Scientific discoveries and advancement affect our lives by providing new policies and regulations that provide broad national direction and by new products that enhance our lives. Technology and engineering translate scientific knowledge into action. At the same time, technological innovations often require further research into materials, devices and processes. Engineers use the knowledge of science, mathematics, economics and appropriate experience to find suitable solutions to the problems and helps in creating an appropriate mathematical model for analysis.

This special issue on Engineering and Technology 2012 of Anvikshiki brings together the latest developments in technology and gives a base for the future work to be done in respective areas.

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I express my sincere gratitude to the editorial board of prestigious journal ANVIKSHIKI for believing in my technical competencies and choosing me as a reviewer of special issue on Engineering and Technology 2012. I understand that with great role comes great responsibilities. I will try to fulfill this highly valued responsibility with best of my technical knowledge and human values. This journal has been a guiding beacon for scientific community for numerous years & has gained the prestige due to it's original & rich articles. The contribution of ANVIKSHIKI in field of scientific research is immense.

I wish for the phenomenal success of special issue on Engineering and Technology,2012 of ANVIKSHINKI.

P K S Dikshit Professor Department of Civil Engineering Institute of Technology Banaras Hindu University Varanasi 221005

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Editorial Note

As my nomination as an Subject Expert and Editor for this Special Issue on Engineering & Technology 2012, I have worked a lot to make it successful. I do whatever task is at hand to the best of my ability. I take pride in my work and give hundred percent every time. For those submissions that were not suitable for publication, we tried to let authors know very quickly of our decision, giving them a chance to submit their manuscript to another journal if they so desire. I am fully aware that the prestige and quality of an ANVIKSHIKI Journal depends upon the altruistic participation of reviewers and the fairness and promptness with which the review process is conducted. In this regard, I wish to express my sincere gratitude to all board members for their nice cooperation and sustained effort. However, because of the increased number of submissions and the diversity of research fields involved, we have a difficult task ahead of us requiring a more rapid tempo of review. At the same time, from now on the authors themselves should assume their own inescapable responsibilities. The editor will return immediately any manuscript that is incomprehensible to reviewers on account of substandard grammar and syntax.

Finally, it is a pleasure to thank my Editor in chief for their nice cooperation and valuable suggestion. Now, we all look forward to embarking in a journey that can take ANVIKSHIKI on to the next plateau of excellence.

I hope you will enjoy reading this issue and we welcome your feedback .

With best regards,

Syster & Sovastora

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STUDIES ON PREPARATION AND CHARACTERIZATION OF PHOSPHATE CONTAINING BIOGLASS-CERAMICS

AJAY KUMAR*

Declaration

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Abstract

This study explores the interaction between bioactive glasses and dentin from extracted human teeth in simulated oral conditions. Bioactive glasses in the Na2O- CaO- SiO2-P2O5 and MgO- CaO- SiO2- P2O5 system were prepared as polished disks¹. The primary aim of the project was to develop better bio-glass-ceramics, which are bio-chemically compatible and non-toxic and to study the effect of doping agent on the physic-chemical properties of 45S5 bioactive glass², so that the end material should be bio-mechanically compatible in their mechanical harmonization with surrounding tissue, and bio-adhesive in their adhesion between the material and living tissues ^{3,4}. Generally, when a glass ceramic is chemically attacked, the initial effect is upon the glassy phase present. So by the addition of cerium oxide in the phosphate glass, the effects of it on chemical durability (dissolution) and mechanical properties through the analysis of glass sample using DTA, TGA, XRD, FTIR, density measurement, compressive strength measurement.

1. Introduction

Hench &Paschall ^{1,5} used a glass with soluble additives and called it bio-glass. According to their report insertion of this material into the bone, where it is soluble in body fluids, resulted in the silica gel formation around the bio-glass and promoted collagen formation.June Wilson et al. ⁶ had studied the toxicology and biocompatibility of 45S5 and 45S5F bio-glasses. In vitro test were carried out in Rat bone cells, rat fibroblasts, human lymphocytes, chick fibroblasts, mouse macro phases. U.Gross and his colleagues ⁹ had carried out bio-compatibility test with two glasses consisting of SiO₂, Na₂O, CaO, Ca₃(PO4)₃, MgO, K2O and SiO², Ca(PO₃)2, Na₂O, CaO systems⁸. The test carried on pigs, rats and human were observed to show astonishing similarity in tissue reaction of different species.Ceramics

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used for the repair and reconstruction of diseased or damaged part of the musculoskeletal system, termed bioceramics, may bioinert (alimina, zirconia), restorable (tri-calcium phosphate), bioactive (hydroxyapatite, bioactive glasses and glass ceramics),or porous for tissue in growth(hydrxyapatite-coated metals ,alumina)^{10,11}.

The bioactive glasses can be employed to repair and to rebuild damaged tissues, particularly hard tissues¹¹. One point that differentiates them from other bioactive ceramics or glass-ceramic is the possibility to tailor a great chemical range of properties and of linking speed to the tissues¹². Therefore is it possible to design glasses with tailored property for a specific clinical application. The bioactive glasses can be produced with the conventional technologies of the glass industry, but it is necessary to verify the purity of the row materials, to avoid the contamination by impurity and the loss of volatile elements, like Na₂O or P₂O₅. The different phases of production, so like the choice of the raw materials, influence the final features of the piece. The bioactive glasses are soft glasses and therefore the final shape can be easily given with conventional tools.¹³. The base components are usually SiO₂- Na₂O-CaO-and $_{P2}O_5$. In table 1.1 are restored the composition (percentages in weight) of the most common bioactive glasses. The most studied of these is the Bioglass 45S5. The abbreviation indicates that it contains 45 % in weight of SiO₂ (oxide creator) and the molar rate between Ca and P is of 5:1. Glasses with significantly lower molar rate (in the form of CaO and P_2O_5) don't generate connections with the bone ^{14,15}.

2. Material and Methods 2.1 Materials Used

Fine-grained quartz was used as the source of silica, while Sodium Carbonate (Na₂CO₃), (Assay 99.9%); Calcium Carbonate (CaCO₃), (Assay 99%); Ammonium Dihydrogen Ortho Phosphate (NH4H2PO4), (Assay 99%); Boric Acid (H₃BO₃), (Assay 99.7%); Potassium Carbonate (K₂CO₃), (Assay 99%); Lithium Carbonate (Li₂CO₃), (Assay 99%); Magnesium Carbonate(MgCO₃), (Assay 99%); was the source of Sodium Oxide (Na₂O); Calcium Oxide (CaO); Phosphorus Pentaoxide (P₂O₅); Boron Trioxide (B₂O₃); Potassium Oxide (K₂O); Lithium Oxide (K₂O); Magnesium Oxide (MgO) respectively^{17,18}.

2.1 Method of Synthesis

For synthesis of bioactive glasses proper amounts of materials used are weighed using an Electronic Balance and mixed homogeneously in an Agate Mortar with an Agate Pestle. Premixed batch is melted in Platinum Crucibles, at 1400°C, for 3 hours using Globar Furnace. Sample is cast in Aluminum Molds, and annealed at 500°C for 4 hours using an Annealing Furnace.

3. Experimental Methods

3.1 Batch making

Composition Selection Formula- (45S5-X)+XMgO X=0.5,2,3,4,6,8 45S5 Composition $(45SiO_2, 24.5Na_2O, 24.5CaO, 6P_2O_5wt \%)$

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3.2 Preparation of Glass powder

Four different types of doped glasses were prepared, and as reference, a glass with the composition corresponding to Bioglass_ 45S5 (hereafter as BG) was also prepared, as reported in Table 1. About 100 g of batch were prepared by mixing reagent grade Na_2CO_3 , $CaCO_3$, $Na_3 PO_4 12H_2O$, SiO₂ and MgO, raw materials in a sealed polyethylene bottle for 1 h. Premixed batches were put into a 50 ml alumina crucible and melted in an electric oven for 1 h at 1400 °C; the samples with MgO (0.5% and 3.2%) were melted at 1400°C for 1 h and re-melted for 3 h (to ensure the homogeneity of the material). The sample with 2% of MgO was respectively melted at 1400 °C for 1 h (re-melted for 3 h) and 4h. The melts were poured, crushed and sieved through 25 mesh screen to produce fine particles^{21,22,23}.

The procedure for the fabrication of bioactive glasses can be briefed as below:

- 1. Raw material was taken in the required proportion and was mixed for at least three hours.
- 2. It was kept in an alumina crucible as well as it was placed in the furnace and was heated under controlled program.
- The temperature of the furnace was increased at the rate of 5°C/minute. The maximum temperature was varied in between 1350°C to 1400°C.
- 4. The time for melting was maintained in between 2 to 4 hours.
- 5. It was followed by pouring, annealing and controlled cooling to remove the stress and strain of the glass

TABLEN01 Experimental compositions of the examined glasses (wt %)

	BG-1	BG-2	BG-3
MgO SiO ₂ Na ₂ O CaO		1.00	2.00
SiO ₂	45.0	45.0	45.0
Na ₂ Ô	24.5	24.5	24.5
CaŌ	24.5	23.5	22.5
P_2O_5	6.0	6.0	6.0

BG———Bio-glass sample

Composition of Sample

BG-1 - 45 SiO₂-24.5CaO-24.5Na₂O-6P₂O-1MgO BG-2 - 45 SiO₂-22.5CaO-24.5Na₂O- 6P₂O₅-2MgO

4. Results and Discussion 4.1 Differential Thermal Analysis Result

Any physical or chemical change occurring to the test sample, which involves the evolution of heat, will cause its temperature to rise temporarily above that of reference sample leading to an exothermic peak. Conversely, a process, which is accompanied by the absorption of heat, will cause the temperature

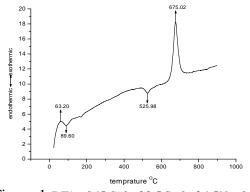


Figure 1 DTA of 45 SiO₂-23.5CaO- 24.5Na₂O-6P₂O₅-1MgO Bio-glass sample no.1

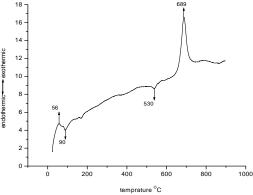


Figure 2 DTA of 45 SiO₂-22.5CaO- 24.5Na₂O-6P₂O₅-2MgO Bio-glass sample no.2

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of the test sample to lag behind that of the reference sample, leading to an endothermic peak. The area under any given peak can be used as a quantitative measure of the amount of heat evolved or absorbed by the physical or chemical changes, which has occurred.

4.2 Outcomes Based on The Test Of DTA

The curves presented in the above figures shows exothermic peaks for different compositions of SiO₂-CaO-Na₂O-P₂O₅-MgO glass with varying MgO /CaO ratios^{1,18}. The glass transitions temperature have been marked in the figures for respective glasses and they have been present against the composition of the glass. The variation in the Tg point with composition of the glass shows the structural changes taking place in the glass²⁰. It also reveals the extent of interaction of oxygen legands with ions of iron in the glass. The variation in the exothermal peaks dictates the change in the nature of the glass with change in concentration of CaO at the cost of MgO. Further it is evident from the exothermic peaks that the crystallization in the glass takes place with the evolution of heat from the glass. The system changes during crystallization from a thermodynamically non-equilibrium state to an equilibrium state . The bioglass ceramics becomes a composite matrix which is formed during controlled crystallization where P₂O₅ acts as a nucleating agent in the glass transition temperature higher would be the crystallization temperature and higher the glass transition temperature higher would be the crystallization temperature in view their liquidus temperature. So, it is mentioned here with that the glass transition temperature varies with the addition of MgO for CaO and P₂O₅ in the glass.

4.3 Density Measurement

Sr. No.	Glass Composition (mole%)	MgO/CaO	MgO/ P ₂ O ₅	Density(gm/cc)
1	45SiO,24.5Na,024.5CaO.6P,O5	0	0	2.74
2	45SiO,23.5Na,023.7CaO.6P,O, 1MgO	0.02	0.08	2.76
3	45SiO ₂ 22.5Na ₂ O24.0CaO.6P ₂ O ₅ 2MgO	0.08	0.34	2.81

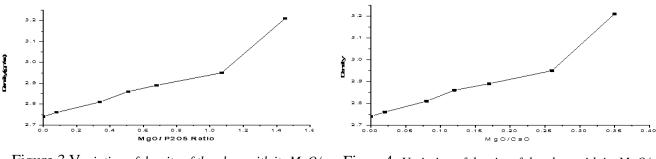
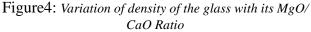


Figure.3 Variation of density of the glass with its MgO/P_2O_5 Ratio



4.4 Outcomes Based on The Result Of Density Mesurment

It is evident from fig.3 and fig.4 and table 2 that the density of the glass has increased with an increase in MgO/CaO ratio and ratio MgO/P2O5 in the 45S5 bioactive glasses. It indicates that the replacement

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of a smaller Ca2+ ion by a heavier Mg ion increases the density of the glass and the system is closely packed 4.5 Compressive Strength

I I I I I I I I I I I I I I I I I I I					
Sr.No.	Glass Composition (mole %)	MgO/CaO	MgO/ P ₂ O ₅ Com	pressiveStrength (M.Pa)	
1	45SiO ₂ 24.5Na ₂ O24.5CaO.6P ₂ O ₅	0	0	155	
2	45SiO,24.5Na,023.5CaO.6P,O,.1M	gO 0.02	0.08	160	
3	45SiO ₂ 24.5Na ₂ O22.5CaO.6P ₂ O ₅ 2M	gO 0.08	0.34	176	

TABLE No 3 Compressive Strength of different Bio active glass samples

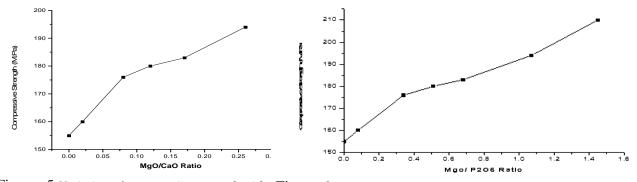


Figure: 5 Variation of compressive strength with Figure 6 Variation of compressive strength with MgO/P₂O₅ Ratio MgO/CaO Ratio

4.6 Outcomes Based on The Result Of Compressive Strength

It is evident from fig.5 and fig.6 and table 3 that the compressive strength of the bio glass has increased with an increase in MgO/CaO ratio and ratio MgO/P₂O₅ in the 45S5 bio active glasses

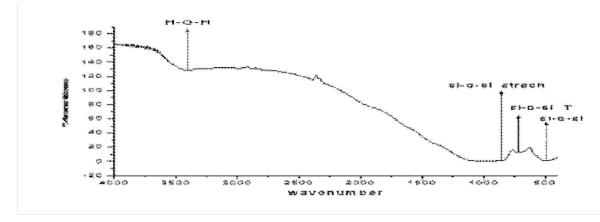


Figure7 FTIR Spectra of 45 SiO₂-23.5CaO- 24.5Na₂O-6P₂O₅-1MgO Bio-glass before SBF for sample no 1

4.7 Fourier Transform Infrared Spectroscopy (FTIR) Experiment for Different Sample With the help of Fourier-Transform infrared Spectroscopy (FTIR) analysis, information about the structure of synthesized bioactive glasses is recorded.

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To analyze the structure of synthesized bioactive glasses a quantity of all the bioactive Glasses is to grinded with potassium bromide finely (to remove scattering effects from large Crystals), used as a reference. This powder mixture is crushed in a mechanical die press to Form a translucent pellet through which the beam of the FTIR can pass. The infrared spectra of all the pellets are collected by using FTIR. T A B L E No.4 *FTIR Transmission Bonds ofBio-glass* 45 SiO₂-23.5CaO- 24.5Na₂O-6P₂O₂-1MgO for sample no. 1

Serial. no.	Wavenumber(cm-1)	Modes of Vibration	bonds
1	3432	strech	Н-О-Н
2	830	strech	SI-O-SI
3	690	bend	SI-O-SI
4	495	bend	SI-O-SI

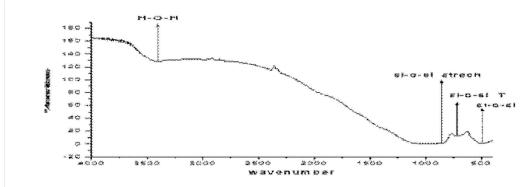
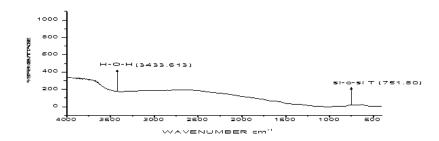


Figure 8 FTIR Spectra of 45 SiO₂-22.5CaO- 24.5Na₂O-6P₂O₅-2MgO Bio-glass before SBF for sample 2 T A B L E No.5 FTIR Transmission Bonds of 45 SiO₂-22.5CaO- 24.5Na₂O-6P₂O₅-2MgO Bio-glass before SBF for sample no.2

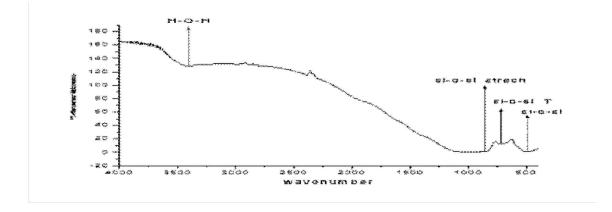
Serial no.	Wave number(cm-1)	Modes of Vibration	bonds
1	3428	strech	H-O-H
2	1052	strech	SI-O-SI
3	948	strech	SI-O-SI
4	723	bend	SI-O-SI
5	496	bend	SI-O-SI

FTIR reflection spectra of the surface of the bio-glass ceramic sample after reaction with SBF for various periods. The specimens BG-1, BG-2were soaked in an cellular simulated body fluid (SBF,100ml) with ion concentration and ph nearly equal to those of human blood plasma. The SBF was prepared according to Kokubo et al. by dissolving reagent grade NaCl, NaHCO3,KCl, K2HPO4.3H2O, MgCl2.6H2O, CaCl2 and Na2SO4 in an ion exchanged water contained in a polystyrene bottle¹⁶. These reagents were added in the order they are listed. The solution was buffered at pH value 7.25 with 50 mM of trishydroxymethyl-aminomethane (CH2OH) 3 CNH2 hereafter as TRIS) and 45 mM hydrogen chloride and its temperature was kept at 37 C. The soaking was carried out at 37 C, under continuous stirring and for various times (1, 5 and 10 days).

After SBF 1 day



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After SBF 10 day

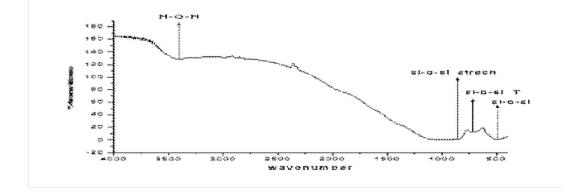
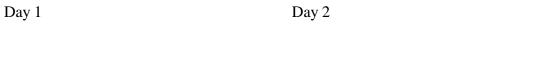
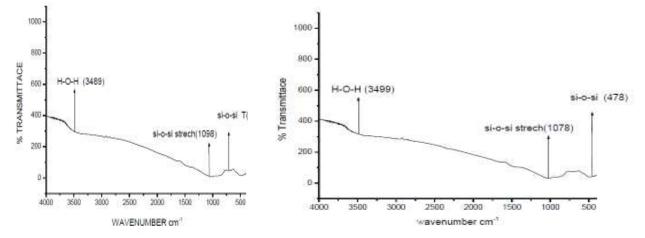


Figure 9 FTIR Spectra of 45 SiO₂-23.5CaO- 24.5Na₂O-6P₂O₅-1MgO Bio-glass after SBF for Sample no.1

"FTIR Transmission Bonds of 45 SiO₂-23.5CaO- 24.5Na₂O-6P₂O₅-1MgO Bio-glass after SBF treatment for sample no.1"

Serial no.	Wave number(cm-1)	Modes of Vibration	bonds
1	3434	strech	H-O-H
2	752	strech	Si-o-si
A B L E No. 7 SBF	After 5 day		
Serial no.	Wave number(cm-1)	Modes of Vibration	bonds
1	3379	bend	H-O-H
2	1025	strech	Si-o-si
3	497	strech	Si-o-si
A B L E No. 8 SBF A	After 10 day		
Serial no.	Wave number(cm-1)	Modes of Vibration	bonds
1	3485	strech	H-O-H
2	1433	strech	p-o
3	540	strech	Si-o-si







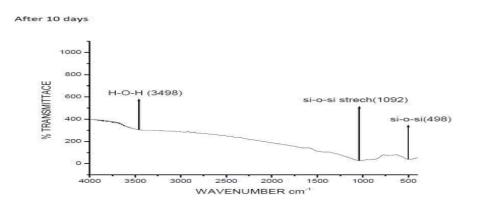


Figure 10 FTIR spectra of 45 SiO₂-22.5CaO- 24.5Na₂O-6P₂O₅-2MgO Bio-glass Sample no.2

"FTIR Transmission Bonds of 45 SiO₂-22.5CaO- 24.5Na₂O-6P₂O₅-2MgO Bio-glass sample no.2" T A B L E No. 9 *SBF After 1 day*

Serial no.	Wave number(cm-1)	Modes of Vibration	bonds
1	3489	strech	Н-О-Н
2	1098	strech	Si-o-si
3	729	strech	Si-o-si
ГАВLЕ No. 10	SBF After 5 day		
Serial no.	Wave number(cm-1)	Modes of Vibration	bonds
1	3499	strech	H-O-H
2	1078	strech	Si-o-si
3	478	strech	Si-o-si

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Serial no.	Wave number(cm-1)	Modes of Vibration	bonds
1	3498	strech	H-O-H
2	1092	strech	Si-o-si
3	498	strech	Si-o-si

TABLENO. 11 SBF After 10 day

4.8 Outcomes Based on The Test Of FTIR

The IR spectra above fig. after 1, 5 and 10days in SBF were markedly changed in the case of BG-1 and BG-.2 Mg the bands due to Si–O–Si group were still present, although very weak, indicating the polymerization of the Si–OH groups formed in the initial stages o fraction as a results of the ion exchange with H^+ of the solution. In addition we observed new bands in the spectral region typical of p–O and p–O vibrations in agreement with the presence of crystalline apatite revealed by XRD techniques. The spectra of BG-1, and BG-2 Mg showed lower modifications and still showed the bands assigned to Si–O (2NBO) and Si–O (NBO) and bands assignable to the P–O vibrations of an apatite crystalline phase were not detected. On the basis of the position of these bands we may propose that in these cases the original glass structure was maintained.

The glasses containing 10 weight % CaO with varying MgO /CaO ratios show analogous FTIR absorption characteristics. The band centered near 500 cm⁻¹,and 3500 cm⁻¹ in these glasses are assigned to stretching mode of two non bridging oxygen atoms which are bonded to phosphorous atoms as O=P=O in PO_4^{3-} tetrahedral and si-o-si. The bonds near 1439 cm⁻¹ and 1430 cm⁻¹ have been assigned to P-O⁻groups. The P-O⁻⁻ absorption bands near 586cm⁻¹ shift towards lower frequencies at around 509 cm⁻¹ as Mgo replaces P_2O_5 in the glass .The band near 1448 cm⁻¹ as attributed to PO_3 groups tends to decrease with increasing substitution of Mgo. Absorption bands near 1448 cm⁻¹ and 509 cm⁻¹ are assigned to the asymmetric and symmetric stretching modes of O=P=O linkage respectively .

7.5 X-Ray analysis of glass samples

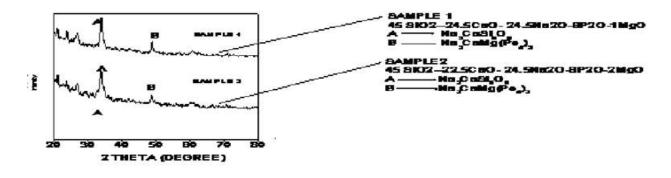


Fig. 13 XRD peak for 45S5 magnesium Doped bio-glass sample after sintering at 800c°

4.9 Outcomes Based on The Test Of XRD

XRD patterns of bioactive glass ceramic show the presence of two main crystalline phase of sodium calcium silicate (Na2 Ca Si3 O8, Na2 Ca Si3 O9).Introduction of MgO leads to the formation of new crystalline phase of sodium calcium magnesium phosphate (Na2 Ca Mg(Po4))2. It dictates that all the SiO2-Na2O-CaO-P2O5 and MgO Doped 45S5 glass samples have been melted properly and converted into crystalline phase.

Conclusions

- The formation of magnesium-containing phosphosilicate glasses is obtained until addition of 2% of MgO. The addition of small quantities of MgO up to 1% to Bio-glass 45S5 does not alter significantly its ability of in vitro apatite formation within few days of immersion in SBF. High magnesium content improves the chemical durability of glasses so the reactivity is negatively affected and magnesium -containing phosphates seem to be preferred with respect to calcium containing ones. The improvement of glass durability is ascribed to the degree of covalent character of Mg–O bond and the hydrolytic dissociation is difficult. The apatite formation is prevented both by glass durability and by magnesium ability to interact with phosphate giving rise to an amorphous phase. The magnesium concentration is always extremely low and the ion is immobilized in a solid phase.
- Increasing the MgOfrom 0-2 mole % causes an increase in density as well as compressive strength of the bio-glass
- Sharp peak was found. So the prepared bio-glass samples were crystalline in nature.
- Absorption bands of O-H and P-O reflecting the vibrations of OH and PO₄ groups in the bio-glass sample have been found at different wave numbers.
- Introduction of MgO leads to the formation of new crystalline phase of sodium calcium magnesium phosphate (Na₂CaMg(PO₄)₂).

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