श्री चित्रा तिरुनाल आयुर्विज्ञान और प्रौद्योगिकी संस्थान, त्रिवेंद्रम, तिरुवनन्तपुरम - 695 011, केरल, भारत
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PROJECT COMPLETION REPORT

1. **Project Number** : **6033**

2. **Title of the Project** : Development of bioactive bone cement based on organically modified ceramic resin

3. **Funding Agency Name** : TDF,SCTIMST

4. **Project Reference Number provided by the Funding Agency**: NIL

5. **Principal Investigator (Name & Address) : Dr.Lizymol P.P. ,Scientist, DEP,DBST,BMTW,SCTIMST**

6. **Co-Investigators (Name & Address): V.KalliyanaKrishnan**

7. **Implementing Institution** : SCTIMST

8. **Collaborating Institutions** : NIL

9. **Date of Commencement** : Date of approval : 9-10-2009 Date of commencement: 04-01-2010

10. **Duration** : 3 years

11. **Date of Completion** : 31-12- 2012

12. **Objectives as approved :** The aim of the project is to develop bioactive bone cement based on organically modified ceramic resin containing calcium as the liquid component and bioactive fillers such as hydroxy apatite along with PMMA or PMMA-PS copolymer as the solid component to overcome the drawbacks of conventional PMMA bone cement.

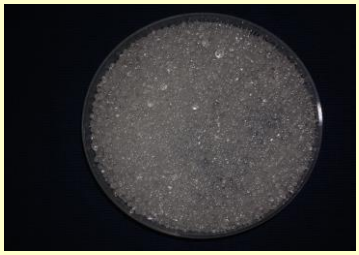
13. **Deviation made from original objectives if any, while implementing the project and reasons there of : NIL**

14. **Field/Experimental work giving full details of summary of methods adopted, data collected supported by necessary tables, charts, diagrams and photographs :**

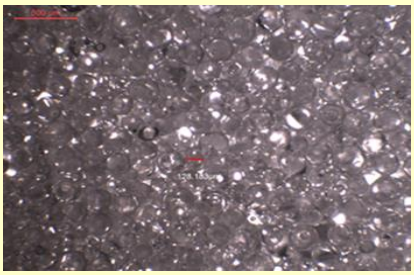
Details published and list is attached

Synthesis of PMMA-PS copolymer

- **Reagents used**
  Disodium hydrogen phosphate (Merck), sodium dihydrogen phosphate, polyvinyl alcohol (Aldrich), Methyl methacrylate (Aldrich), Styrene (Aldrich)



PMMA-PS copolymer



PMMA-PS copolymer-microscopic image

Preparation of bone cement

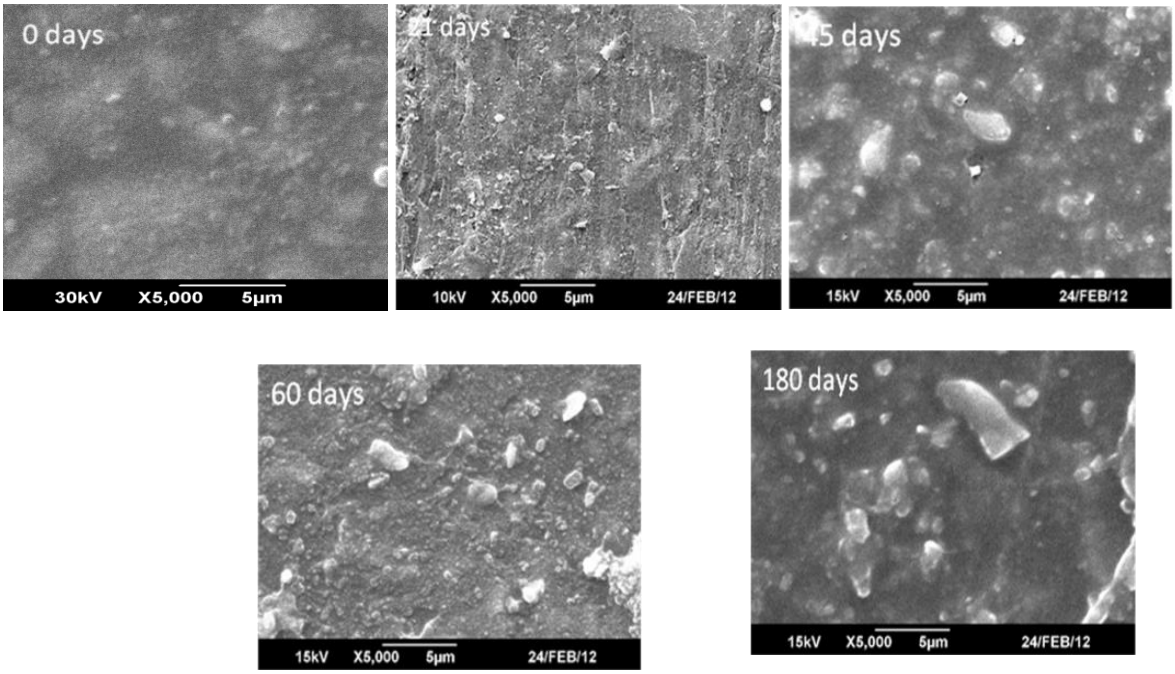
Bone cement consists of liquid component containing Ormoresin with dissolved 4-(dimethyl amino) phenyl ethyl alcohol (DMAPEA) (Aldrich) (accelerator) and solid component containing filler part (mixture of PMMA +HAP/mixture of PMMA-PS copolymer + HAP) and Benzoyl peroxide (BPO) as the initiator. Several formulations of bone cement were prepared with varying concentrations of filler; accelerator and initiator .Working time and setting time of these formulations were determined

15. Detailed anaylsis of results :

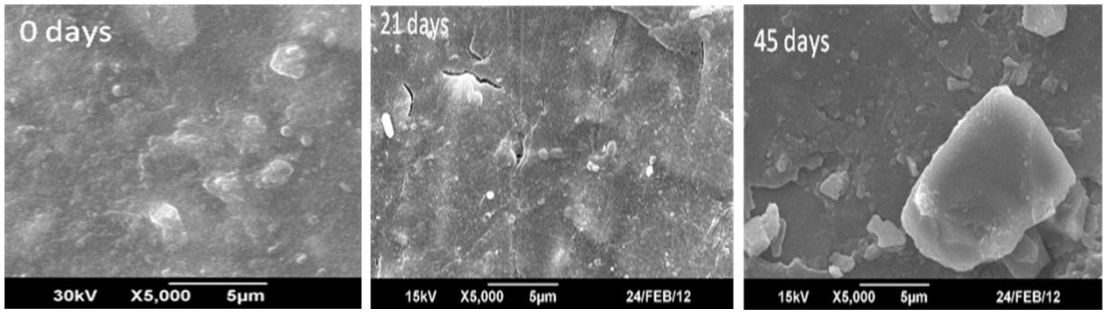
Mechanical properties of bone cement by using Ca-0.5 resin as the liquid part.

Powder part	Liquid part	CS (MPa)	SD (MPa)	DTS (MPa)	SD (MPa)
70%PMMA-PS + 30% HAP + 2.5%BPO	0.5%DMAPEA+50 parts TEGDMA+50 parts Ca-0.5 ALK resin	44.94	13.54414	18.56	1.80
70%PMMA-PS + 30% HAP + 2.5%BPO	MMA + 0.25% DMPT	90.73	30.08698		
70%PMMA-PS + 30% HAP + 2.5%BPO	0.5%DMAPEA+50 parts	86.79	20.65521		

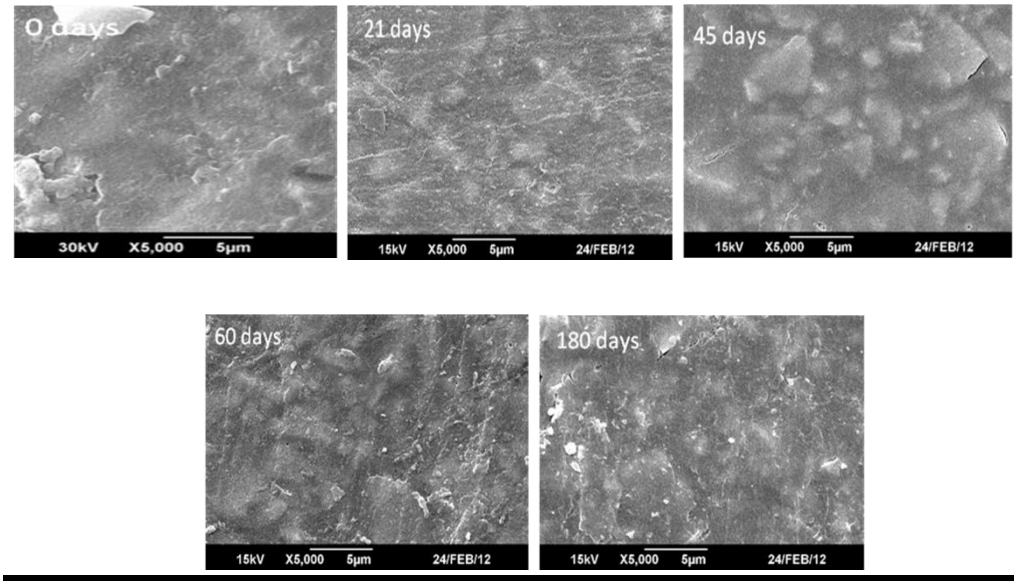
	TEGDMA+50 parts Ca-0.5 ALK resin				
70%PMMA-PS + 30% HAP + 2.5%BPO	0.5%DMAPEA+50 parts TEGDMA+50 parts Ca-0.5 ALK resin	79.39	15.97184		



SEM images showing the surface remineralization on composite prepared from Ca-0.5 resin after storing in SBF for 0, 21, 45, 60 and 180 days



SEM images showing the surface remineralization on composite prepared from Zn-0.5 resin after storing in SBF for 0, 21, 45, 60 and 180 days.



SEM images showing the surface remineralization on composite prepared from Mg-0.5 resin after storing in SBF for 0, 21, 45, 60 and 180 days .

Conclusion

The inorganic content in the resin has a direct influence on the properties of composites made from these resins.

All of the synthesized organically modified ceramic resins were transparent and viscous in nature,and the refractive indices of the resins found to increase with increasing inorganic content.

Increase in hardness of composites prepared from synthesized resin implies the increase in monomer conversion for the composites. Effective bonding between the organic and inorganic parts within the resin and better surface ion concentration on the surface of these composites enhance better properties.

Effect of Barium on the Physcio- chemical properties and shrinkage of calcium containing resins was studied and it was found that Ca0.5 Ba0.5 gave the optimum properties. Further increase of Barium content made a deteriorating effect on the physcio mechanical properties of the resin.

Effect of calcium and zirconium on the physcio mechanical properties and shrinkage of barium containing resins was studied. It was found that Ca0.1 Ba0.1 Zr0.1 gave synergistic effect and gave better properties compared to the other combinations.

16. Summary sheet of not more than 2 pages under following heads : (Title, Introduction, Rationale, Objectives, Methodology, Results, Translational Potential)

- It is discussed above in 14 and 15

17. Contributions made towards increasing the state of knowledge in the subject :

As per our knowledge this is the first attempt to develop a MMA/BisGMA free bone cement. This study provided new process for future developments in bone cement technology developments

18. Conclusions summarising the achievements and indication of scope for future work :

This study provided insight for a new process for future developments in bone cement technology developments. The various formulation of composite materials based on the calcium/Barium/zirconium containing resin and quartz, glass, combination of HAP/PMMA-co –PS filler is found to have excellent compressive strength. Proof of concept has been demonstrated. Further studies to optimise working time, setting time and compressive strength have to be carried out. Safety and efficacy of the material has to be demonstrated

19. **Science and Technology benefits accrued :** Novel calcium/barium/zirconium containing inorganic-organic resins were synthesised which were used as precursors for bone cement. Various parameters were optimised. Synthesis conditions for copolymers of poly methyl methacrylate (PMMA-co-PS) and hydroxyl apatite (HAP) were standardised. Self cure bone cement formulations were prepared

a List of research publications with complete details :

Book Chapters/Journal papers

1. LizymolP.P*.Novel inorganic organic hybrid resins as biomaterials "Recent Advances in Material Science; Series Journal : Composites and Nanocomposites Editor: Dr. A. K. Haghi, Publisher : Apple Academic Press, (Publisher CRC Press) Canada. (Vol. 4). (2013) Chapter: 8 2013/3/1 Pages 89

2. Lizymol P.P*. Physical and Mechanical Properties of Visible Light Cure Composites Based on Novel Organically Modified Ceramic Resins Advanced Materials Research Vol. 685 (2013) pp 50-53

3. Vibha C., **Lizymol P P.*** Development of bioactive multifunctional inorganic-organic hybrid resin with polymerizable methacrylate groups for biomedical applications‖ in Nanoparticles in Polymer Systems for Biomedical Applications, Apple Publications, ICNT 2016 Hard ISBN: 9781771887038,9, November 2018 .

4. **Lizymol P.P*.,** Paul W, Bioactive Composites for Hard Tissue Regeneration. Trends in Biomaterials & Artificial Organs,**2020**, 34 (3) ,84-85

Conference presentations

i. Sreekala S.Sharma*, Lizymol P.P*., V.KalliyanaKrishnan, Novel inorganic – organic hybrid resins for medical/dental applications, Proceedings of the first **International Conference** on Recent Trends in Materials Science and Technology (ICMST) , Indian Institute of Space Science and Technology(IIST), October 29-31, 2010.

ii..Lizymol P.P, Novel inorganic organic hybrid resins as biomaterials, First international Conference on composites and nanocomposites (ICNC), Jan. 7-9, **2011, (Invited talk)Mahatma Gandhi University, Kottayam**

iii. Vibha C.,Lizymol P.P., Effect of exposure time on surface hardness of visible light cure rerstorative material with a new photoinitiator was presented and selected as one of the three short listed papers for young scientist award contest (Ms.Vibha) in **Kerala Science Congress 29-31January 2011**)

iv. Lizymol P.P*, Bioactive composites for medical applications" in the Second **International** Conference onNanomaterials: Synthesis, Characterization and Applications (ICN-2012) during January 12-15, 2012 at **Mahatma Gandhi University, Kottayam, Kerala, India**

v.Lizymol P.P *. 'Effects of copolymer composition and inorganic content on mechanical properties/remineralization of composites for orthopedic applications'

2nd International Conference on Environmental, Biomedical and Biotechnology – ICEBB 2012, 4th and 5th August 2012, **Dubai, UAE**

v. Vibha C., Lizymol P P (2012), Effect of inorganic content on shrinkage of visible light cure composites prepared from inorganic-organic hybrid resins, PolyTech – 2012: International Conference on Advances in Polymer Materials & Nanotechnology, Dec. 15-17, 2012)

b Manpower trained on the project :

i.	**Research Scientists or Research Fellows**	**:**	**NIL**
ii.	**No. of PhD's produced**	**:**	
iii.	**Other Technical Personnel trained**	**:**	**3 (**Sreekala, Vibha and Syamiya)

c Patents taken, if any **:** **1**

Patent application for the patent "A process for the development of bioactive bone cement based on organically modified ceramics resins and hydroxyl apatite (HAP) filler for orthopedic/dental applications" is submitted.

d Products developed, if any **:** **1**

20 Abstract: (In 300 words for possible publication in ..... Bulletin) NA

21 Procurement/Usage of Equipment:

a. Details of Equipment: No new equipment purchased. Existing equipments were used

Sl. No.	Name of Equipment	Make/ Model	Cost (Rs.)	Date of Installation	Utilisation	Remarks regarding maintenance breakdown

b. Suggestions for disposal of equipment(s):Not Applicable

12/12/2023
Lizymol P.P.
(Name and Signature of PIs with date)

Routing: *Signed copy of "Project completion Report" by PI → root@sctimst.ac.in, rpc@sctimst.ac.in*