A COMPARATIVE ANALYSIS OF ADHESION AND BOND STRENGTH OF BIOACTIVE OBTURATING MATERIALS WITH ROOT DENTIN

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ABSTRACT

Obturation of root canal is an important step in root canal therapy. Gutta percha is the most commonly used material to fill the root canal for obturation. In this study composites of gutta percha with 10% hydroxyapatite (HA) and polyurethane (10% and 20%) were investigated to find out their adhesion and bond strength with root dentin after 7 days. Extracted human teeth were used for this study and in vitro root canal obturation was done. After filling the samples were immersed separately in deionised water solution. Push out test and scanning electron microscopy (SEM) was done to find out the adhesion and bond strength of these materials. Push out test and SEM evaluation of these obturating materials showed that gutta percha had maximum bond strength, whereas gutta percha with 10% hydroxyapatite had minimum bond strength as compared to other bioactive materials used in this study. Polyurethane composite with 20% HA was next to gutta percha in terms of its bond strength followed by polyurethane with 10% HA. Gutta percha obturating material proved to be the best regarding adhesion and bonding with the root dentin but polyurethane (with 10% and 20% HA) also looked promising.

Key Words: Polyurethane, Hydroxyapatite (HA), Gutta Percha (GP), Deionised water, Push out test, Scanning Electron Microscopy (SEM).

INTRODUCTION

Dental caries is a localized, progressive destruction of tooth structure and it is the most common cause of pulpitis. The pulpitis causes an intense, unbearable pain in the tooth. Previously patients and dentists used to extract tooth in order to relieve the pain. But now over the past few decades, a vast improvement in the field of material science making it easier to save the tooth rather than extracting it. Root canal treatment is one of the techniques practiced most commonly in dentistry to save the tooth and keep it in a functional position in the oral cavity. The final filling of RCT is called obturation and biocompatible material used to fill the root canal is called an obturating material.

The commercially available materials are although biocompatible but not bioactive. Their basic function is to fill the root canal and seal the apical foramen. The basic objectives of root canal obturating materials according to Noort, are to provide a clean canal, free of bacteria and other debris, provide an apical seal to prevent the fluids from tissues to enter into the canal, irritants leaving the canal and to prevent recontamination due to oral micro-organisms.

Gutta percha is the most widely used obturating material for root canal treatment due to its biocompatibility with oral tissues. Gutta percha was introduced in UK in 1843 and since then it has been used as root canal filling material in endodontics. Gutta percha alone does not adhere well with the root dentin. Different sealers are used for better adhesion and improved bonding. The effect of master cone taper on the bond strength and apical sealing ability of different root canal sealers has been investigated. An in vitro comparison of interradicular dentin bond strength of gutta percha and resilon was done in several studies and push out test results showed that resilon had higher value as compared to the gutta percha obturating material.
Polyurethane (PU) is a polymer. Several polymeric materials are in use these days in a wide range of applications in the field of medicine and dentistry. Their properties are so flexible and can be adjusted according to the requirements of the application in which they are used. Polyurethanes are mostly thermosetting polymers and they are used to coat implant surface as described by Park. Polyurethane liners are in use to enhance the bonding of silicone based facial prosthesis like nasal prosthesis. Polyurethane is under the spotlight to be used in several medical applications such as cardiovascular (CVS) applications.

Hydroxyapatite is one of the most studied calcium phosphate in the field of bio-ceramics. It is a mineral content of bone and teeth and has excellent biocompatibility and excellent mechanical properties. It is the most common biomaterial in all the fields of health care industry. Its osteo-conduction and osteo-integration properties enhance the process of bone regeneration. In orthopedics and dentistry, hydroxyapatite (HA) is used in several applications due to its biocompatibility with human tissues.

METHODOLOGY

Present study was carried out at the school of engineering and material science, Queen Mary University of London, UK. For this study, ten extracted human teeth were selected. They were of all types, incisor, canines, premolars and molars. The teeth free from any carious lesion and with straight roots were selected. The teeth were properly cleaned of any debris including plaque and calculus with the help of manual scaler. All other materials, Gutta percha points, Polyurethane, hydroxyapatite composite, extracted human teeth, hedstrom files # 15 to # 80 from Manni (Japan), Poly methyl methacrylate, chloroforms, Deionsed water, and Electron Microscope were available from the lab of Material Science.

In vitro root canal treatment of teeth

The roots selected for experiments were endodontically treated. First hedstrom files (H-files from Manni, Japan) # 15 was inserted inside the canal and dental pulp was removed. The technique was to insert the file into the root canal and a quarter turn was given. The pulp entraps in the threads of H-file and is removed easily by simply pulling out the H-file. Care must be taken not to give more than a quarter turn to avoid the breakage of file inside the canal. The procedure was repeated till the H-file passed easily through the apical foramen on the root. Then next H-file # 20 was inserted into the root canal, quarter turn was given and file was pulled out. In this way, H-file not only
A comparative analysis of adhesion and bond strength

removed the pulpal tissue from the canal but also shaped up the canal with its threads. Same procedure was repeated with file # 25 to # 80 and canals were prepared. All the prepared roots were immersed in the ethanol solution before filling. After canal preparation, teeth were divided into different groups for filling with different obturating materials.

**Filling of roots with Gutta Percha**

Gutta percha alone has a very poor bonding ability with root dentin. Thus a sealing agent, Sealapex by Kerr, was applied to enhance the adhesion of gutta percha with root dentin. Homogenous mixture of Sealapex paste was prepared on glass slab and applied to the inner wall of the dentin. The bonding agent was set within 4-5 minutes. With the help of Obtura™ II (Kerr dental, USA), which is a gutta percha heating obturating system, gutta percha was obturated in the root canal. The temperature of Obtura was raised to 200°C and gutta percha was added from the top of gun (hand piece). It melts at this temperature and easily fills the root canal. Temperature changes to room temperature in just a few seconds after ejected from hand piece. Two teeth were obturated with guts percha and immersed in deiodinised water. The samples were left in the incubator at 37°C for 7 days.

**Mixing of hydroxyapatite with gutta percha points**

Gutta percha points (1.5 gms) were taken and put in a glass beaker. The beaker was put in oven at 200°C for 15 minutes. Beaker was removed from oven. Gutta percha points became soft. A small amount of chloroform was added into the beaker and stirring was done to mix the softened Gutta percha points in chloroform. After mixing, pre ball milled hydroxyapatite of about 0.15gm (10% of weight of GP points) was added to the solution and mixed with stirrer.

Mixture was left in the fume cupboard to allow chloroform to evaporate and gutta percha with 10% concentration of hydroxyapratite was left. When material acquired a semi viscous state, It was filled into the root canal. Two teeth were obturated with GP + 10% HA and immersed in deiodinised water. The filled teeth were left in the incubator at 37°C for 7 days.

**Obturation of root canal with polyurethane and 10% hydroxyapatite (PU + 10% HA) Composite material**

Approximately 1.47gm of polyurethane with 10% HA was weighed and put in a clean glass beaker. The material was kept in a heat oven at 180°C for 15 minutes and it melted into semi viscous consistency. Beaker was removed from oven and with the help of a spreader, PU + 10% HA in semi viscous state was filled into the prepared teeth. Two teeth were filled with PU + 10% HA without sealer and immersed in deiodinised water. The samples were left in the incubator at 37°C for 7 days.

**Obturation of root canal with Polyurethane and 20% Hydroxyapatite (PU + 20% HA) Composite material**

Approximately 1.5 gm of bioactive material (PU + 20% HA) was kept in a heat oven at 200°C for 12 minutes. After 15 minutes the material melts into semi viscous consistency. Then with the help of a spreader, PU + 20% HA in this semi viscous state was filled into the prepared teeth. Two teeth were filled with PU + 10% HA without sealer and immersed in deiodinised water. The samples were left in the incubator at 37°C for 7 days.
Mounting of obturated specimens in the mould and sample preparation for push out tests

Before experiments, the specimens were mounted on the moulds. This was done by using a conventional ice cube trays. The material used to mount the roots into the mould was self curing poly methyl meth Acrylate (PMMA).

Dried roots were inserted in self cured PMMA separately in each block. The container with roots is put inside the fume cupboard, till all the residual monomer evaporates and stars to set. When PMMA cools down to room temperature, the moulds were taken out and put in respective containers. Extra PMMA was removed with cutter. Thin slices of each sample were cut with help of cooling diamond blade machine (Cuto 1 Jean Wirtz, UK). Three slices of 4mm width were obtained from each root sample and put in their respective containers and kept in incubator at 37°C till the day of push out test.

Push out test

It was performed on INSTRON 5564 (Materials Testing Machines, INSTRON, USA). The load used for pushing out the material from the root was 100 N and speed of sharp knob was adjusted to 0.5 mm/min. the knob passed through the filling area and pushed the filling out of the sample. After it, sample was removed and saved for scanning electron microscopy (SEM). Same procedure was repeated for all the slices of all samples. All the samples were mounted strongly on the round aluminium stubs with the help of conductive carbon cement and left in fume cupboard so that the carbon cement dries properly and adapts with aluminium stubs. Then dried samples were coated with carbon in BULZERS CED 030 (BAL-TEC, Germany) equipment in lab. Now the scanning electron microscopic examination was done using FEI Inspect F microscope (USA) in the lab. to visualize the amount of obturating material still attached to the root sample after push out test.

Table 1 shows the mean push out strength values of the obturating materials in deionised water. Three slices of all obturating materials in deionised water were analysed for push out test and then the mean value for the bond strength in MPa was calculated. Table shows that gutta percha has the maximum bond strength value followed by polyurethane with 20% hydroxyapatite (HA). Where as GP+10% HA has least bond strength value.

DISCUSSION

The main objective behind this study was to do a comparative analysis among the bioactive obturating materials to find out an obturating material that has an improved adhesion and bond strength with the root dentin. This was a comprehensive study over the time period of 7 days.

Gutta percha (GP) is known, not to have good bond strength with the root dentin, because of this reason root canal sealers are used to improve sealing and bonding. Gutta percha with HA is supposed to bind well with the root dentin as HA is the main inorganic component of the tooth dentin. Polyurethane with 10% and 20% HA, seem promising and might prove to be a new addition in the family of obturating materials.

Push out test was carried out to find out the bond strength under compression and scanning electron microscopy (SEM) of the sample afterwards to find out the adhesion of the material with the root dentin wall. The solution used in this study was deionised water.
From the results of push out bond strength and scanning electron microscopic analysis of obturating materials, it is clear that gutta percha has the maximum bond strength among all the materials used in this study. Although the mean bond strength was less than other studies but it is still excellent keeping in view the duration of the study as seen in other studies.5,14

Unexpectedly GP with 10% HA has the least push out bond strength value. Polyurethane with 20% HA had the second highest mean push out test value (0.733 MPa) followed by polyurethane with 10% HA (0.648 MPa).

On analyzing the bond strength values of Gutta Percha with root dentin many reasons need to be considered. One may be the use of technique of obturating this material. Gutta Percha was obturated with Obtura II. In this system, Gutta Percha melts and gets evenly adapted on to the walls of the root dentin.11 Second factor that helped GP in attaining maximum strength was the use of dentin bonding agent. This enhances the sealing as well as bonding of GP for better prognosis of root canal therapy.6,2,13 The combination of dentin bonding agent and obturating by thermofil obturating method had helped gutta percha to adhere well to the root dentin.7,8

The obturation technique can be one of the reasons for lowest bond strength of GP + 10% HA. Chloroform was used for softening of GP and its mixing with 10% HA. After obturation, chloroform got evaporated leaving voids and spaces between material and dentin.1 During push out test, material showed least resistance. Another reason could be the low viscosity at time of filling this material as seen in studies.9,16

Polyurethane with 10% hydroxyapatite also showed good results regarding bond strength with root dentin. Material showed better adhesion than GP with same consistency of HA. The SEM of polyurethane with 10% HA shows good adherence with root dentin even after the push out test was performed.

The composite of polyurethane with 20% HA proved to be the best among all the bioactive materials used in this study. It was the only material that had the bond strength close to that of GP. The SEM also shows good adhesion of polyurethane with 20% HA. There was a lot of material still attached with the root dentin after push out test. This bioactive material looks promising and might show even better results when tested after long duration. According to Hsieh,10, it has excellent potential as a root canal obturating material.

Polyurethane showed excellent consistency when it was heated. The material was easy to handle and condense for filling into the root canal. It gave excellent working time at high temperature and its sticky consistency made it possible to adhere well with root dentin walls. It may be the reason which gave polyurethane better results than GP with 10% HA. SEM of polyurethane (10% and 20% HA) showed that the material is well adapted with the surrounding root dentin even after push out force. This study showed that the composites of polyurethane with 10% and 20% HA had ability to bind well with the root dentin.

CONCLUSION

The results of this comprehensive study on the adhesion and bond strength of bioactive materials with root dentin are positive, as polyurethane with hydroxyapatite proved to be a material that can show excellent adhesion with root dentin. Polyurethane with 20% HA proved to be promising and the only bioactive obturating material used in this study, the bond strength of which challenges that of gutta percha in both solutions. Polyurethane seems to be promising and can be a good addition among the obturating materials used in clinical dental practice due to its binding with root dentin. The incorporation of HA encourages the bond strength between an obturating material and root dentin.

There is still a lot of work needs to be done to improve the adhesion, mechanical properties of these obturating materials and to see their effects on underlying bone and tissues. These materials seem promising and have a very bright future in the field of endodontics if their mechanical properties are improved. It will help improve the prognosis of root canal therapy and betterment for the patient as seen in studies by.12,14,15

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CONTRIBUTION BY AUTHORS

1 Khawaja Rashid Hassan: Performed the practical work and lab work for the study. He did root canal of extracted teeth, obturation, cutting of sections and then performed the push out test and electron microscopy. He took pictures of the resulted tooth sections. This whole study and experiments were done under supervision of College Faculty Supervisors in the department of School of Engineering and Material Science, Queen Mary University of London.

2 Muhammad Rizwan: Helped Rashid in doing root canal preparations and obturation of the selected teeth and prepared for mounting and cutting section to perform push out test and electron microscopy which was done by dr Rashid in Lab. He arranged the whole study into article form, its pattern according to instructions by PODJ. He arranged the text into its proper mode (ie introduction, methodology, results, references), checked further corrections, setting of pictures and tables, drawing graph, and then getting final print outs to be sent for review and consideration for publishing.

3 Sadia Rashid: Selected and provided the extracted teeth. She searched various references related to this study and literature material from the library and internet sources. She selected the literature contents and gathered information about this study and previous studies done about same materials.