

The leakage study between restorative and pulp capping materials and diffusion analysis by Fick laws

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ABSTRACT

The investigation of adhesion behaviour of restorative and pulp Capping Materials in dental treatment is an important subject for the development of new materials and prevent unwanted conditions such as secondary caries prevalence and pain due to the leakage. The aim of the study is the development of new methods for leakage studies between the restorative and pulp capping materials. The micro-CT experiment was supported by the obtained mechanism by using Fick diffusion laws for leakage. It is found that the X-ray micro tomography analysis should be supported by the other techniques like electron microscopy and energy dispersive X-ray spectroscopy. Diffusion mechanism calculations have been identified by EDX analysis and it is found that there are two different diffusion mechanisms in the interface region between the pulp capping and the restorative materials. So the suffer duration after dental treatment is strongly dependent on the strong of inner part of the interface region.

Keywords: Leakage, Fick Law, Dental materials, Ag diffusion.

1. INTRODUCTION

The adhesives used as a dental materials are important for the success of clinical treatments. It is a big challenge to seal without the leakage between resin-dentin interfaces [1]. The leakage in dental science which is defined as the leaking of bacteria, fluids, molecules and ions from the cavity wall and the restorative material has been recognized as one of the most important reasons for the failure and re-occurrence of restorations. The micro-leakage occurs with the findings of secondary caries and pulp inflammation after a period following restorative materials implantation when postoperative sensitivity, fillings and tooth edge discoloration and leakage depth are profound [2-4]. The most important factors affecting the prognosis of pulp capping can be given as bacterial contamination through caries or saliva and/or micro-leakage between the filler material and the pulp. If the bacterial contamination is prevented, it was observed that healing and Dentine Bridge are able to be formed [4-6]. For this reason, to reduce bacterial micro-leakage, the selection of restorative material is important for the treatment of the vital teeth.

The leakage mechanism in dental science is important for understanding the pain after dental treatment and developing new bio-compatible materials [7]. There are different reasons of leakage after dental treatment: polymerization shrinkage has been observed during the curing of restorative materials made of polymer, differences in coefficient of thermal expansion between dental tissue and restorative material and water absorption of restorative material are pointed out the factors which affect the clinical success in terms of micro-leakage [8]. In addition, when bond strength of restorative materials increases, micro-leakage will decrease [8-10]. Although leakage studies are performed as in vivo and in vitro, most of the studies in the literature are performed as in vitro. But researchers state that it is very difficult

to compare the results because of the using of different dental materials, the cavity preparation methods, the shape of the restoration, the duration of sample preparation and testing, the maturing shape of the samples and heat treatment conditions.

Observation of temporary pain in patients as a result of sweet, sour, salty drinks and foods give the most apparent clue of micro-leakage-related sensitivity. Some of the liquid penetrates into the dentin and can cause painful osmotic irritations. These problematic conditions are permanent and can grow up to pulpitis [11-13].

The pulp capping is the treatment which has a purpose of ensuring the integrity of pulp after the dressing the affected part with the healing substance and the viability of pulp in deep carious teeth [14].

Calcium hydroxide [Ca(OH)₂] which was discovered by Nygren in 1838 is widely preferred by dental studies among the pulp capping materials [15]. This is because Ca⁺² ions are well tolerated by the tissue and have importance in terms of cell proliferation, blood coagulation and mineralization. Moreover, Ca(OH)₂ has an impact in the pulp; it has a caustic effect with the alkaline pH on one side and blocking the enzyme on the other side. Under the area of the coagulative necrosis formed with Ca(OH)₂, the reserve mesenchymal cells are first changed into fibroblasts, then into odontoblasts that forms the matrix [16]. The enzymatic activity that is lost with the influence of Ca(OH)₂ restarts in the cells after seven days. In the literature, the first repaired dentin was seen after 12 days and it was determined that dentine bridges were formed in about 3 to 4 weeks [17].

In this study, Voco composite (Arabesque composite resin) materials were used for preparing the samples. Arabesque composite resin is the micro hybrid composite mixed with ceramic

glass materials. These materials have high strength and stability against abrasion, excellent aesthetics and tooth alignment, good polishing and suitable for all type of cavities.

Santos *et al.*, investigated the leakage for three different types of composite restorative materials and one type of glass ionomer cement and it was found that the maximum leakage occurred in the glass ionomer cements [18]. Yassini *et al.*, studied the effect of mechanical loading and polishing upon the leakage in restorative materials and glass ionomer cements with the scanning electron microscope. The maximum leakage was found in glass ionomer cements [19]. The marginal leakage of human in vitro teeth is investigated by Zhao *et al.*, using X- ray micro tomography. They found that there was a leakage of Ag ions in the restoration and dentine walls [20]. Juloski *et al.*, studied the effects of 5 types of different materials (3 groups of bulk fill composite, 1 type of conventional composite and 1 type of flowable composite) microleakage in Class II cavities by measuring the silver nitrate

penetration depth. The microleakage in bulk fill composites was found to be more than in any other groups [21]. Simi *et al.*, investigated the effects of usage of posterior nano composite restorations together with adhesive liner, resin-modified glass ionomer cement and flowable composite upon the leakage; and then they identified that the base materials used under the composite restorative materials reduced the leakage at a considerable extent [22].

The aim of this study is to reveal the mechanism and the reasons of the leakage between restorative materials and Ca(OH)₂ of pulp capping materials. We fabricated the sample like dentin and solution of Ag ion with different pressure and temperatures for simulation of various states in mouth and after this process, the cross section of the samples was investigated by SEM, EDX, Micro CT analysis. The diffusion mechanisms of the intersection areas were determined.

2. EXPERIMENTAL SECTION

We have fabricated 8 different samples with same properties as seen in the Figure 1. Dycal Ca(OH)₂ pulp capping material was placed into the cylindrical molds of 5mm x 2 mm. After polymerization of samples with LED (Light Emitting Diode) which has a wavelength of 400 nm for 40 seconds, Clearfil SE Bond (Kuraray, Japan) bonding agent was applied as a layer on the capping material. The arabesque composite was placed into the cylindrical molds of 9 mm x 5 mm and the capping material prepared in advance was adapted into composite (Figure 1). It was closed with nail polish, leaving a gap of 1 mm around the capping material. The prepared samples as seen in the Figure 2 were placed into the bottles containing 0.1 N Ag(NO₃)₂ then a 8 kg/cm² load was applied.

Each of the samples is incubated in water bath for the temperature range of 30 °C- 55 °C, with an increase of 5 °C by using Julabo 5 water bath for 12 hours. After thermal processing, microleakage investigations between the pulp capping material and the restorative material were analyzed by Micro CT (Micro Computed Tomography Model 1172) device. Then in order to examine the micro leakage in cross section of the sample and the leak-induced diffusion mechanism, it was cut into two slices with IsoMet 1000 Precision Saw device. The surface morphology and elemental distribution analysis in the cross section area were analyzed by SEM, Leo Evo 40 VP and Bruker EDX unit combined by SEM system.

3. RESULTS SECTION

3.1. Microstructural analysis of the samples.

The fabricated samples were inserted the Ag solution for 12 h at different heat treatment conditions. The main idea of this process is that the determination and the understanding of the leakage between pulp capping materials and restorative materials.

Generally, the leakage between tooth and restorative materials have been investigated by dentists and scientist who are working in material science and related subjects. Especially, the subsequent effects of the leakage can be determined as percolation indirectly in dental tissue, secondary carries, post-operative



Figure 1. A. 5 mm x 1.5 mm standard Teflon mold for pulp capping material; B. Capping material obtained from the Teflon mold; C. 9 mm x 4 mm Teflon mold; D. Capping material and restorative material were placed into 9 mm x 4 mm Teflon molds. E and F standard samples of 5mm x 4 mm G. The restorative material which was placed onto capping material was closed with nail polish so that silver nitrate solution could penetrate from one place.

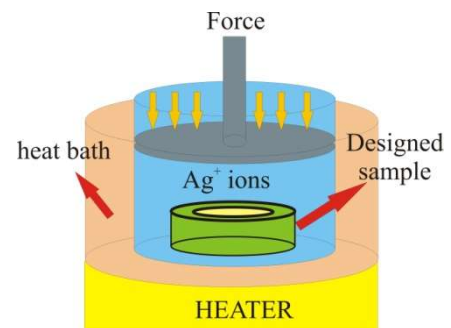


Figure 2. The experimental setup used for investigation of the infiltration mechanism.

sensitivity, and the loss of aesthetic after coloration. In this context, it is important to determine the leakage mechanisms between these two materials. The cross section of the samples which exposed to the Ag diffusion for different temperature and the durations were investigated by EDX dot mapping as given in Figure 3.

There are two different regions in SEM-EDX graph in the Figure 3. The outer section fabricated from restorative materials and the inner part is the pulp capping materials. The elemental distribution of both sections was also given in Figure 3. It is clear

that the elemental distributions of both sections are much different from each other. The elemental distribution graphs obtained from EDX measurement of whole samples for un-heat treated sample were given in Figure 4 and Table 1.

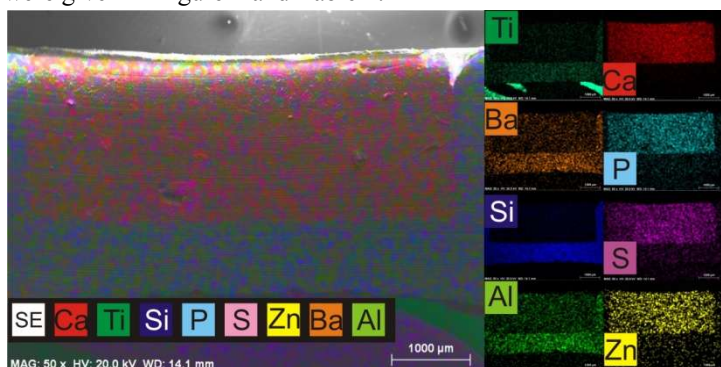


Figure 3. EDX mapping analysis of the cross section of the sample.

While the restorative materials have Al-Ba-Si-Ti rich compositions, Ca-P-S-Zn elements are high in the pulp capping material.

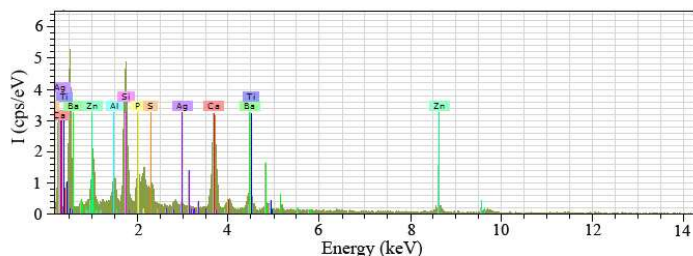


Figure 4. EDX graph of the un-processed sample.

Table 1. Elemental distribution of the sample obtained from EDX analysis.

Element	C. Weight wt. %	C. atomic At. %	Error %
Al	5.81	8.82	0.2
Si	18.18	26.52	0.5
P	13.35	17.66	0.3
S	5.63	7.19	0.2
Ca	21.98	22.46	0.4
Ti	2.03	1.74	0.2
Zn	16.57	10.38	0.4
Ba	16.46	5.24	0.3

The Ag ion in solution was diffused to the samples fabricated with different ratio depending on the heat treatment conditions. It was expected that the Ag ions are generally diffused exponentially inside to sample and the Ag diffusion should be diminished after several distances of micro meter from the surface of the materials. So, the leakage in the interface region between restorative material and pulp capping material should be much more than that of the other region of the samples due to unification problems as adhesions of the materials, cracks and holes during to formations. Thus we focused on this region for leakage and deformation after dental treatment and investigated the leakage region in detail and we tried to find purpose the solution paths as seen in the following section.

We found that during to microstructural investigation of the samples there are a lot of cracks and holes in the cross sections of the different samples. One of the examples for this kind of structure was given in Figure 5.

After vital pulp therapy in tooth, the sudden thermal changes observed within the capping materials can be caused by the expansion of cracks and holes. These regions energetically metastable and they easily make bonds whatever finds in environment for decreasing the internal energy. In this case, the regions become larger and so the leakage will increase and reach the nerve region of the tooth and the patients will suffer. It is well known that patients have postoperative sensitivities and pains in the results of compliance problems between tooth and restorative materials and the leakage extending up to pulp capping regions and holes and cracks in the pulp capping materials.

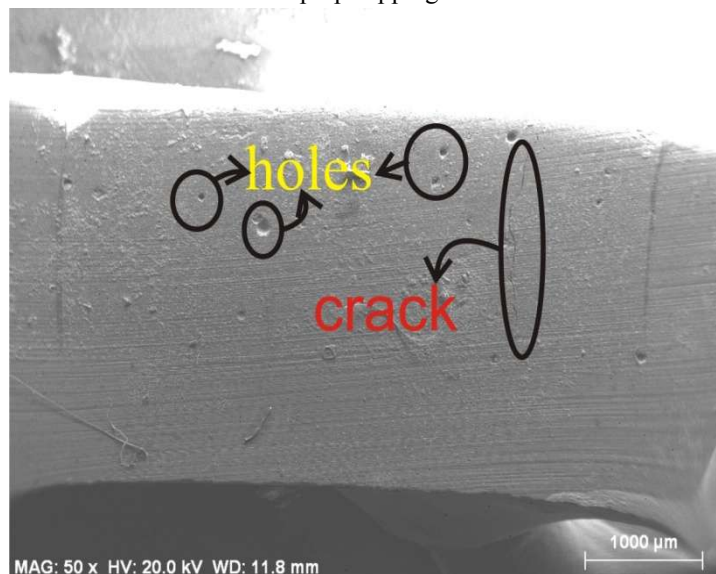


Figure 5. SEM image of the of the cross section of the sample.

The most important precaution to be taken for the prevention of these problems should be the development of new materials which have low thermal expansion and high density. Especially the cases such as suddenly turning on and off or non-homogen application of the LED light during the polymerization can cause the cracks and holes in the restorative materials. So, the leakage can reach to nerve and patient feels pain.

The other important factor for crack and hole formation is that the pulp capping materials that is in the form of gel cannot be fully polymerized due to preparation of non-uniform structure of gel and so, it can also cause cracks and holes in the materials. However, the non-uniform LED applications during to polymerization also cause the stress inside the sample and so the cracks easily occur. Thus, it is shown that there are two reasons for pain after cure of tooth: one is due to materials used and the other is due to clinicians.

3.2. Ag diffusion in the interface region of pulp capping and restorative materials.

We investigated the diffusion mechanism in the interface region of the fabricated samples by cutting the sample from the middle after Ag diffusion for different temperature and duration. Figure 6 shows the Ag dot mapping analysis for the interface region of sample processed for different thermal treatments. As seen in the figure, some region dot intensities are higher than that of the other region which means the higher amount of Ag in the cross-section.

It is easily seen that the Ag concentration is higher in the close region of the surface of the material. It also appears that some region during the interface section have higher dot intensity

which is an evidence of the leakage between pulp capping material and restorative materials.

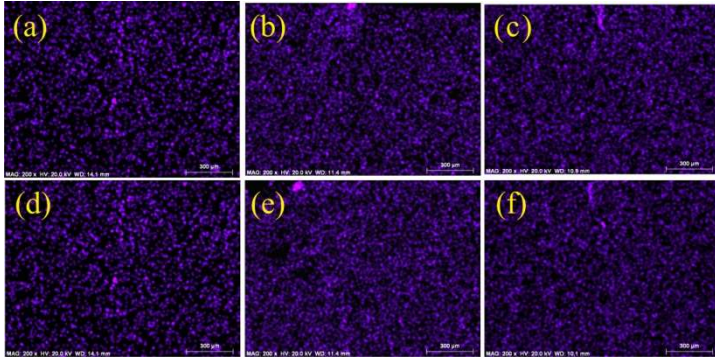


Figure 6. EDX dot mapping of Ag in the cross-section region of the sample after heat treatment at (a) 30 °C, (b) 35 °C, (c) 40 °C, (d) 45 °C, (e) 50 °C, (f) 55 °C.

Actually, we only investigated one cross-section of the sample (2D analysis), if we cut the sample into different slices, it will be seen that there are a lot of high intensity Ag region in the sample. So the micro structural investigations in this study give us a road map for leakage mechanism of the restorative materials and pulp capping materials which have been used by dental science.

For examining quantitatively the amount of diffused Ag ions, Ag content was measured by EDX dot analysis from surface to substructure of the interface region and we investigated the Ag leakage in the system. According to EDX dot analysis, it is clear that there was a definite leakage in the interface region of pulp capping and restorative materials. The cracks and holes which are formed during the dental treatment have energetically metastable regions and they easily cause an accumulation of Ag ions in these regions hence the Ag ions in these regions trigger the growing of holes and cracks. Furthermore, the thermal fluctuations due to consuming of hot and cold foods can cause the growing of the hole and crack regions. In this condition, the leaked materials inside the tooth reach the nerve region of tooth which gives pain to the patient. Therewithal, it can also cause the percolation, secondary decay, post-operative sensitivity and disappearing of aesthetics after staining.

3.3. Diffusion mechanism and calculations.

The determination of the leakage mechanism between the restorative and pulp capping materials has great importance for the development of ways to treat and determine the exact reason of pain after dental surgery and relief it. For this purpose, the understanding of the leakage mechanism for both materials will give great directions for subsequent studies.

The figure 7 shows the Ag concentration change with distance from surface to subsequent region of the samples depending on the thermal treatments. Basically, the expected change of Ag concentration with distance after diffusion should be in the form of exponential decrease curve from the surface and there should be an accumulation in the subsequent region of the interface regions. Although the obtained curve from EDX dot mapping analysis shows almost expected structure as mentioned above, some dots in the interface region have higher concentration than that of the other regions as seen in Figure 7. This case is an evidence of the discussion given in previous section. So the EDX dot mapping process for the determination of the leakage

mechanism in the tooth study is a reliable method for this kind of studies.

The obtained results demonstrate that Ag ions can easily diffuse to interface region and cause the deformation of the materials. The ionic radii of Ag^{2+} is 108 pm that is greater than the other elements and the atomic number is bigger than that of 46 such as C, B, H and N that is important for human life. So we concluded that the atoms or ions which are smaller radii can diffuse and easily reach the nerve inside the tooth.

According to Figure 7, the concentration of Ag ions were increased by increasing thermal treatment temperatures. The increase of the Ag content with temperature is expected. Because an increase in the temperature will increase the kinetic energy and the oscillation frequency of the atoms so the mobility of the diffused atoms will increase then it can cause the increase of diffusion rate and concentrations. We analyzed the diffusion mechanism by using EDX dot mapping data in detail.

The atomic concentration distribution among the fabricated layers are shown schematically in Figure 8 at $t=0$ and $t>0$.

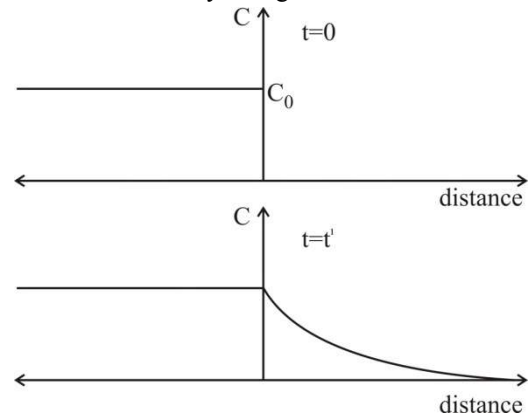


Figure 8. Schematic presentation of diffusion mechanism for $t=0$ and $t>0$

Ag ions in solution diffuse from regions of higher concentration to regions of lower one with heat treatment and time. Therefore, the percentage concentration of Ag ions in the material changed with increasing the heat treatment conditions such as temperature and time. If the concentration at some point changes with time, Fick's second diffusion law can be used [23];

$$\frac{\partial c}{\partial t} = \frac{\partial}{\partial x} \left(D \frac{\partial c}{\partial x} \right) \quad (1)$$

where, c is the concentration and D is the the diffusion coefficient. Schematic illustration of the designed material is shown in Figure 7.

We assume that $Ag=100\%$ for $x>+L$ and $x<-L$, $Ag=0$, for $-L \leq x \leq +L$ at $t=0$. When the diffusion is started, the concentration of Ag in the designed material region changes with the time and temperature. A solution of the diffusion equation in this case can be obtained in the following manner: Imagine that the diffused region for Ag ions consists of n slices. If α_i is the distance between the center of the i -th slice and $x=0$, the concentration at any given value of x at any t time can be given;

$$c(x,t) = \frac{c^l}{2\sqrt{\pi \cdot D \cdot t}} \sum_{i=1}^n \Delta \alpha_i \cdot \exp \left[-\frac{(x - \alpha_i)^2}{4 \cdot D \cdot t} \right] \quad (2)$$

where, $\Delta\alpha_i$ is the thickness of the each slice, c^l the starting concentration of the diffusing elements. When $n \rightarrow \infty$ and $\Delta\alpha_i \rightarrow 0$, the equation (2) becomes:

$$c(x,t) = \frac{c^l}{2\sqrt{\pi \cdot D \cdot t}} \int_0^\infty \exp\left[-\frac{(x-\alpha_i)^2}{4 \cdot D \cdot t}\right] d\alpha \quad (3)$$

The solution of equation (3) is the form of error function as given below;

$$c(x,t) = \frac{c^l}{2} \left(1 + \operatorname{erf}\left(\frac{x}{2\sqrt{D \cdot t}}\right) \right) \quad (4)$$

Spatial concentration ratios of Ag ions obtained from EDX analysis are shown in Figure 7.

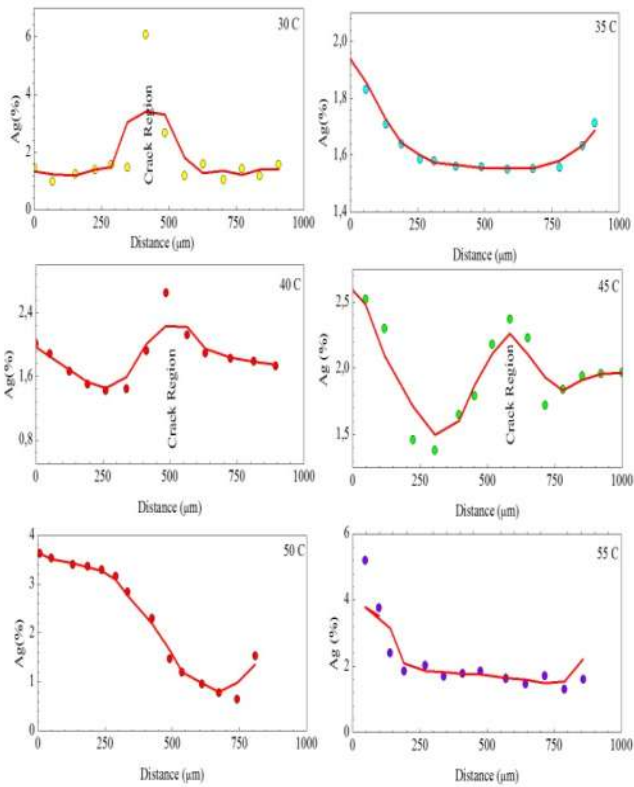


Figure 7. Ag diffusion in the processed Sample depending on different temperatures.

Diffusion coefficients for Ag ions were calculated by substituting of the obtained concentration values into Equation 3 and 4. We found that there are two diffusion regions in the samples one is the calculated diffusion coefficients are presented in Figure 9 depending of heat treatment conditions.

Empirically, D can be defined by the following equation [24, 25],

$$D = D_0 \exp\left(-\frac{Q}{RT}\right)$$

Where D_0 is pre-exponential term and Q is the activation energy for diffusion and both of them may vary with composition but they are independent from temperature. Experimentally the activation energy for diffusion can be obtained by plotting $\ln D - 1/T$ graph in the inset of the figure 9. The slope of this graph gives activation energy value as:

$$\frac{d \ln D}{d 1/T} = -\frac{Q}{R}$$

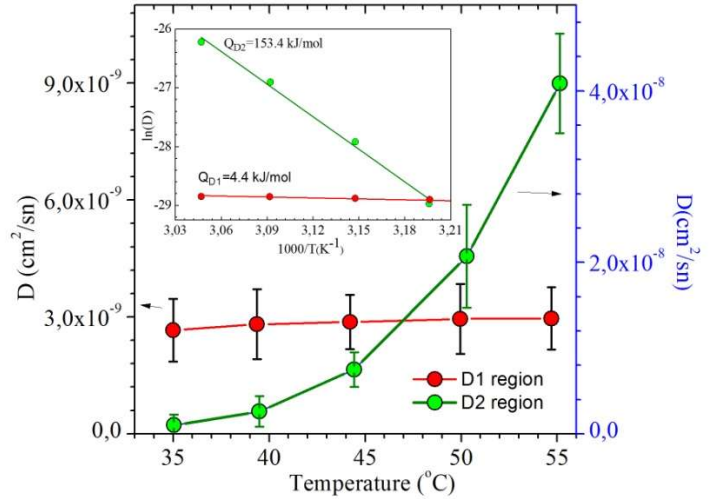


Figure 9. Temperature dependence of diffusion coefficient for different region of the samples.

The calculated activation energy for the region 1 and 2 are found as 4.4 and 153.4 kJ/mol. The results show that the diffusion in the first region that is close to surface is very easy which the effects such as mechanical excitations and small thermal fluctuations can trigger the diffusion. In the second region that is close to nerve part of the tooth, the diffusion is much more difficult than that of the first region. The leaked atoms in the close region of the surface accumulate the cracks and hole regions and causes the increasing of the deformation and so the patient feels pain after a certain time of the treatment. The time of feeling pain varies from person to person as known by dentists. We predicted that the differentiation in time depends on the accumulation time and the slow diffusion in second part of the interface. To decrease the leakage in dentine, a thermal barrier coating can be used in the inner region of restorative materials.

The material selection for barrier coating in dental work is one of the challenging issues for researchers. While some materials such as YSZ exhibit low value of thermal conductivity at specific temperatures, the thermodynamic stability in contact region may not have desired properties. There are three major difficulties for candidate thermal barrier coating in dental research. The first is selecting oxide materials that have the potential for low thermal conductivity. The second difficulty is assessing the thermodynamic stability of the barrier coatings. The third difficulty is determining of biocompatibility of the barrier coatings. The low thermal conducting barrier layers should have molecular weight as large as possible, complex crystal structure, non-directional molecular bonding and a large number of different atoms per molecule [29,30].

3.4. X-ray Micro-tomography results.

Micro - CT Skyscan model 1172 in high resolution was used under the situations of 100 kV acceleration voltage, 100 μ A beam current, 0.5 Al filter, 360° rotation at 0.8° step, 2000x 2000 pixel, 10 Image Pixel Size, frame averaging 2. The incisions were restructured with NReco and were analyzed with SkyscanCtan program.

The incision displays before and after the silver nitrate load are seen in Figure 10. In Figure 10a, radiopaque spots can be observed in some parts between pulp capping material and restorative material. In Figure 10.b, an increase in radiopacity was seen after the loading. Also, it is thought that silver nitrate was piled up into cracks and holes in the capping material as seen in EDX analysis. Yet, while looking at the incision images, it is not possible to distinguish whether radiopaque observed between the restorative material and capping material results from zinc in material or silver nitrate. For this reason, the use of only micro-CT is not a sufficient method in leakage studies. According to the results obtained in EDX analysis, when you consider radiopaque areas obtained from micro CT images as silver-based, especially the gaps in the structure are filled with different elements and causes crack applying pressure around. Then this bothers the patients in later stages after the treatment by causing sensitivity.

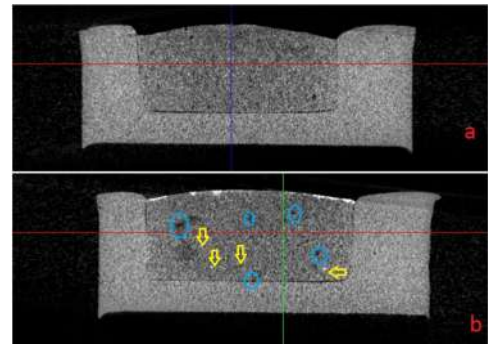


Figure 10. a. Micro - Ct image of control group before load cycling. b. After mechanical loading, silver nitrate solution penetrated into the gap. As seen in blue rings, an increase in porosity within capping material was observed just after the composite was polymerized with LED light and after the capping materials were hardened. As shown with yellow arrow marks, the silver nitrate was leaked into porous structures within materials. The silver nitrate between the restorative material and the capping material is shown with black lines.

4. CONCLUSIONS

In this study, we presented the microstructure and mechanism of the diffusion and leakage between restorative and pulp capping material. Non-homogeneous light application during polymerization or the sudden opening and closing of LED caused a deformation in the used materials such as cracks and holes. These deformation regions can behave as accumulation regions for leaked elements or ions. The diffusion rate in the interface region have two different types of behavior; first is fast and has low activation energy and second is slow and has high activation

energy. The second region behavior is important for reaching the nerve of the tooth and so the diffusion and the leakage can take certain time depending on the amount of deformations in the close region of the nerve. As a conclusion, it is found that the diffusion mechanism between two materials can be investigated by Fick law which can give more valuable information for future studies in this field. The adhesive properties of the two components can be improved by this information.

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