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The mechanical and heat conducting properties of dental composites

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ABSTRACT

In this study, it was studied the microstructural, mechanical and heat conducting properties of Ca added and un-added commercial light-cure restorative composite resins, in detail. The main goal of the study is the investigation of the mechanical strength and the thermal conductivity of the samples by Ca addition which the Ca ions have biocompatible properties. We used 0 %, 3 %, 6 % and 9 % Ca addition in the commercial light-cure restorative composite resin named Cavex Nano, Amaris, Tetrik N Flow, Solitare. The structural properties were analyzed by using XRD, FTIR, SEM, and optical microscope. The mechanical and heat conducting properties of the samples were investigated by micro hardness and thermal conductivity measurement system, respectively. It is found that there are two main structures in the materials; one is the organic content and the other is the crystalline content. We found that the Ca addition causes diffusion in the main matrix or accumulated in the inergranular region of the composite resins depending on the commercial materials. The thermal and mechanical properties showed a strong relation between the composite resin and Ca content. It is concluded that the diffusion of Ca into the main matrix causes an improvement of both the mechanical and thermal conducting properties which can be used in commercial composite resins in the near future.

KEYWORDS: *thermal conductivity, biomaterials, mechanical strength.*

1. INTRODUCTION

Dental composite resins were started to use in the middle of 1960' s as a commercial dental restoration materials [1-3]. Their mechanical, esthetic and biocompatible properties have made a major impact in their widespread usage in clinical studies and the manufacturers focus more on the developing and finding of new dental composite materials. Generally, the composite resins consist of initiating agent, resin matrix and inorganic fillers [4].

Some of the important properties expected from the dental composite resins are high mechanical strength, low thermal conductivity and structural stability [5, 6]. These properties can be provided by improving or modifying the inorganic fillers of the composite resin. The manufacturers use Ti, Al, Ba, Mo, Zr, etc. for increasing the physical properties of the materials [7, 8].

Increasing the mechanical strength and decreasing the heat conductivity are the two of the important properties of the inorganic fillers of the dental composites [9]. Furthermore, the inorganic fillers in the dental composite resins cause the protection of polymerization shrinkage during to light curing process.

To improve mechanical, thermal and structural stability of the inorganic fillers, scientist have been working on finding new materials, new fabrication techniques and/or modification of commercial dental composite resins. For this reason, SiO_2, ZrO_2, TiO_2, Al_2O_3 and B_2O_3 which can improve the mechanical strength, thermal conductivity or structural stability of the system are doped, added or substituted in the dental composite resins [10]. Generally, the doping of the elements or compounds in the composite resins are chosen to be between 5% -30% ratio. The excess content of them in the main matrix causes the formation of defects which the mechanical and thermal properties exhibit worse behavior when compared by the undoped materials [11].Calcium particles have been filled into resins to develop composites [12, 13]. In a study by Skrtic et al. it was reported that non-uniformity of the doping material in the composite surfaces provides defects for the large calcium content in the materials [14]. It is postulated that cracks that form upon soaking of the composites penetrate to reservoirs and serve as the avenues (channels) for the remineralizing Ca ions to diffuse out to the external milieu. In other study by Xu et al. it was reported that nano-composites including Ca ions in the materials release to combat caries, and they investigated the physical effects of Ca adding in the materials [15].

With a view to provide supplementary information to the study of composite resins, this research was guided to examine the mechanical, thermal and structural properties of the Ca added and unadded composite resins in detail. It is found that G-aenial Universal Flo showed better mechanical and lower thermal conductivity which can be used for commercial productions.

2. EXPERIMENTAL SECTION

We used four different commercial composite resins given in Table 1 and CaO (alfa-aesar cas no: 1305-78-8) was added and mixed to the system at ratios of 3%, 6% and 9% at 10 min. in a dark room due to the solidification of the materials from the gel form. After obtaining a homogeneous mixture, the pure composites and Ca added composites were poured into the Teflon mould which have 8 mm x 2 mm diameters. Transparent band was placed on the samples and they were polymerized with LED

(Light Emitting Diode) which has a wavelength of 400 nm for 40 seconds. Investigations were carried out for 48 samples. 12 samples were prepared for each composite group. Four composites were made with the following fillers: (1) 0% Ca (control group n=3), (2) 3% Ca (n=3), (3) 6% Ca (n=3) and (4) 9% Ca (n=3).

Table 1. The general properties of the commercial dental composite resins.

Material	Manufacturer	Type	Filler	Filler / Size/ Range	Filler Weight (%)	Microhardness at room temperature	Ingredients Matrix System	Flexural Modulus (GPa)	References
Tetric N-Flow (TF)	Ivoclar Vivadent, Schaan, Liechtenstein N03326	Hybrid	Ba glass, Ba–F–Al–B–Si glass, mixed oxides, SiO2, YbF3	0.7 µm	63.8	35.36	Bis-GMA, UDMA, TEGDMA Barium glass, Ytterbium fluoride, and Silica	5.3	[42]
Amaris Flow (AF)	Voco GmbH, Cuxhaven, Germany	Nanohybrid	Glass fillers	Not-indicated	64	Not-indicated	Bis-GMA; UDMA; TEDGMA; Glass Fillers Nanofiller silica	Not-indicated	[43]
Solitaire (ST)	Heraeus Kulzer GmbH, Germany	Not indicated	Silicon dioxide, Fluoride–barium–aluminum–borosilicate glass, Fluoride–aluminumsilicate glass	0.02–23 µm	66	41.7	(meth)acrylate monomers	Not-indicated	[44]
Cavex Nano (CN)	Quadrant (Hollond BV,Netherlands.)	Hybrid	Ba-Al-F-silikat glass silicon dioxide	(0.02-2 µm)	68	Not-indicated	Bis-GMA	Not-indicated	[45]

FTIR Spectra were performed by using pelkin Elmer FTIR analysis system using KBr. The structural properties of the materials were performed by XRD pattern using CuK$_\alpha$ radiation between 2-80^o and Rad-B D max diffractometer. The phases in the materials were analyzed by using Jade 4.0. The micro-structural characterization was performed with Scanning electron microscope (SEM, Leo EVO- 40 VPX) with attached Bruker X flash detector 4010 energy dispersive X ray spectroscope unit [EDX].

Optical microscope analysis of the samples was performed with LeicaDM4000M microscope with 500x magnification. The microhardness measurements were performed at room temperature by a Vickers indenter hardness tester [Shimadzu HMV-G21] fitted with a pyramid on square base diamond indenter. The loads of 10-25 g were chosen and it was applied for 15 s to the all samples. Indentations were made at five different places for each sample to find average hardness values. The micro hardness measured by Vickers method which is defined as the ratio of the load applied [in kg] to the projected area of the indentation [in mm^2] and can be expressed by the following relation [16]:

$$H_V = 1.854[\frac{P}{d^2}]$$

where H$_v$ is the Vickers micro hardness value, *P* is the applied load in kg and *d* is the diagonal length of the impression.

The heat conductivity of the samples was analyzed by the home made thermal conductivity measurement system. We used two brass blocks which the first base has a heater inside it and the second is the heat sink and both of them have k-type thermocouple inserted the surface of the blocks. We calibrated the system using Al powder (alfa Aesar cas no:7429-90-5 which have thermal conductivity value 205 W/Km) [17]. We used 120 W power with PID temperature to reach 55 ^{o}C temperature which is the highest temperature that can be applied without decomposition of the biomaterials.

3. RESULTS SECTION

3.1. FT-IR Analysis.

The FTIR spectrum in the range of 400-4000 cm^{-1} of the samples which undoped and 9% Ca addition were given in the Figure 1. It is known that the dental composite resin in this study have BisGMA, UDMA and TEGDMA organic binder which have C, H, N and O. FTIR spectrum can give an information about the molecular bond structure in the samples. The band in the spectrum at 3432-3476 is due to O-H vibration in the structure, the C-H stretching peak were observed in the range of 2923-2957 and the band at around 1070 is related to C-O-C bond in composites. The peaks at 1401, 1627 and 1724 were assigned for anti-symmetric $(CO_3)_2$-stretching, NH-CO group stretching and C=O stretching, respectively. When FTIR spectrum of CaO are investigated, CaO bonds gives a peak around 450cm^{-1} which it was not observed in this study due to the minor addition content of Ca in the samples.

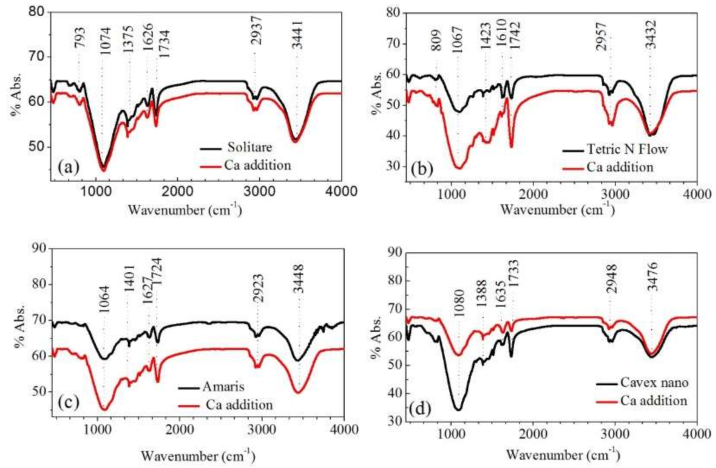

Figure 1. FTIR of %9 Ca added and the commercial samples.

3.2. Micro Structural Analysis.

XRD pattern of the samples for pure and including of 9 % Ca of the materials are given in Figure 2. It is easily seen from the XRD pattern that there are two solid phases, one is the organic binder such as BisGMA, UDMA and TEGDMA that gives a broad halo in the XRD pattern between 20-30^o and the second is the inorganic materials which give the peaks in the XRD pattern such as Luicide, Al_2O_3, SiO_2, etc. The other reason of the observation of the broad halo in the XRD pattern can be due to the overlap of the peaks belongs to the phases and the nano-sized particles of the inorganic content as seen in the Figure 2. So, the amorphous-like structure of the XRD pattern were observed for the samples which are named as solitare, amaris and cavexnano and the sample named tetrik N-flow have huge peaks when compared the others. The possible phases of this sample were determined as SiO_2, $Al_2BaSi_2O_8$, WO_3 for pure sample and CaO for the modified samples.

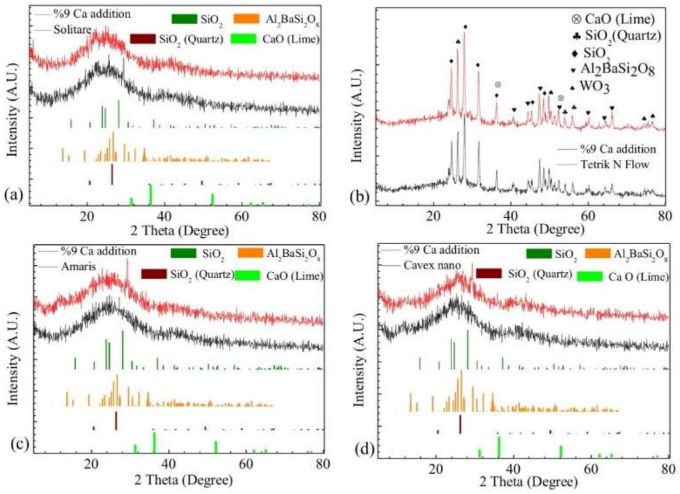

Figure 2. XRD of %9 Ca added commercial samples.

For determination of the structural change by Ca addition in the dental composite resin, we performed EDX-dot mapping for all samples as given in the Figure 3a-d. The Ca added Cavex Nano showed two phase regions and the elemental distributions of these regions were also given in the Figure 3.a. According to Figure 3.a, we can say that A regions include homogeneous Si, Al, Ba compositions, B regions have Ca rich compositions. The elemental dot analyses of these regions were also given in Table 2. We can say that Ca didn't incorporate in the main matrix of the dental composite resin named Cavex Nano.

In Figure 3.b, the elemental distribution of the sample named Amaris was given and we observed three different regions that the main phase is named as A in the figure. A regions have the composition including Si, Al and Ba phases, the B regions consist of Si-rich phases. The C regions consist of Ca-rich phases. Tetrik N-flow composite have three different phase regions in the SEM images that A-region consist of Si, Al, Ba, Ge and W phases and the B-regions consist of Al and W rich compositions. The C-regions have Ca-rich phases as seen in the Table 2. Meanwhile, the dental composite resin named Solitare have three different regions that A indicate the phases including Al, Si and Ba and the B regions have the phases of Al and Si rich compositions and the C-regions have Ca-rich phases. According to surface analysis we observed unsolved or accumulated phases during to light application which is unwanted state for dental treatment and the dental materials named Cavex Nano and Tetrik N flow have almost homogeneous small grains with the lowest size when compared the others.

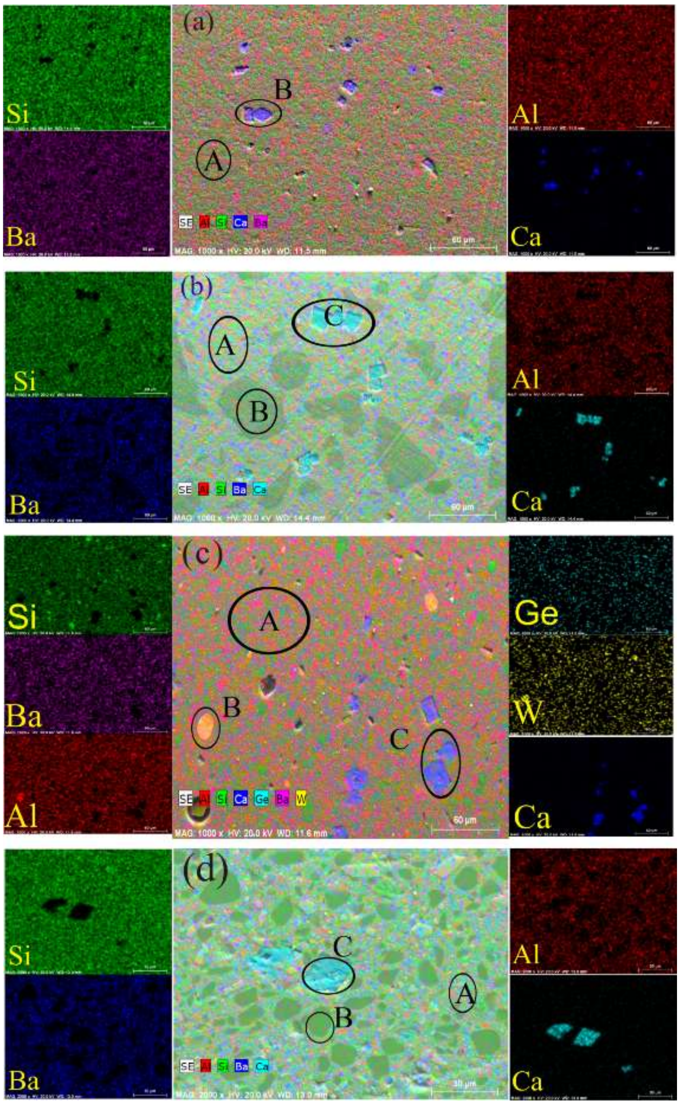

Figure 3. EDX dot mapping of the materials of **(a)** Cavex Nano, **(b)** Amaris, **(c)** Tetrik N-flow and **(d)** Solitare.

3.3. Optical Surface Photographs Analysis.

Optical surface photographs of the un-added commercial materials and 9% Ca added materials were given in Figure 4.a-h. The pure commercial sample surface after light application shows similar surface structure with the SEM analysis. The EDX dot mapping analysis and optical microscope images showed that there are arbitrarily dispersed particles for the composite resins named Amaris, Tetric N flow and Solitare, Figure 4 c, e and g, respectively. Although the filler particle does not have a homogeneous size distribution in the matrix for these samples, the sample named Cavex Nano have homogenous phase structure with the particle size of <1µm. The average particle size of the filler in the matrix of Amaris is about 1 and 30 µm and the particle size distribution is more non-uniform when compared the others. The particle size distribution of the samples named Tetric N flow and Solitare were changed from 1 to 10µm. The addition of Ca for all samples showed a particle size distribution of 1-10 µm and Ca were caused the formation of non-homogeneous main matrix since it was predicted that the Ca ions may cause the degradation of the grains in the main matrix of the composite resins. We also observed an accumulation of Ca particles for all samples.

Table 2. Elemental dot analysis of the surface of the materials in the Figure 3.

Ca doped Sample	A-regions	B-regions	C-regions	Un-added sample
Tetric N-Flow **(TF)**	%27.56 Al, %40,67 Si, 2.31 Ca, %11.14 Ba, %18,32 W	%57.5 Al, %0.46 Ge, %42.04 W	%20.97 Al, %55.48 Ca, %8.13 Ba, %15.41 W	%31.13Al, %45.11Si, %13.98Ba, %9.78W
Amaris Flow **(AF)**	%15.67 Al, %69.4 Si, %14.93 Ba	%9.31Al, %81.42 Si, %9.28 Ba	%100 Ca	Same A and B regions
Solitaire **(ST)**	%13.29 Al, %72.92 Si, %13.79 Ba	%11.95Al, %75.41 Si, %12.64 Ba	%0.65Al, %7.8Si, %88.67 Ca, %2.89Ba	Same A and B regions
Cavex Nano (CN)	%14.36 Al, %69.30 Si, %16.35 Ba	%4.94 Si, %95.06 Ca	-	Same with A region

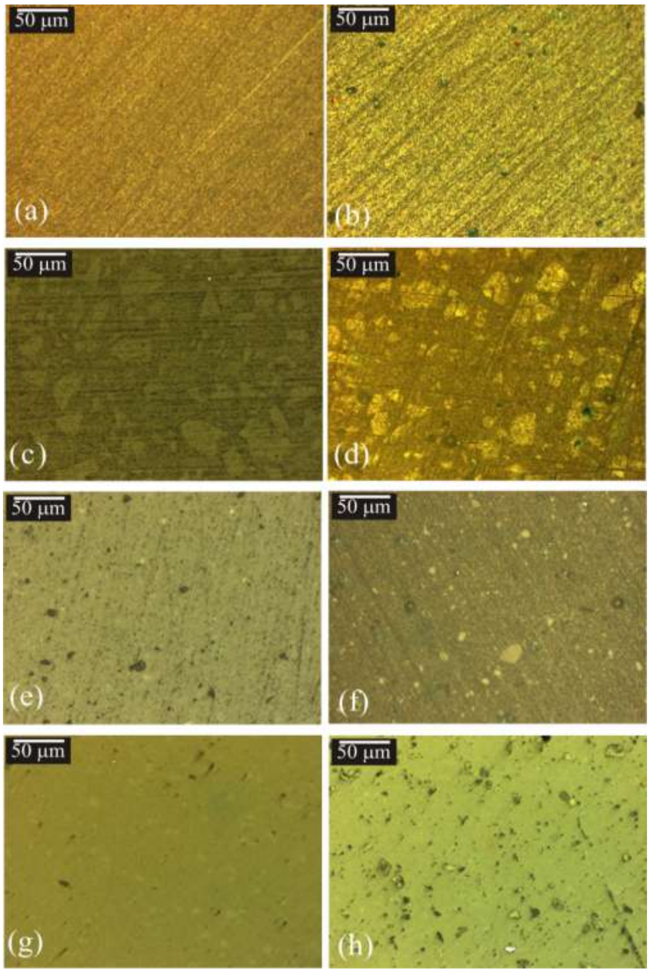

Figure 4. Optical images of (a and b) Cavex Nano, (c and d) Amaris, (e and f) Tetrik N-flow and (g and h) Solitare for un-added and 9% Ca added, respectively.

3.4. Hardness Analysis.

The hardness properties of the dental composite resins are intensely studied for improving the properties in the clinical studies of dental science [18-20].The hardness values of the samples were determined by using the average hardness of the matrix and filler particles. It is expected that the filler particles which have an almost uniform distribution within the resin matrix display high hardness value. In contrast, non-uniform distribution of filler particles within the resin matrix may cause a decrease in the hardness value [21].

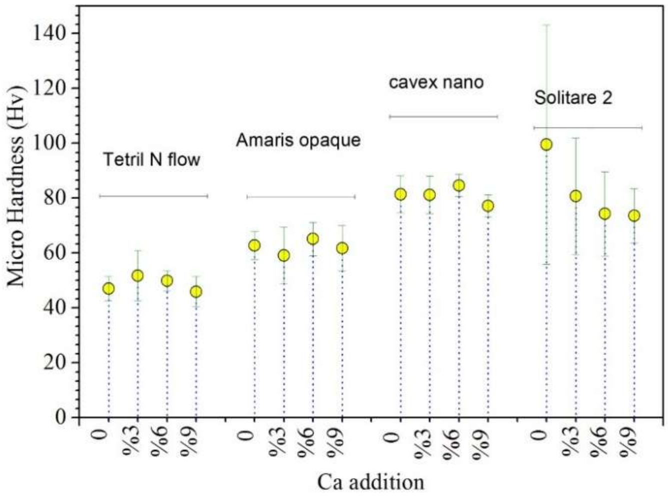

Figure 5. Micro hardness values of the dental composite depending on the Ca content in the main matrix.

When the effect on the mechanical properties of the Ca content in the main matrix were investigated, the hardness values of the Tetrik N flow were found as 46.55 Hv and it was increased by %3 Ca addition which is 51.6 Hv and then it slightly decreased by increasing Ca content from 51.6 Hv to 45.2 Hv as shown in the Figure 5. The hardness of Ca reinforcing Amaris showed almost same hardness value when compared the un-added samples. The material named Cavex nano showed an increase of the hardness value for the addition of %6 Ca which have a promising result for commercial applications. Although the highest hardness value was observed for the sample named Solitare as 98.6 Hv, the Ca addition caused a fast decrease of the hardness value from 98.6 Hv to 72.4 Hv by increasing Ca content. So we concluded that Ca content in the resin matrix exhibit a strong correlation due to the change of the hardness by Ca addition.

3.5. Thermal Conductivity.

The thermal conductivity in dental materials is one of the important cases for the improving the physical properties of the dental composites [22, 23]. To simplify the determination of heat conduction coefficient, we take into account one dimensional systems since the experimental setup is based on the one dimensional heat transfer from heater block. The samples have cylindrical geometry which is important for the solving of the heat transfer equation. One dimensional heat equation can be given as [24];

$$\frac{\partial^2 T}{\partial x^2} = \frac{1}{\alpha}\frac{\partial T}{\partial t}$$

where α is the thermal diffusivity of the sample. The solution of this equation for the rectangular infinite can be given as [24]:

$$T = \sum_{n=1}^{\infty} \frac{4\sin(\lambda_n)}{2\lambda_n + \sin(2\lambda_n)} e^{-\lambda_n^2 \tau} \cos(\lambda_n x/L)$$

Where the λ_n can be found from the equation of$\lambda_n \tan(\lambda_n)$=Biot number, t is the dimensionless time, *x* is the distance, and L is the thickness of the plane from the center.

The temperature of the sample changes from the initial temperature T_i to the temperature of the heating blocks T_s and so the equation for the relation of temperature gradient and thermal conductivity can be given as [24]:

$$Q = -\kappa\frac{dT}{dx}$$

where Q is the heat flux, T is the Temperature in Kelvin and κ is the thermal conductivity coefficient of the materials. The energy of heat into the materials can scatter during the conduction from hot region to cold region by phonon in the lattice. The phonon scattering strongly depends on the structure, lattice symmetry, dislocation and impurities in the materials. The determination of the heat flux is one of the important issues since some of the energy will be lost from the environment and setup of the system. So, we calibrated the system by using a piece of Al which the dimensions and thermal conductivity coefficient were known and the thermal conductivity of the pure and Ca added samples are relatively measured. It is well known that the thermal conductivity of the oxide samples have almost same value above 200 K and we calculated the steady state thermal conductivity values of the undoped and Ca doped samples which are given in the Figure 6. Although the κ values of the commercial materials are low enough to use in dental treatment which it was expected have the lowest thermal conduction of the heat from surface to dental pulp nerves, while the lowest κ was obtained as 2.79 W/mK for Tetrik N flow, the highest value (=5.01W/mK) belongs to Amaris among the other materials in this study as seen in the Figure 5.

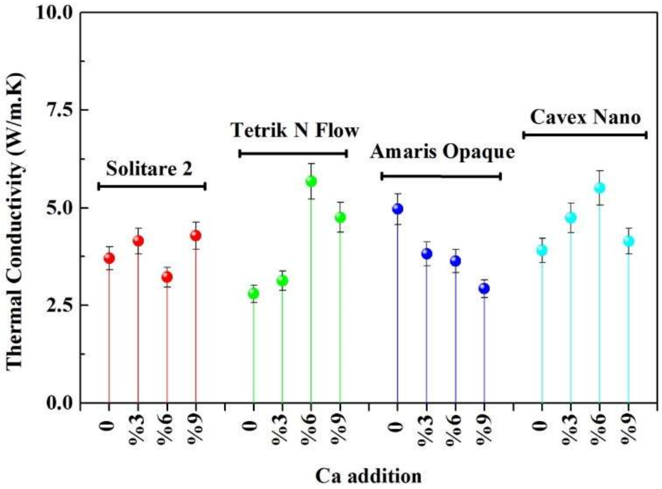

Figure 6. Room temperature thermal conductivity data of the commercial samples by Ca addition.

The κ values of Solitare show almost same value with Ca addition in the sample. The thermal conductivity of the samples named as Tetrik N Flow and Cavex Nano were increased up to %6 Ca additions and then started to decrease with increasing Ca content. The sample named Amaris showed the best performance by Ca addition that the κ values decreased from 4.96W/mK to 2.92W/mK which is a promising result for commercial applications.

4. CONCLUSIONS

The Dental composite resins can be classified by the type of the filler particles for the investigation of the structural, physical properties and the clinical applications [25-28]. The filler material technology is particularly significant because filler contents used by different research groups are affected the mechanical, heat conducting and structural properties of the composite resins [29-32]. For the improving of the physical and dental properties of the composite resins, the manufacturers used Ti, Al, Ba, Mo, Zr, W, etc. [33-35]. In this study, we used an additive material as CaO (at ratio of 3 %, 6 % and 9 %) for the commercial dental composite resins and it was investigated the effects on the physical and mechanical properties.

According to XRD analysis, it was observed a broad halo which can be due to organic binder and overlapping of the filler materials that the possible filler materials were shown in the Figure 2. The main phases of the samples were found as SiO_2 and $AlBaSi_2O_2$ with amorphous organic structure and the other inorganic phases are changed from one commercial material to another. The sample named Tetrik N flow has sharp XRD peaks when compared with the other dental resins in this study and it can be due to the high content of the crystalline filler particles in the main matrix. The Ca peaks in the XRD pattern which it was added to the commercial dental materials in this study didn't observed due to low content and overlapping of the crystalline peaks.

All samples in this study are same phases in XRD pattern such as SiO_2, Al_2O_3 and $BaAl_2Si_2O_8$. The minor phases in the samples may not be seen in the XRD pattern.

It is well known that the matrix particles with strong bonding can have the ability of the high mechanical strength. Earlier studies about the chemical bonding between the resin matrix and filler showed that they have higher wear resistance than the other type of materials [36, 37]. In contrast, large filler particles have less wear resistance properties [38]. Vilchis and colleagues [39] mentioned that the shape of the filler particles in the matrix of the composite materials may cause a change in the hardness values depending on the size and content of the filler particles. They found that the bond between the resin matrix and filler particles show an increase of the hardness.

In this study, the EDX dot mapping and Optical microscope analysis of the composite resins showed the surface structures which the filler particle size, shape and content is an important effect on the dental properties of the composite resins. Jaarda et al.[40] stated that filler particle numbers and sizes of composite resins affected the main properties of dental resins which were consistent with our study. We found that after adding Ca to the composite resins, the particle size of the materials almost didn't affect. It is well known that micro-hardness values of the composite resins show a strong relationship between the particle size and the mechanical strength. So the obtained hardness values didn't change much which support above discussion. It is mentioned by Vilchis et al. [39] that the chemical compositions of the composite resins were different in every composite resin. Filler contents show interesting differences in elemental composition and Si seem to be a common filler component.

After elemental dot analyses, interestingly we found that, tungsten (W) have been identified in the sample named Tetrik N Flow. Tungsten material is an agent that increases the mechanical properties of the composite resins. When we analyzed the thermal conductivity values, Tetrik N Flow has a minimum value and it can be due to the tungsten element in this composite. It can be predicted that the W ions shows heat retention properties in the composite resin and can cause a decrease of the thermal conductivity. The thermal conductivity of Cavex Nano was changed much when compared the others and it can be related the increase of the particle size by Ca addition. So, it can be said that the thermal conductivity can be considered as of the increasing particle size.

Vicker's hardness data of the composites was used to determine the surface hardness with respect to filler loading. The sample named Solitare showed a decrease on the micro hardness by Ca addition and we concluded that the decrease of the hardness by the addition of Ca ions may be due to the non-bonding structure of Ca in the sample.

According to Hardness results, it is found that the addition of Ca in Cavex Nano and Tetrik N flow have better results when compared undoped material and it can be used in these materials. Berger et al. examined the effectiveness of the surface hardness of the particle size. Their study suggests that the particle size of composite resins do not directly impact the surface hardness [41] Similar results were observed in our study.

The composition of composite resins is highly complex. By means of EDX in this study, general information on the elemental compositions of the composite resins evaluated. Common resin matrix elements such as Al and Si were detected in all the materials with comparable concentrations. The filler contents showed great diversity, whereby elements such as Ge, W, K and Na were detected.

One of the most interesting findings of this study was the elemental content of the Tetrik N flow. Which Tungsten (W) was detected in this material with the EDX dot mapping analysis. Although tungsten improves the mechanical properties of materials, micro hardness values of Tetrik N flow were found lowest when compared with the other materials in this study.

In our study, the adding of Ca in the commercial materials cause two types of behavior; one is settling in the intergrain region without any chemical reaction. In this case, while the mechanical properties go worse due to multiphase structure of the matrix, the heat conduction was increased since this region may behave as a heat reservoir which has higher heat storage properties and the thermal conductivity can increase. Second is the Ca diffuse into the main matrix that the materials which have homogeneous phase formation with the multiphase structure have stronger bonding character than that of the un-added composition and so the mechanical properties can increase by Ca addition in the dental composite resins. The thermal conductivity of the commercial and Ca added samples showed different behavior depending on the commercial dental composite. In this study, the following conclusions were drawn:

1. The mechanical properties of the materials strongly depend on the filler particles size and elemental distribution of the composite resins.
2. Though Ca added some composite resins could offer the increasing mechanical and physical properties, further studies should be performed for the better results for commercial applications.
3. The mechanical strength can improve and the heat conductivity of the commercial dental composite decrease for a certain level of Ca addition.

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6. ACKNOWLEDGEMENTS

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