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The evaluation of thermogravimetric analysis and differential thermal analysis of different glass ionomer cements

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Abstract

The purpose of this study is to investigate the evaluation of Thermogravimetric analysis and differential thermal analysis of 5 different glass ionomer cements; EQUIA Forte Fil (GC, Tokyo, Japan, EQF), Fuji II LC Light-Cured Glass-Ionomer Cement for Restorative Filling (GC Corp, Tokyo, Japan, F2S), Fuji IX Conventional glass ionomer Cement for Restorative Filling (GC Corp, Tokyo, Japan, F9S), Glass carbomer (Glass Carbomer, GCP), Ionoseal (Glass ionomer cement, VOCO, Cuxhaven, IOS). They were filled in teflon molds (height: 4 mm, width: 6 mm) and stored in distilled water at 37 °C / 24 hours. The Thermogravimetric analysis and differential thermal observations were made on a Schimadzu DTG-60 AH by heating from room temperature to 950 °C at a heating rate of 10 °C/min. The average sample size was 20 mg and the nitrogen flow rate was 50 ml/min. DTA was used to determine the temperature of the onset crystallisation and melting. TG was used to determine the weight loss by the effect of high temperature.

Keywords: Glass ionomer cement, Thermogravimetric analysis, differential thermal analysis

1. Introduction

In today's modern dental practice, advances in adhesive techniques and protective and developed in accordance with preventive concepts and the protection of healthy tooth tissues is at the forefront. Ensuring the integrity of dental hard tissues and single-session direct restoration applied for protection techniques become more preferred in this context. As permanent direct restorative materials amalgam, composite resin and glass ionomer cements. It is used in routine clinical applications. Restorative material selection, areas of use, advantages and disadvantages important to consider are issues ^[1, 2].

The chemical content of glass ionomer cements and improved due to changes in powder-liquid ratio have expanded the range of applications in clinical practice. Glass ionomer cements are more aesthetic than amalgam restorations, but this is not as successful as composite resins. However, the availability of anticariogenic potentials due to fluoride release, biocompatibility and chemical adaptations to tooth tissues have made glass ionomer cements a featured material group ^[3, 4].

Physical and mechanical properties improved from other aspects; mechanical properties such as fracture toughness, hardness and abrasion resistance increase their use as restorative materials in areas exposed to intense stress in the back regions. For this purpose there are different studies on the physical and mechanical properties of glass ionomer cements. But there are few studies on Thermogravimetric analysis and differential thermal analysis of glass ionomer cements. For this purpose, Thermogravimetric analysis and differential thermal analysis of the materials were performed in this study.

2. Materials and methods

Glass ionomer cements were used in five different brands in the study. These materials; EQUIA Forte Fil (GC, Tokyo, Japan, EQF), Fuji II LC Light-Cured Glass-Ionomer Cement for Restorative Filling (GC Corp, Tokyo, Japan, F2S), Fuji IX Conventional glass ionomer Cement for Restorative Filling (GC Corp, Tokyo, Japan, F9S), Glass carbomer (Glass Carbomer, GCP), Ionoseal (Glass ionomer cement, VOCO, Cuxhaven, IOS).

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The study was conducted *in vitro*. After placing the restorative materials in the molds (4mm height, 4 mm width), strip bands were placed on the upper and lower surfaces of the molds and pressed to form a flat surface with glass slides and then with LED light device (Elipar Freelight II, 3M-ESPE, St. Paul, MN, USA) and manufacturer's instructions 20/40 sec polymerized. Some materials were expected to be chemically polymerized. These cured materials were removed from the molds. As a result, a sample of 4 mm in diameter and 4 mm in height was obtained. Samples were stored in distilled deionized water at 37 ° C for 2 weeks.

The TG/DTA observations were made on a Shimadzu DTG-60 AH by heating from room temperature to 950 °C at a heating rate of 10 °C/min. The average sample size was 20 mg and the nitrogen flow rate was 50 ml/min. DTA was used to determine the temperature of the onset crystallisation and melting. TG was used to determine the weight loss by the effect of high temperature.

3. Results & Discussion (Times New Roman, 12, Bold)

A general description of the thermal analysis according to ICTAC and Mackenzie (1979): 'A group of techniques where the physical properties of the substance and / or reaction products are measured as a function of the temperature when a controlled temperature program is applied. In a thermal analysis method according to the present invention, a physical property should be measured, a temperature program expressed as a function of the measurement temperature, and an analysis controlled temperature program ^[4-6].

In a complete thermal analysis system mass loss of material, transformation temperatures and energies, dimensional changes, viscoelastic properties are observed. These parameters can be used to illuminate chemical reactions and dynamic properties, composition analysis, product quality control ^[7].

Various thermal analysis methods have been developed taking into account the differences between mass, volume and temperature during controlled heating of a substance which is thermally inert with the substance to be investigated. The most common methods of thermal analysis are thermogravimetry (TG), differential thermal analysis (DTA) and differential scanning calorimetry (DSC). These three methods give very chemical information about the physical information of the sample ^[8].

Thermogravimetry (TG); the mass is measured as a function of time or temperature while the sample is subjected to a controlled temperature program. In Thermogravimetric analyzes, the change in the mass of the sample is plotted against the temperature. These graphs are called the mass loss curve, the thermal distortion curve, or the thermogram. In a thermogram, ordinate is the mass, and abscissa is the time or temperature ^[9].

In order for a sample to be thermogravimetrically analyzed, the temperature change must make a change in the mass of the sample. However, transformations that cause energy change may not necessarily produce a mass change. In such cases, nothing can be observed in the Thermogravimetric examination ^[6].

In Thermogravimetric analysis, the sample is heated in a special crucible or scales cuvette in the oven according to a certain temperature program. In the meantime, the mass is continuously recorded. In such a system, the beam emerging from a source is passed through a thin hole and dropped onto a pair of photocells. When the balance is balanced, the intensity of the light falls on the photocell. When the balance

is out of balance, the beam sealer at the end of the scale and the slit on the top of the scale are displaced. Therefore, the intensity of the light falling on the photosensor changes. This change signal is amplified in the electronic circuit and the recorder is sent. This signal is a measure of the mass change directly in the sample. At the same time, this difference signal is sent to the moving jaw attached to the scale so that the balance reaches the balance position again ^[5].

In thermogravimetry, thermostats are used which operate at a maximum range of 5-20 mg. Only the sample container and the holder of the scale are located in the oven, the other parts are insulated. The temperature ranges of furnaces used in thermogravimetry equipment are usually room temperature to 1500 ° C. Heating or cooling rates can reach up to 200 ° C / min. However, generally heating rates of 5-25 ° C / min are used. To protect the balance from oven heat, the oven is insulated and there is a cooling system outside ^[4].

Differential Thermal Analysis (DTA) In differential thermal analysis, the same temperature program is applied to a thermally inert comparison material with the sample, and the temperature difference between the two is measured. Usually, the sample and the reference material are heated so that the temperature of the sample increases linearly over time. In the DTA technique, a differential thermocouple is immersed in a sample and a thermally inert reference material, and the temperature measurement is carried out with this system. The net voltage output is zero when the temperature of the sample and the reference material are equal. If the temperatures of these two materials are different, the voltage difference of the system is a direct measure of the temperature difference. A graph showing the variation of this temperature difference versus time or temperature of one of the substances forms the DTA thermogram. The physical or chemical change caused by the temperature creates a maximum curve that is a function of the time of the temperature. You can comment on temperature and conversion speed by taking advantage of this maximum. In a DTA thermogram, the temperature difference is in the ordinate and the abscissa is in the time or temperature ^[3].

If the heat flow in the furnace, sample and reference material are the same, the temperature difference is zero and the recorder will emit the mains. If a temperature change occurs in the sample, the temperature distribution in the sample is different from that of the inert substance, and the temperature changes are recorded as deviations in the main stream. The direction of this slope depends on the temperature difference distribution between the sample and the inert substance and shows the structure of the thermal process. Endothermic processes are negative, and exothermic processes cause a positive temperature difference ^[9].

Events such as melting, evaporation, sublimation, absorption and desorption are endothermic. Adsorption is usually an exothermic change. Crystal phase changes can be exothermic or endothermic. Chemical reactions form both endothermic and exothermic peaks. DTA can be used for both qualitative and quantitative analysis. The temperature at which the pigments are observed and the peaks are used for the identification of the composition of the sample. The peak area is directly proportional to the reaction temperature and the amount of the substance. The proportionality factor is influenced by experimental parameters such as the initial temperature of the oven, the heating rate, and the particle size of the sample. The shape of the curve helps in the evaluation of the reaction kinetics ^[7].

According to the results of EQF thermogravimetry analysis;

the heating rate of the thermogram is 5 K / min. and is performed in the atmosphere of air. The material is stable up to 50 ° C, not mass loss. A loss of mass at 100 ° C has occurred. In a thermogram, if this mass reduction is around 100 ° C, it corresponds to evaporation of water. So at this temperature we can say that the water in the sample is evaporated. The substance is stable up to 400 ° C. After 400 ° C, the matter is degraded and a mass decrease comes to the foreground (Figure 1). Any event that the heat is absorbed or released by DTA can be observed. Events such as melting, evaporation, sublimation, absorption and desorption are endothermic. Adsorption is usually an exothermic change. Crystal phase changes can be exothermic or endothermic.

According to the results of GCP thermogravimetry analysis; the heating rate of the thermogram is 5 K / min. and is performed in the atmosphere of air. The material is stable up to 55 ° C, not mass loss. A loss of mass at 100 ° C has occurred. In a thermogram, if this mass reduction is around 100 ° C, it corresponds to evaporation of water. So at this temperature we can say that the water in the sample is evaporated. The substance is stable up to 450 ° C. After 450 ° C, the matter is degraded and a mass decrease comes to the foreground (Figure 2).

According to the results of FS9 thermogravimetry analysis; the heating rate of the thermogram is 5 K / min. and is performed in the atmosphere of air. The material is stable up

to 40 ° C, not mass loss. A loss of mass at 100 ° C has occurred. In a thermogram, if this mass reduction is around 100 ° C, it corresponds to evaporation of water. So at this temperature we can say that the water in the sample is evaporated. The substance is stable up to 410 ° C. After 410 ° C, the matter is degraded and a mass decrease comes to the foreground (Figure 3).

According to the results of FS2 thermogravimetry analysis; the heating rate of the thermogram is 5 K / min. and is performed in the atmosphere of air. The material is stable up to 55 ° C, not mass loss. A loss of mass at 100 ° C has occurred. In a thermogram, if this mass reduction is around 100 ° C, it corresponds to evaporation of water. So at this temperature we can say that the water in the sample is evaporated. The substance is stable up to 350 ° C. After 350 ° C, the matter is degraded and a mass decrease comes to the foreground (Figure 4).

According to the results of IOS thermogravimetry analysis; the heating rate of the thermogram is 5 K / min. and is performed in the atmosphere of air. The material is stable up to 300 ° C, not mass loss. After 300 ° C, the matter is degraded and a mass decrease comes to the foreground (Figure 5).

3.1 Tables and Figures

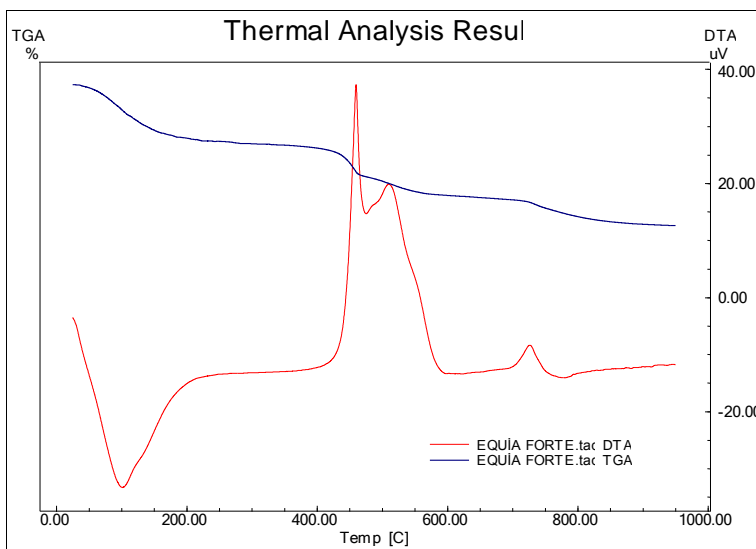


Fig 1: The Thermogravimetric and differential thermal analysis of EQF

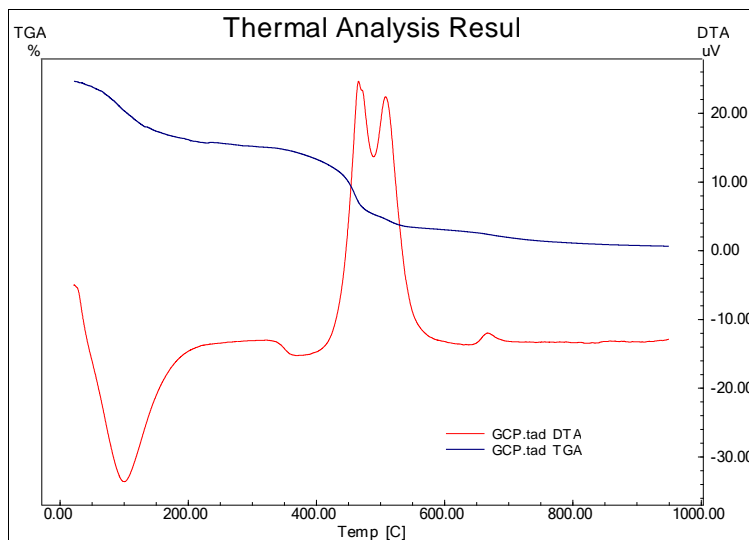


Fig 2: The Thermogravimetric and differential thermal analysis of GCP

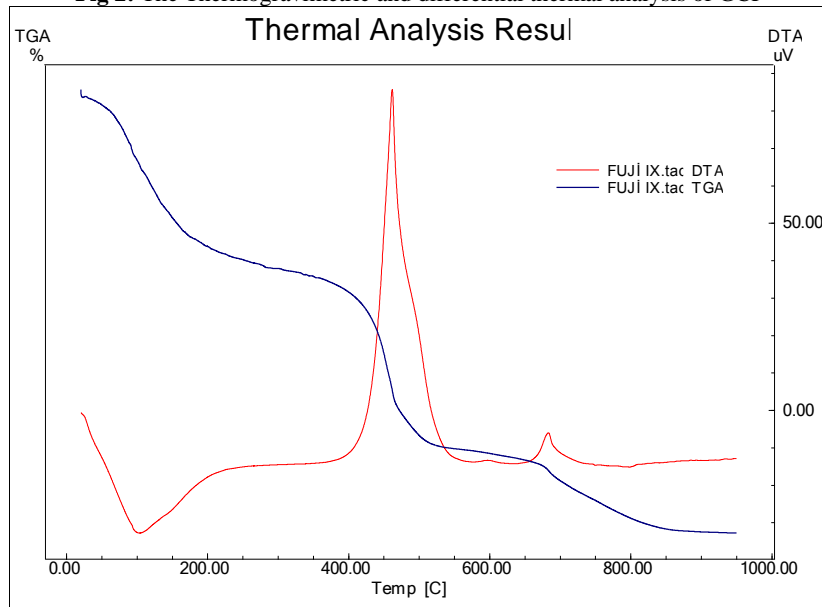


Fig 3: The Thermogravimetric and differential thermal analysis of FS9

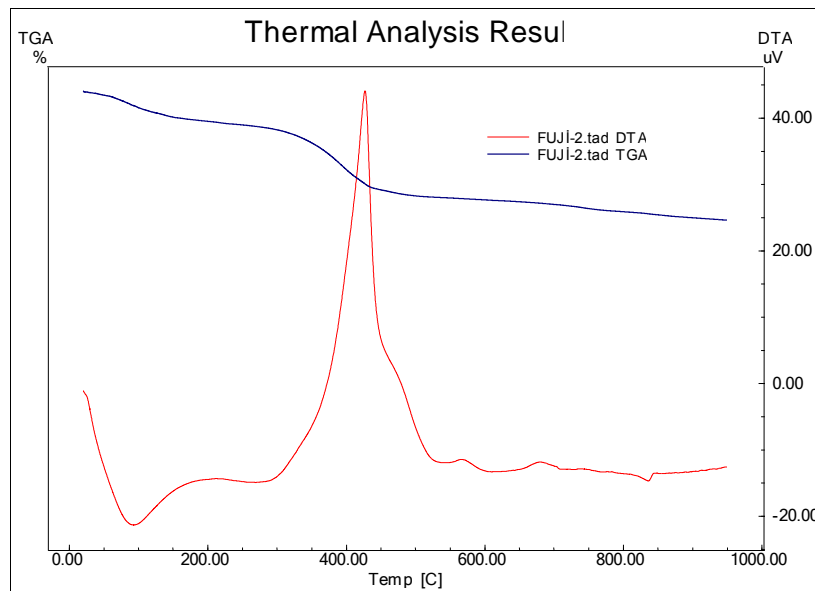


Fig 4: The Thermogravimetric and differential thermal analysis of FS2

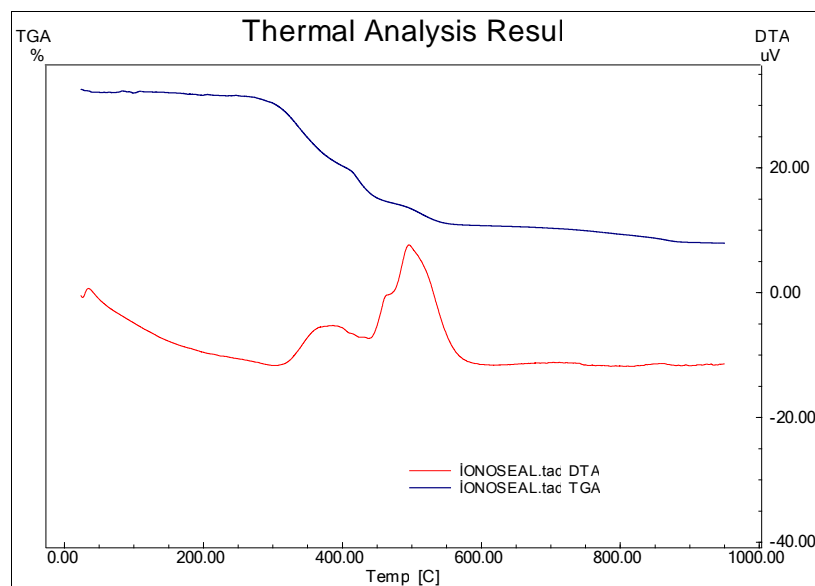


Fig 5: The Thermogravimetric and differential thermal analysis of IOS

4. Conclusions

In accordance with the exothermic peaks observed in DTA analyses; equia forte, GCP, Fuji IX and Ioneseal showed two exothermic peaks and these peaks mean the crystallisation occurred. The Fuji 2 sample both exothermic and endothermic peaks were observed. The exothermic peak refers to crystallisation and endothermic peak refers to melting.

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