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M.Sc. in Engineering Physics

MUHAMMED HALIDO ĞLU

REPUBLIC OF TURKEY
GAZİANTEP UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

A STUDY ON THE DOSIMETRIC PROPERTIES OF POLYCARBOXYLATE CEMENT USED IN DENTAL TREATMENTS

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Supervisor
Prof. Dr. Hüseyin TOKTAMIŞ

by
Muhammed HALIDOĞLU
January 2022

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Muhammed HALIDOĞLU

ABSTRACT

A STUDY ON THE DOSIMETRIC PROPERTIES OF POLYCARBOXYLATE CEMENT USED IN DENTAL TREATMENTS

HALIDOGLU, Muhammed
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44 pages

Zinc Polycarboxylate cement is one of the few dental materials that demonstrate true adhesion to tooth structure. The powder is primarily zinc oxide, and the liquid is polyacrylic acid or a copolymer of that acid. In this study, the dosimetry properties of Zinc Polycarboxylate cement using thermoluminescence (TL) were investigated and determined the effectiveness of its use as a good dosimeter. As a result, the sample shows a good TL properties with three main peaks found around 140 ℃, 220 ℃ and 330 ℃. It has a wide linear dose response between 144 Gy and 2.3 KGy and good reusability with eight cycles of measurement.

Keywords: Thermoluminescence, Dosimetry Properties, Zinc Polycarboxylate

ÖZET

DİŞ TEDAVİLERİNDE KULLANILAN POLİKARBOKSİLAT SEMENTİN DOZİMETRİK ÖZELLİKLERİ ÜZERİNE BİR ARAŞTIRMA

HALIDOGLU, Muhammed
Yüksek Lisans Tezi, Fizik Mühendisliği
Danışman: Prof. Dr. Hüseyin TOKTAMIŞ
Ocak 2022
44 sayfa

Çinko Polikarboksilat çimento, diş yapısına gerçek yapıştırıcı görevi gösteren birkaç diş materyalinden biridir. Toz esas olarak çinko oksittir ve sıvı, poliakrilik asit veya bu asidin bir kopolimeridir. Bu çalışmada, Çinko Polikarboksilat çimentonun termolüminesans (TL) yöntemi kullanılarak dozimetrik özellikleri araştırılmış ve iyi bir dozimetre olarak kullanımının etkinliği belirlenmiştir. Sonuç olarak, numune 140 °C, 220 °C ve 330 °C civarında bulunan üç ana termolüminesans tepe noktaları ile net bir TL ışıma eğrisine sahiptir. 144 Gy ile 2.3 kGy arasında iyi bir lineer doz yanıtına ve sekiz ölçüm döngüsü ile iyi tekrarlanabilirliğe sahiptir..

Anahtar Kelimeler: Termolüminesans, Dozimetrik Özellikleri, Çinko Polikarboksilat.

‘’Dedicated to my family, Cebrail and Ravan’’

ACKNOWLEDGEMENTS

I would like to thank my supervisor, Prof. Dr. Hüseyin TOKTAMIŞ for his guidance and support throughout the study. I am thankful for his encouragement and motivation.

I would like to express my love and gratitude to my family for their support, always best wishes.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

Page

CHAPTER I
INTRODUCTION

1.1.Introduction

The phenomena of light and luminescence have always been important to humankind since ancient times, as man has been persistent in his attempts to search for the causes of light and luminescence in general. Aurora, rotting fish, and glowing worms are some of the most critical daily sightings that ancient humans saw for long periods, fascinated by its beauty and the beauty of the luminescence it emitted. In his book "A History of Luminescence: From the Earliest Times until 1900," E. Newton Harvey mentioned many stories from ancient civilizations to the modern era about light and glowing that old man saw and tried to explain these phenomena. It is considered the oldest written text on luminescence in Chinese civilization during the "Song Dynasty." The text talks about a jewel that has been described as a sacred stone, which has been called "hashi-no-tama," that illuminates itself and spreads light. [1]

In one of the observations made by Robert Boyle to the Royal Society of London in the United Kingdom in 1663, he observed a new type of light glimmering from a crystal (natural diamond) when he warmed it slightly using his hands. Robert Boyle may have been the first to describe phosphorescence and thermal luminescence through his studies of diamonds, shining wood, fish, and meat.

This phrase first arose around the end of the eighteenth century by the German scientist Eilhardt Wiedemann, where the term was derived from Latin, and the term lumen means light. The materials that exhibit such a phenomenon were defined as luminescent materials. The term phosphorous was coined in the seventeenth century by the Italian scientist Vincentinus Casciarolo. The first classification of phosphorous by type of excitation was by Ellard Weidmann, and the first to report thermoluminescence was the scientist Schmidt in 1895. But the real study began in 1940 in the USA by Farrington Daniels and his group at the University of Wisconsin.

1.2. Luminescence

The phenomenon of luminescence can be defined as the light emitted by exciting an electron of the atom in the ground state (low energy state) to an excited state of higher energy, where the electron returns this absorbed energy by retreating to the ground state in the form of light in the visible region. There are many examples in nature; These include -but are not limited to- glow worms, fireflies, some marine bacteria, etc. This phenomenon, the "luminescence phenomenon," is one of the most important practical phenomena that concern many different fields such as archeology, geology, medicine, chemistry, and physics. It has many applications and broad uses in research and development.

One of the most important laws regulating the phenomenon of luminescence is the Stokes law; When a substance absorbs energy from radiation, it re-emits this energy in the form of light of a longer wavelength. The wavelength emitted is a hue of the material used and not the radiation absorbed. The emitted light can be within the visible or invisible spectrum (ultraviolet or infrared radiation) and does not include black body radiation. This process (luminescence) is based on two main steps: excitation of the atom from a lower energy state to a higher energy state and light emission (photons).

After absorption of radiation, the process of emitting light takes a characteristic time τ_c. This time is considered one of the parameters that classify and sub-classify the luminescence process into two main parts, fluorescence and phosphorylation.

Accordingly, by knowing the period for the emission to occur, the luminescence can be classified into:

1. Fluorescence: where the characteristic time value is $\tau_c < 10^{-8}\ s$.
2. Phosphorescence: The characteristic time value is $\tau_c > 10^{-8}\ s$; Both classifications are temperature dependent.

Also, the phenomenon of Phosphorescence can be sub-classified into:

2.1. Long periods, which are called thermoluminescence (on which this thesis is based), and during which the characteristic time is within the range $minutes\ < \tau_c < 4.6 \times 10^9\ years$, and;

2.2. Short periods, during which the characteristic time is $\tau_c < 10^{-4}\ s$.

Each of these aforementioned classifications and sub-classifications has advantages and characteristics relied upon in much scientific research in various fields.

The figure below shows the classifications and sub-classifications of the luminescence phenomenon in proportion to the characteristic time τ_c.

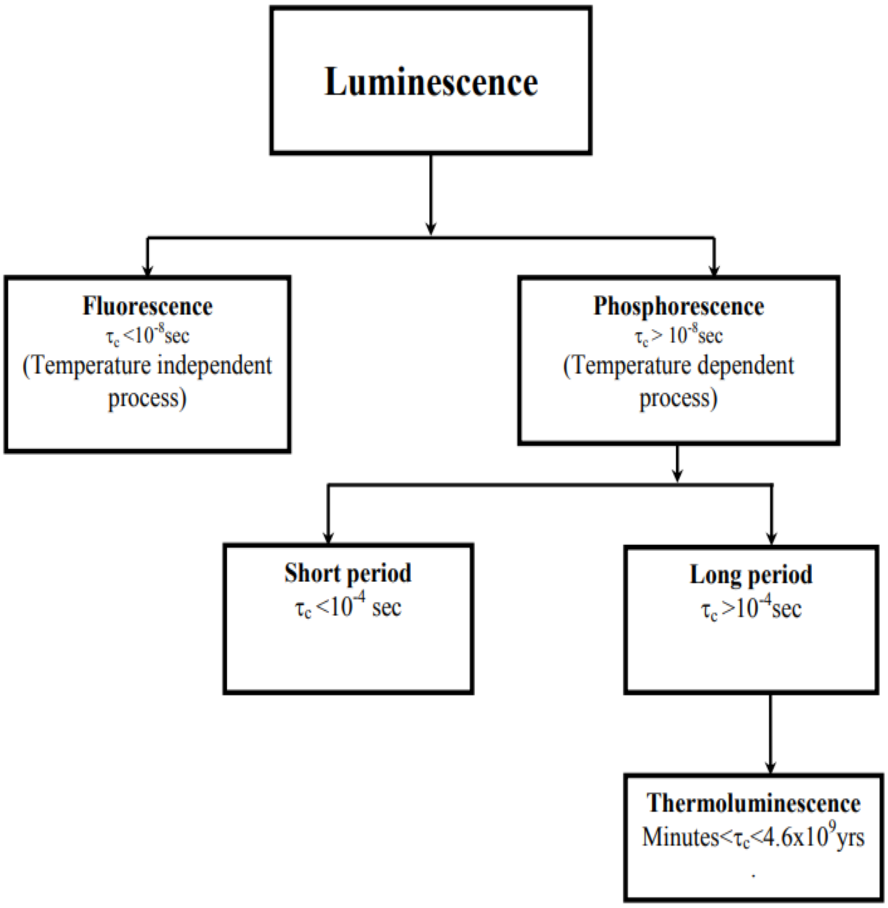


Figure 1.1 Classifications and sub-classifications of the luminescence phenomenon [2]

1.3.Thermoluminescence

The thermally stimulated luminescence or the light emitted by thermally stimulated crystals is called thermoluminescence (TL). This stimulation or excitation can occur not only by heat, ionizing radiation, or non-ionizing radiation but also by pressure (piezo-TL), friction (tribo-TL), or light (photo-TL). Where the last three modes are referred to is called "spurious TL."

The curve resulting from plotting the light emitted intensity I(T) as a function of temperature T is called the glow-curve. This curve contains one or more peaks, and each peak is ideally linked to an electron or a hole trapping center. Thus, Robert Boyle may have described TL for the first time in his historical notes of the Royal Society of London in 1663, where he heated the diamond and described that process with the phrase, “I also brought it to some kind of glimmering light, by taking it into bed with me and holding it a good while upon a warm part of my naked body.

The in-depth study of thermoluminescence began in the early twentieth century, and in 1927 Wake published a paper on the thermoluminescence of fluorite stimulated by X-rays. In 1945, Randall and Wilkins developed the first quantitative theoretical calculation. Whence, detailed research studies and experiments on the properties of the thermoluminescence theory increased, and many research papers and books were issued, including but not limited to Horowitz in 1984; McKeever, Moscovitch, and Townsend in 1995; and Chen and Pagonis in 2011 [3].

In simple terms, thermoluminescence (TL) is defined as the phenomenon of a material (insulator or semiconductor) when it is thermally stimulated. This definition does not include the phenomenon of black body radiation resulting from heating the material, which leads to the emission of infrared radiation. The intensity increases with the increase in the temperature applied to the sample. Where TL occurs after the material is stimulated and electrons are transferred from lower energy levels to higher energy levels, based on this, three main conditions for the occurrence of the thermoluminescence phenomenon can be determined. Firstly, the material is either an insulator or semiconductor. Secondly, the material is exposed to radiation that causes ionization, giving it the energy to move electrons to higher energy levels. Third, flashing occurs after the material has been heated [1]. From the above, we can notice the difference in TL from the glow, as the TL cannot be produced by merely cooling the sample and reheating it again. Still, rather it must be re-stimulated with ionizing radiation and then re-heating. The TL storage capacity is a convenient feature for dosimetry applications [4].

The thermoluminescence theory characteristics described by understanding the energy band theory of solids in ideal crystal insulators or semiconductors. Since these materials have two energy bands, most of the electrons reside in the lower band, which is the valence band VB, and the second-highest band that the electrons can occupy is the conduction band CB. These two domains are separated by a gap called the forbidden bandgap or the energy gab E_g. However, electrons can have forbidden energies in a crystal if there are (or create) defects in the crystal's structure.

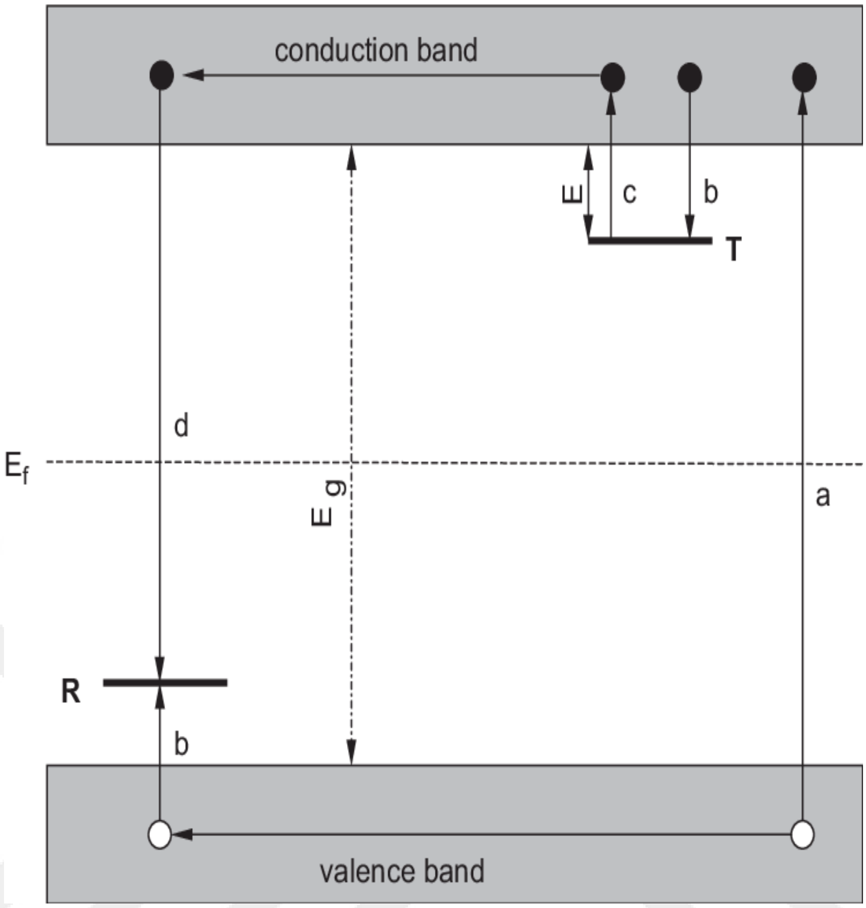


Figure 1.2 Simple model of energy level. [2]

1.4. Thermoluminescence Simple Model

The simple thermoluminescence model assumes two energy levels, the first located below the lower section of the conduction band (denoted by T). The second (denoted by R) is located above the top of the valance band (See figure 1.2) The highest level above the Fermi equilibrium level E_F is the T level, and this level is empty in the equilibrium state, that is before the ionization by radiation occurs. Therefore, the T level is a potential electron trap, and the R level is a potential hole trap and can act as rearrangement centers. When the material absorbs radiation of energy greater than the forbidden gap $h\upsilon > E_g$, ionization occurs in the valence electrons, leading to free electrons in the conduction band and free holes in the valence band (transition a in *Figure 1.2*). These free charge carriers either recombine or become trapped in the electron trap (T) or the hole trap (R) [4].

A certain amount of energy is released during direct recombination, stimulating a luminous center (which may coincide with the recombination center). When light is emitted, the luminescent center relaxes and returns to its ground state. Radioluminescence is the phenomenon of direct ($< 10^{-8}\ sec$) recombination of free electrons and holes under light emission. However, a fraction of charge carriers is trapped in semiconductors and insulators: electrons at T and holes at R. (transition b) in *figure 1.2*. The Arrhenius equation (*equation 1.1*) reflects the likelihood of an electron being released from the trap per unit time.

$$p = s^{-1} \exp\left\{-\frac{E}{kT}\right\} \tag{1.1}$$

Where; p is the probability per unit time, and s is called the attempt-to-escape factor or the frequency factor. In this model (TL simple model), the frequency factor s is considered to be not temperature dependent. E is the energy that the electron needs to break free from the trap to the conduction band (*figure 1.2*), and it is called the activation energy also expresses the depth of the trap, T is the absolute temperature, and K is the Boltzmann constant $K = 8.617 \times\ 10^{-5}\ eV/K$.

If the trap depth $E \gg KT_0$, where T_0 is the temperature at the start of the irradiation, the trapped electrons will remain so for a long time despite the radiation exposure. As it is known, holes and electrons form together and annihilate together (in pairs), so there are a number of holes in the R level (above Fermi equilibrium level E_F) equal to the number of electrons trapped in the T level (below Fermi equilibrium level E_F). This state is called a non-equilibrium state and returns to the equilibrium state governed by the coefficients s and E (Equation 1.1). Return to equilibrium state is always possible, but since the relaxation rate is slow due to the perturbation resulting from exposure to ionizing radiation was performed at a low temperature, the non-equilibrium state is unstable and will remain for an indefinite period. [5]

1.5. Applications of Thermoluminescence

In 1989 applications of a thermistor were discussed by Mahesh et al, which depend on the principle that the intensity of the emission is related to the total radiation dose received. The most important of these applications is the dating of geological samples, archaeological finds, and thermoluminescence dosimetry (TLD). There is a lot of recent research on the applications of TL in various fields, including semiconductors, defects in solid materials, glass and ceramics, and applications associated with petroleum exploration [2].

The present research on TL refer to high applicability in different fields such as archaeology, medicine, analysis of defects in solids, radiation dosimetry, geology, improve quality of some industrial products as glass and ceramics, and semiconductor products. in addition to radiation physics and petroleum exploration.

As mentioned previously, this study is concerned with determining the dosimetric properties of polycarboxylate cement to be used as a thermoluminescence dosimeter (TLD).

1.5.1. Thermoluminescence Dosimetry

The main parts of a thermoluminescence dosimeter (TLD) are a plate for placing and heating the sample, a photomultiplier tube (PMT) that detects the emission produced after heating, this tube's function is to convert the emission of TL into an electrical signal (this signal is proportional to the output photon) and an electrometer that records the electrical signal converted from the photomultiplier tube [6]. The main parts of the thermoluminescence dosimeter are shown in Fig. 1.3.

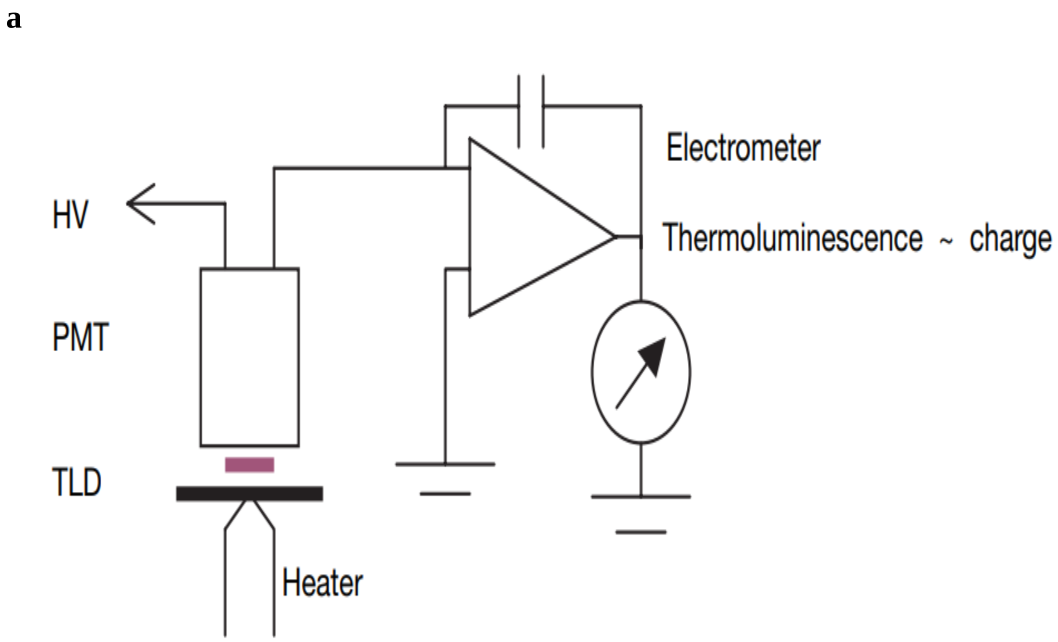


Figure 1.3 (a) The main parts of the thermoluminescence dosimeter [6]

b

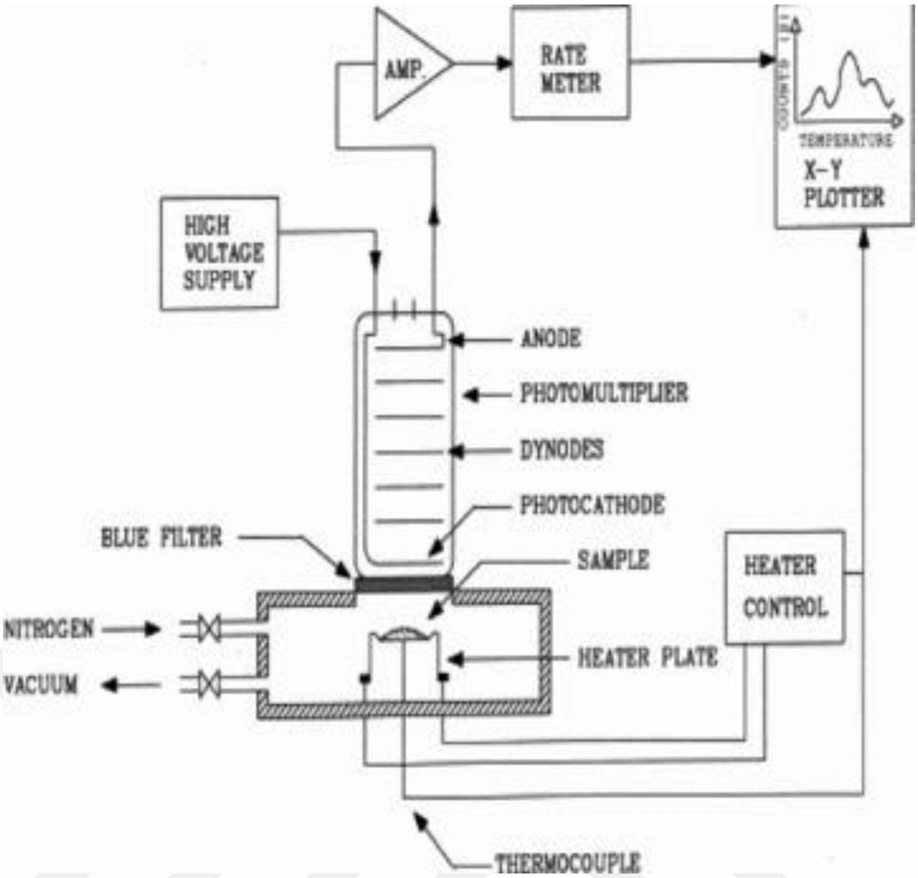


Figure 1.3 (b) Schematic diagram of thermoluminescence reader system[6]

Before using thermoluminescence dosimeters (TLD), they should be calibrated by applying some correction factors that would derive the absorbed dose from the thermoluminescence reading. These factors include fading, energy, and nonlinear dose-response.

Thermoluminescence dosimeters (TLD) have many geological, environmental, physical, and medical applications.

The intensity of thermoluminescence is proportional to the dosimetry temperature T. Keeping the heating rate constant makes the temperature T proportional to time t, allowing the thermoluminescence intensity to be displayed as a function of t if the TLD measuring device has a recorder output. The resultant curve is referred to as the TLD glow curve. In general, a thermoluminescence glow curve is obtained by plotting the emitted light versus the crystal temperature. (Figure 1.4).

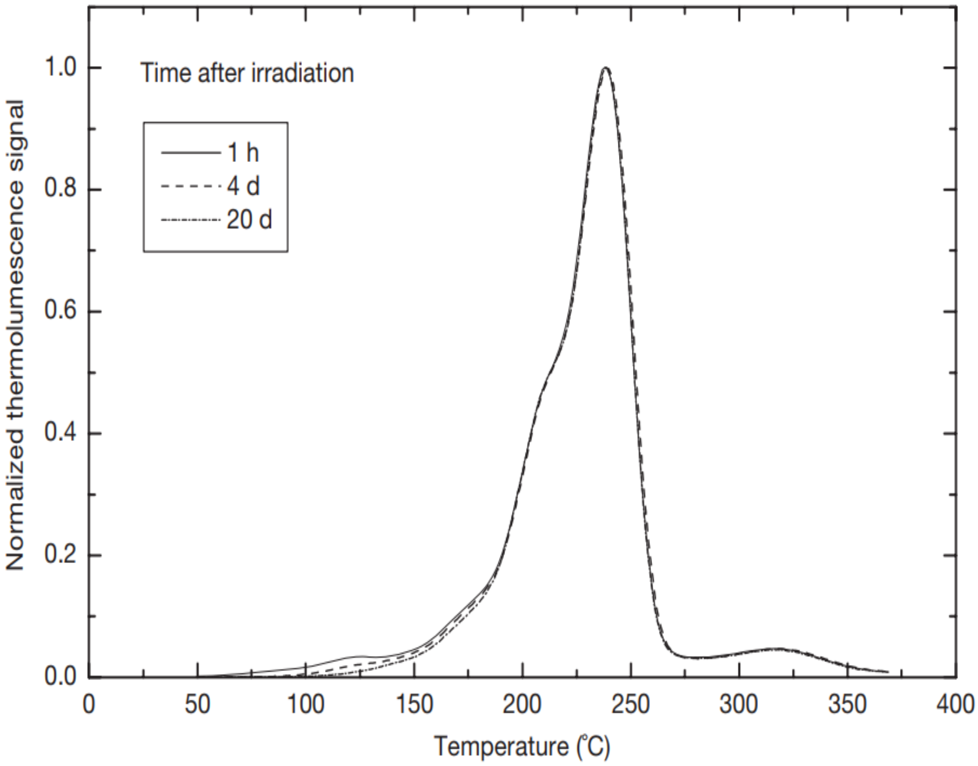


Figure 1.4 Thermoluminescence glow curve example [6]

1.5.2. Archeology

The thermal luminescence technique aids in determining the ages of archaeological pottery finds and provides a quick and accurate method of detecting artefacts.

When the pottery specimen is fired, the 'TL clock' begins. Whatever has been stored in TL over geological time due to internal and external irradiation is thought to be erased during firing, which takes place at temperatures of around 1000°C. Thus, the age is determined by measuring the accumulated TL since the sample release represents the archaeological age from the historian's perspective.

The samples' TL is primarily due to some TL-sensitive mineral elements in the sample's main clay matrix. Internal radiation results from the radioactive emission of U, Th, and K contents in the clay formation, as well as external radiation from the ocean's radioactive contents and the cosmic background at the burial site. The total radiation rate is usually less than 1 rad per year, with the intrinsic component accounting for most of it. Soil irradiation accounts for about 20% of the total, with cosmic rays accounting for a small percentage. If the total radiation rate (rad/yr) of the sample can be estimated and the accumulated TL in the sample has been measured and expressed in terms of absorbed dose reads) by appropriate calibration techniques, the archaeological age can be calculated by dividing the former by the latter. Variability in TL response to alpha, beta, and gamma radiation components, thermal and non-thermal fading properties, nonlinear TL dose accumulation behavior, heterogeneous distribution of minerals in the clay matrix, presence of a heterogeneous mixture of minerals contributing to TL, and so on. There are also practical challenges in measuring the typical accumulated TL signal, which is typically low and must be distinguished from competing IR and spurious signals. [7]

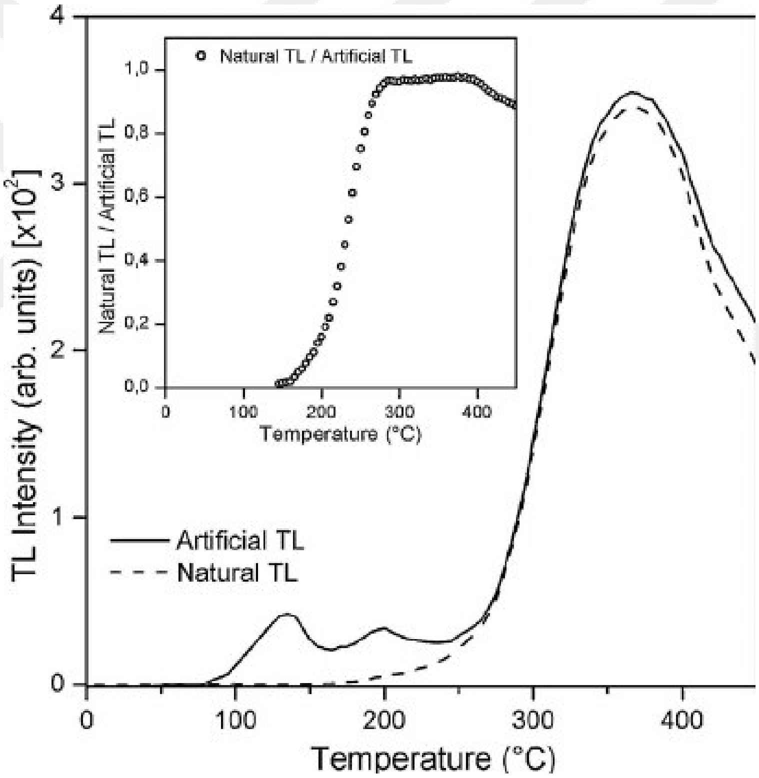


Figure 1.5 TL glow curves of natural sample and sample after gamma-ray irradiation. In inset the Plateau Test, ratio of the natural TL intensity and artificial TL. [7]

1.5.3. Biology and Biochemistry

Attempts were successful in studying hydroxychloroquine, amino acids, urea, nucleic acids, proteins, plant leaves, algae, and bacteria: TL results could indicate a weaker stability of orthoformates of benzoic acids. [8] [9]

1.5.4. Forensic Sciences

Forensic science's primary goal is to develop and standardize methods for comparing evidence with similar materials of known origin, which are always available in small quantities and must be analyzed non-destructively for evidentiary purposes. It is possible to use TL as an attractive technique in certain materials frequently used in criminal cases, such as glass or soil. [10] [11]

1.5.5. Geology

Fumed quartz crystals collected from a non-radioactive intrusive layer between radioactive veins in the sediments have been successfully used to estimate the age of radioactive mineralization of the sediment [12]. Studies in limestone sediment formation have found functional links between transient liquid (TL) and tectonic/magmatic events. The use of TL to date small rocky ocean sediments has recently been investigated. While the TL dissolution equation is used, it has been successfully integrated over the temporal temperature conditions achieved during magma cooling in various limestone and basalt regions of northern Arizona to determine magma temperatures [13].

1.5.6. Quality Control in Industry

Glass, ceramic, and semiconductor products can benefit from TL methods for quality control. In the case of textile fibers, the low-temperature TL glow curve changes can be correlated with structural differences and/or chemical tracer impurities. On the other hand, the industries have yet to pay attention to these issues. [14]

CHAPTER II

TL GLOW CURVES AND KINETIC ORDERS

2.1. TL Glow Curves

The glow curve depicts the relationship between the temperature of the sample used to release the trapped electrons and the intensity of the TL emitted after these electrons' recombination in the centers. [2], [15]. There have been several researches on borate glass to analyses its glow curve using different compositions (dopants) [16], [17], which have been conducted on different types of dopants. According to Chen 1969 [18], [19], the three-parameter model (Peak shape method) is among the best-developed methods. In accordance with the Peak Shape Method, TL may be defined by three parameters: activation energy (E), order of kinetics (b), and frequency factor (s). Several different approaches have been devised to calculate trap parameters from the TL glow curve. Peak form methods, starting rise methods, varied heating rates methods, and curve-fitting are some of the approaches that are often employed in the analysis [20].

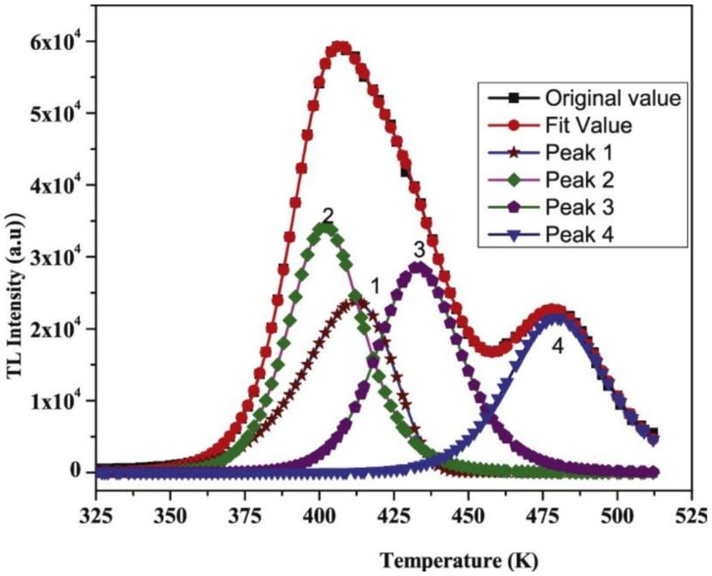


Figure 2.1 TL glow curves of natural sample of γ-exposed Eu3+ doped K3Y(PO4)2 nanophosphors. [7]

2.2. Kinetic Orders

The thermoluminescence glow peaks are usually calculated for a simple model (the model with one recombination center) using a general approach. In other cases, when the doses received by the studied sample are dispensed, an adjustments in the position and shape of the glow peaks is spotted, so these glow peaks are fitted to the general kinematic model and calculate the three kinetic parameters (Trap Depth E, frequency factor s, and the order of kinetics b) by finding the best fit. [18]

The importance of these kinetic parameters comes from the fact that they give the traps' evident characteristics; for example, if the TL fades at a low temperature, this means that the depth of the studied trap is relatively low. Or if the frequency factor produced is high, this means that there is a fading of TL. The kinetic parameters also give information on whether the charge carriers (either electrons or holes) will be trapped again after reheating the sample one more time or not. [19]

2.2.1. First order Kinetics (Randall and Wilkins model)

To study the glow curve, Randall and Wilkins used mathematical representation, as their mathematical analysis was focused on the energy band model known as first-order kinetics. [20]

In the figure (the simple model), consisting of the conduction band CB, the valence band VB, and between them are two levels of unstable states (localized levels), the first acts as an electron trap T and the second acts as a recombination center R; where E represents the energy needed to release the trapped electron and also expresses the depth of the trap [20]

If the temperature is kept constant, and we consider that the concentration of trapped electrons in the T trap is n, then this concentration decreases with time, and thus we can write the equation:

$$\frac{dt}{n} = -pn \tag{2.1}$$

And by integrating this equation we obtain:

$$\int_{n_0}^{n} \frac{dn}{n} = -\int_{t_0}^{t} p.dt \tag{2.2}$$

And thus:

$$n = n_0 \, exp \, exp \left[-s \, exp \, exp \left(-\frac{E}{kT} \right) . t \right] \qquad (2.3)$$

Where;

n_0 is the concentration of the trapped electron at T level at the initial time t_0, with the following:

- The irradiation of TL of the sample occurs at a sufficient low temperature ensures that no re-released electrons from the trap T, and that the released electrons are not trapped again.
- The TL efficiency of the recombination center R is temperature-independent.
- At the conduction band CB; the lifetime of the electrons is short.
- All released electrons from the trap T recombine at the luminescence center.

Based on previous assumptions, at constant temperature the intensity of thermoluminescence I is directly proportional to the re-trapping rate dn/dt, thus we can write:

$$I = -c \left(\frac{dn}{dt} \right) = c \, p \, n \qquad (2.4)$$

This equation demonstrates an exponential decay of the phosphorescence.

Relaying on the equation 2.5, the intensity of the thermoluminescence can be written as follow:

$$I(t) = n_0 \, s \, exp \, exp \left(-\frac{E}{kT} \right) \, exp \, exp \left[-s \, t \, exp \, exp \left(-\frac{E}{kT} \right) \right] \qquad (2.5)$$

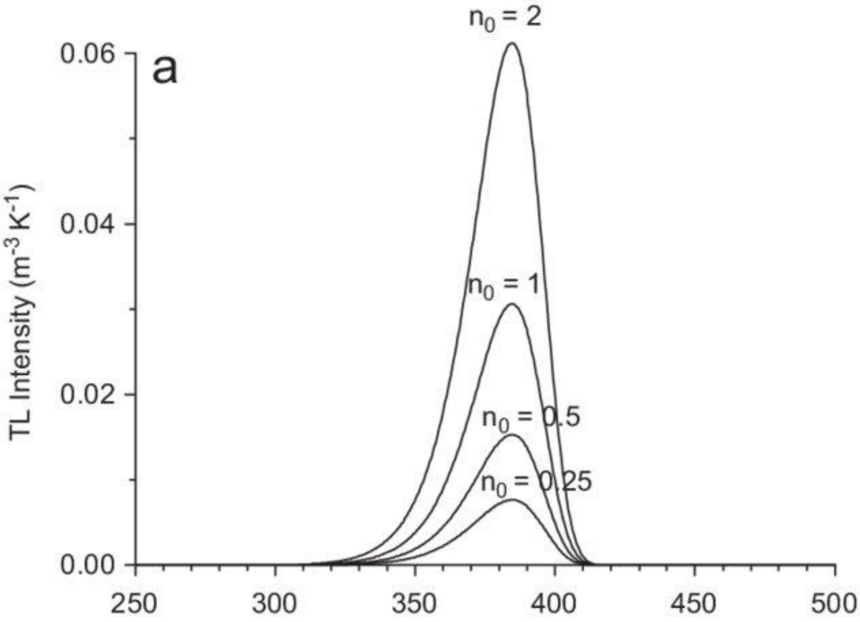


Figure 2.2 (a) Properties of the Randall-Wilkins first-order TL equation, showing: variation with the concentration of trapped charge carriers after irradiation n 0

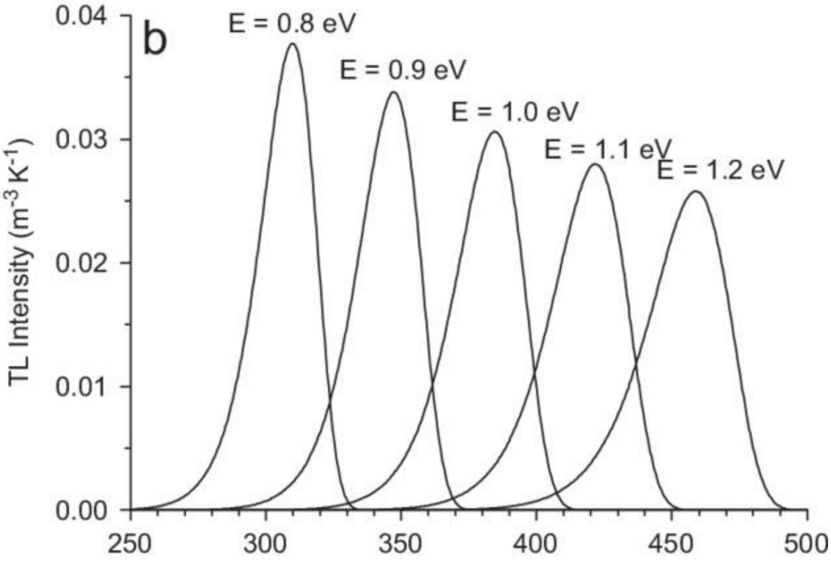


Figure 2.2 (b) the variation with the activation energy E

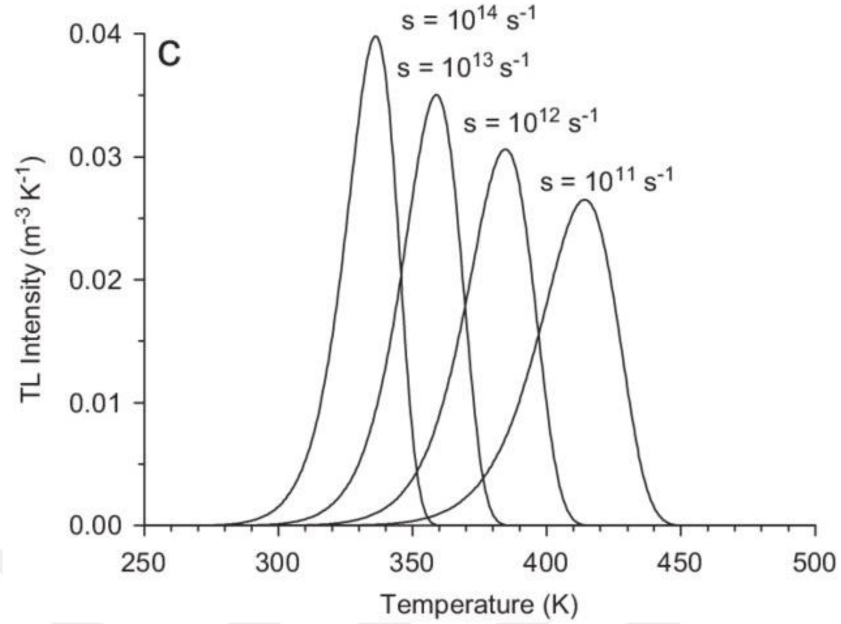


Figure 2.2 (c) the variation with the escape frequency s; Parameter values: n 0 =1 m −3 ; E =1 eV; s =1×10 12 s −1 , of which one parameter is varied while the others are kept constant. The heating rate = 1 K s −1 for all curves. Note that the peak maximum position does not vary with n 0. [5]

2.2.2. Second Order Kinetics (Garlick and Gibson model)

In their research on phosphorescence in 1948, Garlick and Gibson considered the situation in which a free charge carrier has a high probability of being trapped or recombining within a recombination center. It is necessary to use the term second order kinetics to describe a situation in which re-trapping is occurring.

They made the assumption that an escaping electron from a trap has an equal chance of being re-trapped or of recombining with a hole in a recombination center, whichever occurs first.

$$I(T) = \frac{n_0 s \exp(-E/kT)}{\left[1 + \frac{s}{\beta}\int_{T_0}^{T} \exp(-E/kT')\, dT'\right]^2} \tag{2.6}$$

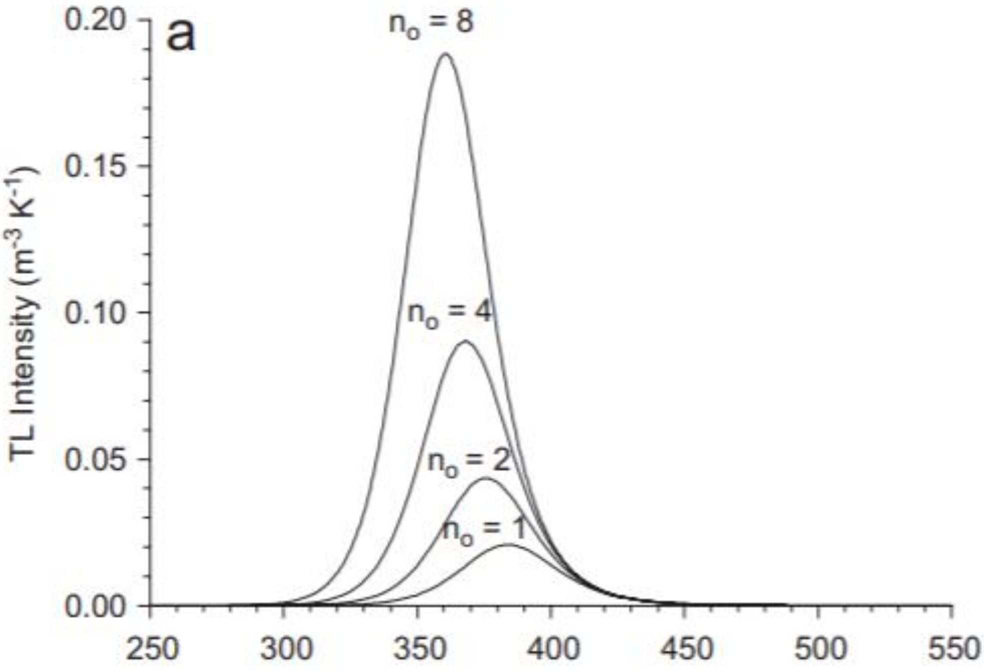


Figure 2.3 (a) variation with the concentration of trapped charge carriers after irradiation n0.

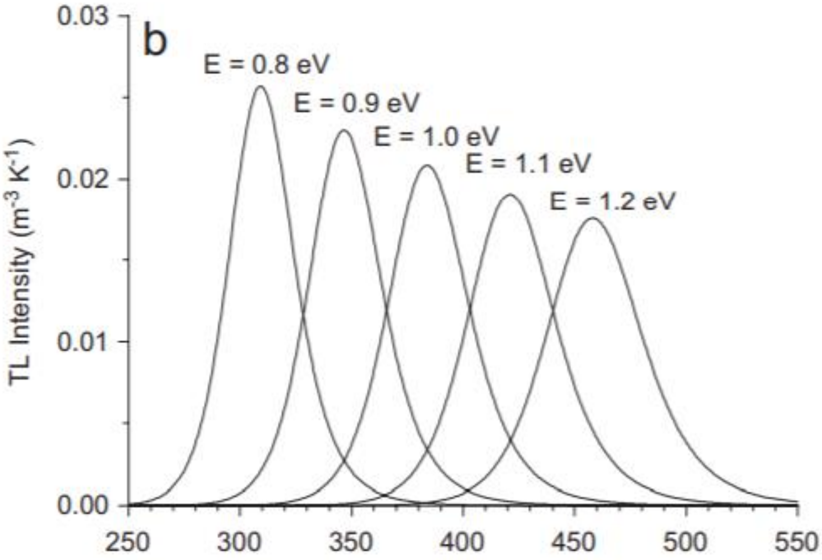


Figure 2.3 (b) the variation with the activation energy E.

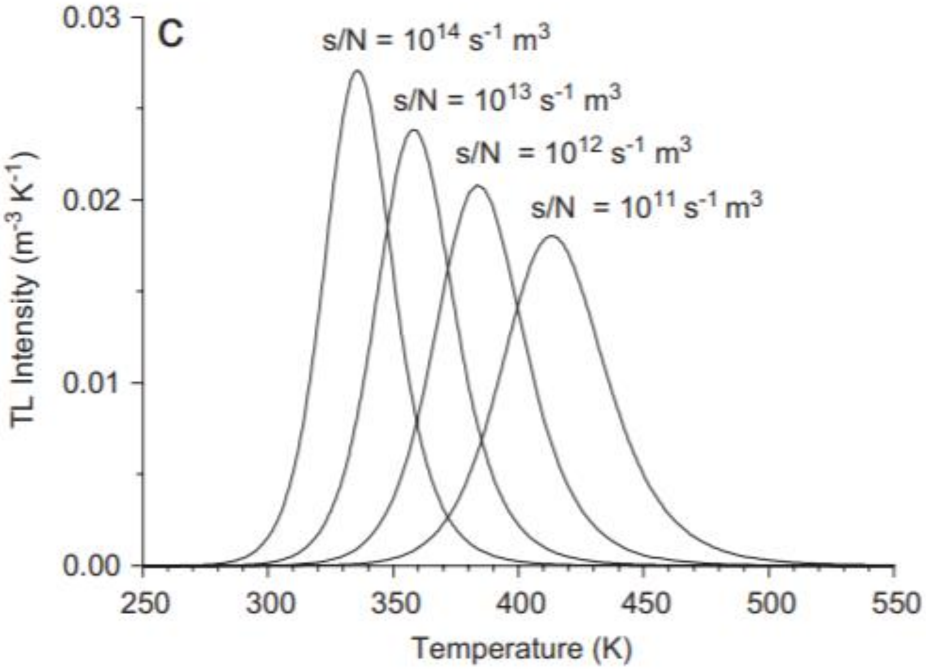


Figure 2.3 (c) the variation with s/N. Parameter values: n0 = 1 m−3; E = 1 eV; s/N = 1 × 1012 s−1 m3, of which one parameter is varied while the others are kept constant. The heating rate = 1Ks−1 for all curves. [5]

2.2.3. General – Order Kinetic

When using a linear heating rate function, several basic features may be utilized to differentiate between first and second-order glow peaks. According to Mary and Partridge, a so-called General Order Kinetic was devised in 1964 to overcome numerous examples and research, and it is expressing the intensity of TL in the following equation, as shown. [20]

$$I = \frac{s^{''}}{\beta} n_0 \exp\left\{-\frac{E}{kT}\right\}\left[1 + (b-1)\frac{s''}{\beta} \times \int_{T_0}^{T} \exp\left\{-\frac{E}{kT}\right\} dT'\right]^{\frac{-b}{b-1}} \qquad (2.7)$$

The previous equation is a general equation and can be used for the first order by making $b = 1$ and for the second order by $b = 2$. This is shown in the figure below.

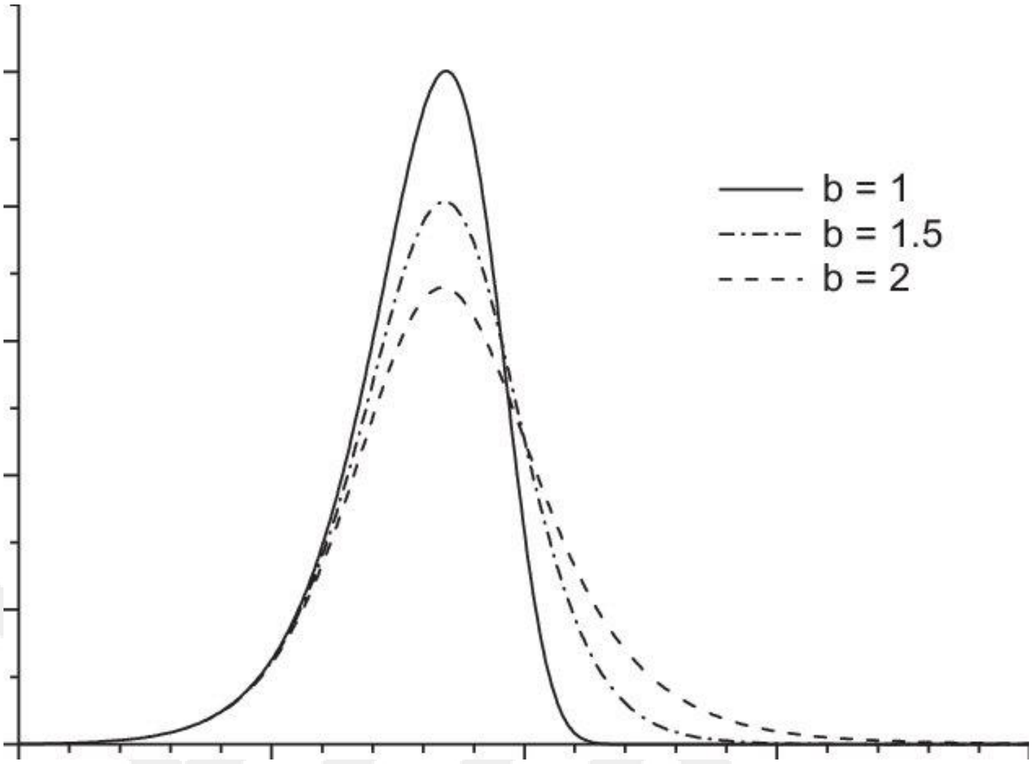


Figure 2.4 Comparison of first-order (b = 1), second-order (b = 2) and intermediate-order (b = 1.5) TL glow peaks. with E = 1 eV, s = 1 × 10 12 s −1 and = 1 K s −1. The peaks are normalized to the first-order glow peak. Note that all peaks coincide in the initial rising part of the curve. [5]

CHAPTER III
MATERIAL USED IN THE EXPERIMENT

3.1. Polycarboxylate Cement

Polycarboxylate cement is one of the most important materials used in dental treatments, and the powder consists of 72% zinc oxide, 7% magnesium oxide, other oxides like aluminum oxide and bismuth. Sometimes the powder may contain stannous fluoride to increase strength, modifies setting time and imparts anticariogenic properties. The aqueous solution added to the powder consists of polyacrylic acid (32-48%) or other carboxylic acids (itaconic, maleic, tricarballylic acids) in common with the acrylic acid [21].

These materials (liquid and powder) are biocompatible. And this powder substance (polycarboxylate zinc) was chosen for this experiment as it is used safely in humans. The luting agents for permanent fixation must meet several requirements for this purpose. Biocompatibility is an essential advantage emphasized by ANSI / ADA for the biological evaluation of dental materials used in humans [22].

Figure 3.1 Adhesor Carbofine Polycarboxylate Cement. [22]

3.1.1. Biologic Properties

Polycarboxylate cement is considered to have excellent biological properties, and it shows proper adhesion to the tooth structure. Polycarboxylate cement consists of zinc oxide powder and a liquid from polyacrylic acid and a co-polymer for this acid. When mixing the liquid with the powder, the cement bonds against the tooth structure via a chemical bond between this fluid and the calcium in the enamel and dentin. [3]

3.1.2. Chemical Properties

Zinc Polycarboxylate has a pH of 1.7, which is easily neutralized by the addition of powder that raises the pH above Zinc Phosphate cement. An initial low pH does not bother the Pulp with Zinc Polycarboxylate cement. Polyacrylic acid molecules' enormous size, which restricts their diffusion through dentinal tubules, may be to blame for this. Similar to ZOE cement, it is very biocompatible with the pulp.

The fluoride leakage from this cement is only 15 to 20 percent of that from glass ionomer cement. Stronger than Zinc Phosphate Cement in Compressive Strength, but weaker than Zinc Phosphate Cement in Tensile Strength (Slightly higher than Zinc phosphate cement). [23]

Solubility: In water, the cement's solubility is just 0.6 percent, but it's much higher in acids. Zinc phosphate cement is a good comparison.

Zinc Phosphate cement's viscosity is somewhat greater than that of this kind of cement. When placed on the tooth, the cement looks to be heavy, yet it spreads easily.

Thermal insulation is good in a 1.5mm thickness of the mixed powder liquid. It's not as good as the other cement in insulating heat as it should be at a thickness of less than 1.5 mm. Because of the significant amount of unreacted zinc oxide, it is opaque. [23]

3.1.3. Physical Properties

The physical properties of polycarboxylate cement are greatly affected by the mixing ratio of the components (mixing the liquid with zinc oxide powder during dental fixation). The compressive strength is approximately 100 MPa, while the elastic standards are from 5 to about 12 GPa, which is less than the elastic means found in dentin. After the acid is distilled out, some formulations are mixed with water and the powder is allowed to freeze, they may have an expanded effect. However, to a gold, the skin and dentin (the underlying substance inorganic material) have an adhesive strength that is less than poracin. Lactose has never been found to be irritating to the tooth pulp. Many experts suggest that higher solubility, in the biological media, as opposed to zinc phosphate, does not pose a significant clinical issues Cements of this type are easily set, but their crown duration of working time is short, and the placement of the seal must be made quickly. Cement appears too thick to pour when mixed in the proper proportions, and additional liquid seems to want to flow, however, additional stirring will thin it down. The concrete should be supported since it is unstable and increase in viscosity while adding more liquid would compromise the material physical properties; additionally, it will lower the pressure resistance. [21].

3.2. X-ray Diffraction Analysis of polycarboxylate cement

The most challenging problem in the design and manipulation of dental materials is creating materials with complicated physical and functional features that match those of real tissues. The ultimate objective of restorative dentistry is to design a replacement material that either duplicates the qualities and performance of natural tissue or is ultimately assimilated and substituted by new tissue that is equal in composition and function [24].

The XRD study of the zinc-polycarboxylate-powder revealed the presence of two crystalline phases, ZnO and SnO_2. Because the level of tin oxide varies depending on the commercial cement first used, it is found in only trace amounts in certain cements while it is absent from others. Due to the amorphous nature of this molecule, no reflections of zinc polycarboxylate were seen. The cement must thus be a combination of unreacted oxides ZnO (primarily) and SnO_2 and the amorphous zinc polycarboxylate matrix formed by the setting reaction of these oxides in the presence polyacrylic acid and water. In this case, the findings are consistent with the core link structure postulated for this kind of cement [25].

a

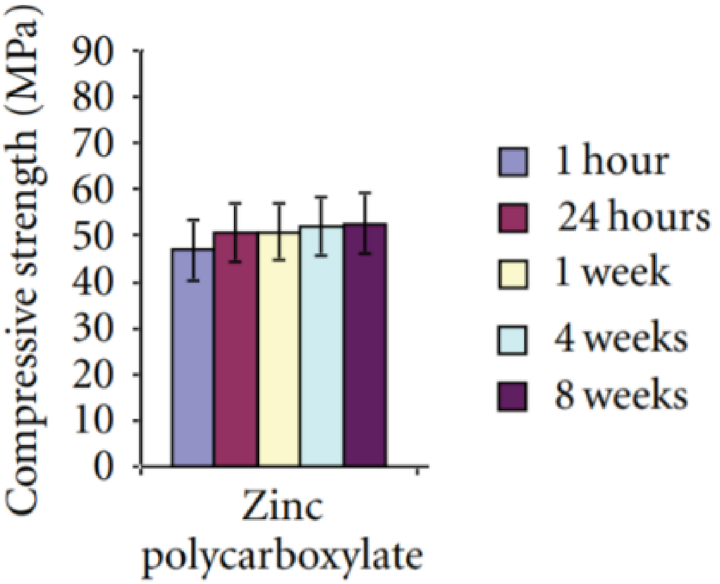


Figure 3.2 (a) Histogram showing the mean compressive strength of zinc polycarboxylate cement [26]

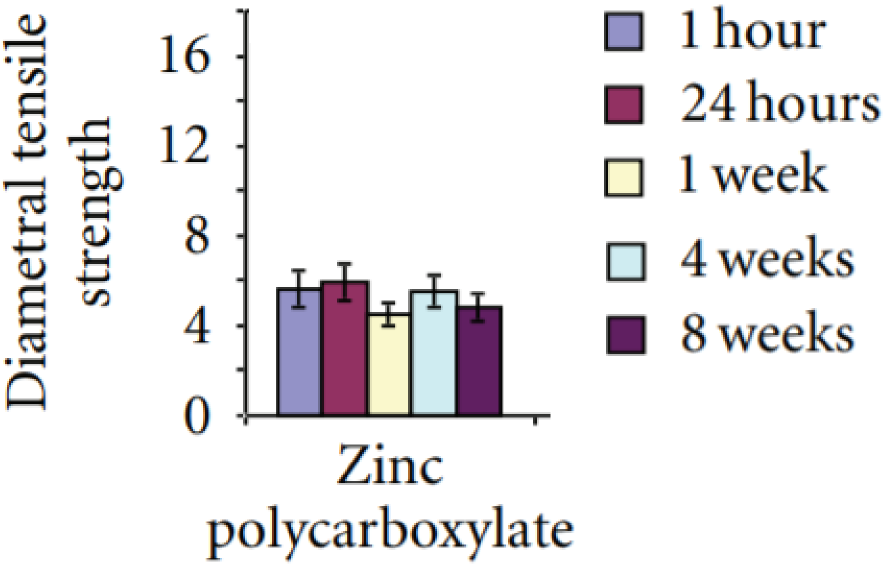


Figure 3.2 (b) histogram showing the mean diametral strength of zinc polycarboxylate cement [26]

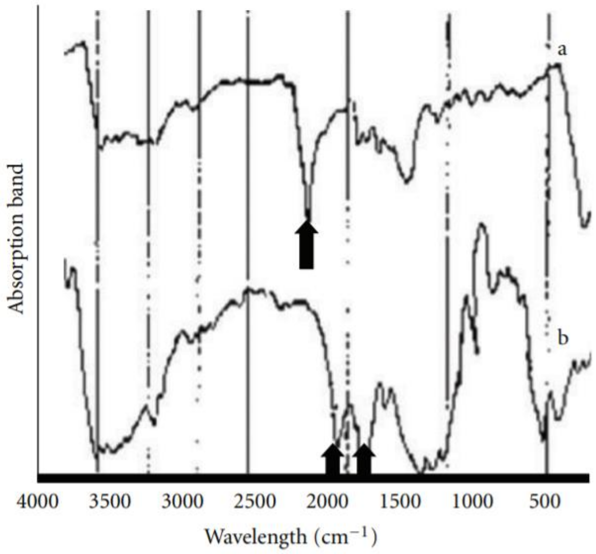


Figure 3.3 IR spectra of polymeric acids (modified polyacrylic acid, modified polyalkenoic acid, 35% w/w aqueous solution of PMVE-Ma (a) showed the absorption bands of carboxylic group (C=O) between 1635 to 1640 cm−1 cm (arrows). IR spectra of set cements (b) showed the absorption bands between 1558 and 1401 cm−1 indicating the formation of carboxylic salts (arrows). [26]

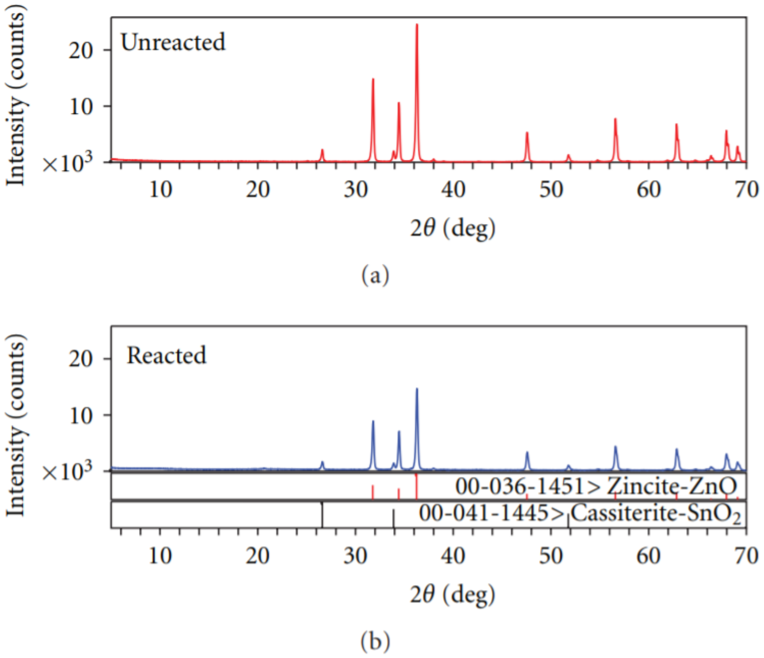


Figure 3.4 (a) X-ray diffraction pattern of unreacted zinc polycarboxylate powder, (b) X-ray diffraction pattern of set zinc polycarboxylate cement. [26]

3.3. Related Studies

Accidental dose measurement using luminescence and dosimetry can calculate dose characteristics in the open air is an increasingly prevalent technique. Several kinds of porcelain materials used for dental restorations, including materials used in core constructions and materials used for the veneer layer (extruded porcelain), were investigated on the assumption that their attributes could be used in accidental radiation dosimetry. Dosing from a few mGy up to several tens of mGy. For most of the samples, a linear dose-response was observed. After being characterized as a major fading, the luminescence signals are only effective for short-term dose assessment. [27].

Researchers have been working on materials that can convert energy radiations (X-rays, -rays) into visible light, which is readily observable with ordinary detectors since Rontgen discovered X-rays and the subsequent usage of the first dosimeter [28]. There is now a great deal of interest in the synthesis and processing of sophisticated materials. The sol-gel technique is a low-temperature approach for creating ceramics and glasses that are purer and more homogeneous than standard high-temperature procedures. The sol-gel process has been used to manufacture a broad range of compositions (mostly oxides) in a variety of forms, including powders, fibers, coatings, thin films, monoliths and composites, and porous membranes. [29]. The hydrolysis and condensation of alkoxides are involved in the zirconia sol-gel process. By carefully regulating the synthesis conditions, the sol morphology may be guided toward branching polymeric structures. The management of relative rates of hydrolysis and polycondensation, as well as the processes of these reactions, are the major instruments for this difference [29]. This novel approach of preparing nano phosphors has lately been intensively researched for the manufacturing of high-efficiency luminous materials. It is vital to maintain a high degree of crystallization throughout the manufacture of ZrO_2, and its probable relationship with the concentration of faults should be thoroughly investigated. For this objective, new approaches for producing well-defined nano-powders have been developed. A recent investigation on nanocrystalline ZrO_2 produced from amorphous zirconia showed a number of different nanocrystalline processing properties. [29]

Thermoluminescence (TL) is a popular method for measuring ionizing radiation dose. TL is a technique in which the stored energy by a phosphor that has previously been subjected to ionizing radiation is released as light during the heating of the material. The light output of a phosphor is proportional to the dose of irradiation delivered to it. Numerous TL materials have been investigated in terms of preparation techniques and characteristics. Metal oxides with suitable activators have been identified to produce a potential TL phosphors class [29]. The TL dosimetry (TLD) approach has the benefit of enabling dosimeters to be put in outdoor solar radiation monitoring stations without the need for extra monitoring or logistical concerns. For this job, ZrO_2 is proposed as a good TLD material. [29]

Figure 3.5 depicts the thermoluminescence (TL) glow curve of zirconium oxide generated by the sol-gel technique, which exhibits a primary maximum at various places depending on the source irradiation. As a result, zirconium oxide beta irradiated (dashed line (---)) produces the maximum at about 180 ℃. Zirconium oxide subjected to an X-ray diagnostic beam (solid line (___)) exhibits two peaks, one at 160 ℃ and the other at 260 ℃, with a maximum at about 260 ℃. The second peak is the most visible in the picture, with an amplitude almost 1.5 times the first. [29]

The shape of the glow curve remains nearly constant for exposures ranging from 2 to $100\ Gy$, but the relative heights of the two peaks vary as a function of beta-absorbed dose. Zirconium oxide gamma-irradiated (dashed and dotted line (-..-..-..) shows a TL glow curve that is very similar to that obtained by $X-ray$ exposure. When comparing light output total, the luminous ceramic gamma irradiation exhibits around half the efficiency of the X-ray exposed. WHEN BETA IRRADIATED, the TL sensitivity of zirconium oxide is lower than when ZrO_2 X-ray photon beams are irradiated. The breadth of the significant peak shows that the activation energy of the traps is distributed in a continuous manner. [29]

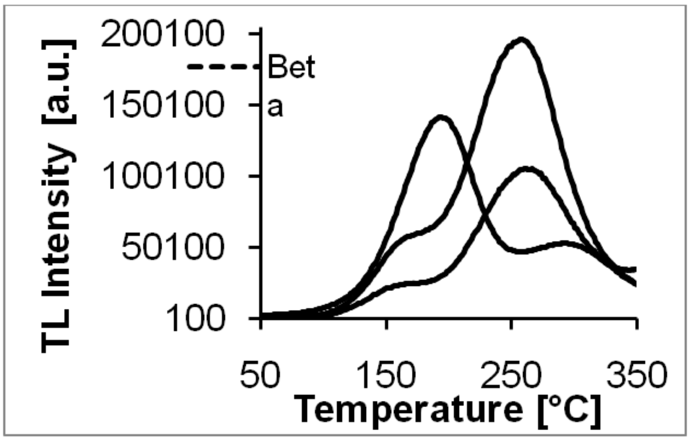


Figure 3.5 (TL) glow curve of zirconium oxide obtained by sol-gel method. [30]

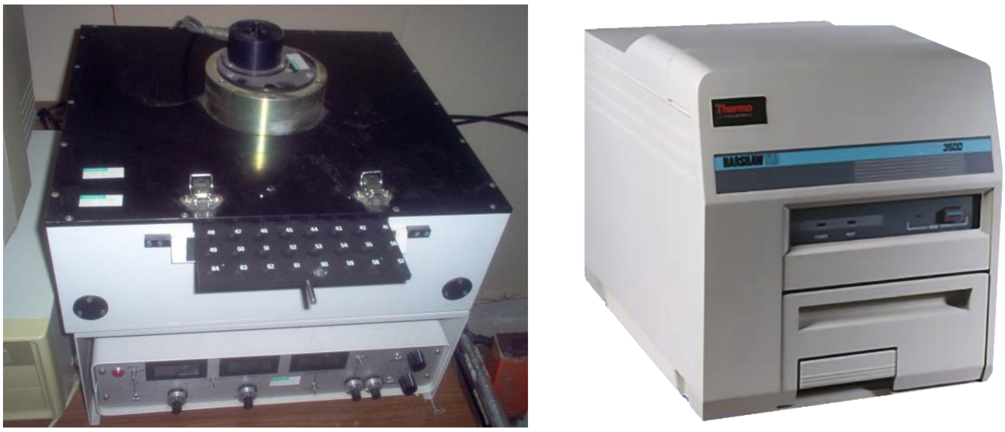
CHAPTER IV
EXPERIMENTAL PREPARATIONS

4.1. Experimental Preparations

Before starting the experiment, ten samples are weighed, each sample weighing $10\,mg$, are measured. Samples are irradiated in different doses and for different durations, according to the study's nature to be performed.

Samples are irradiated using a 90Sr-90Y beta source that delivers 0.04 Gy/Sec, with a recommended lifetime in a laboratory of about 15 years. Its particle range in the air is only about 10 meters. Figure 4.1 (a) shows the irradiator includes 90Sr-90Y beta source to irradiate the samples. This radioactive source is installed on a 9010 optical dating system linked with a computer to control the amount of doses and the irradiation duration. [31]

Figure 4.1 (b) shows the Harshaw TLD System 3500 to read the irradiated samples.



A b

Figure 4.1 (a) the irradiator includes 90Sr-90Y beta source, (b) Harshaw TLD System 3500. [31]

After the sample is irradiated, it is read using a Harshaw TLD System 3500 linked to a computer to set the reading settings such as temperature control, heating rate, and other options. The results are extracted from the computer, analyzed, and charts using Origin Pro Lab application.

Figure 4.2 depicts a conventional TL dosimetry reader (TLD). The irradiation dosimeter is fixed on a tray housed inside a readout chamber to allow for easy reading. The dosimeter is heated at two distinct temperatures: the first is used for preheating to remove noisy peaks, and the second is used to heat the dosimeter once it has been preheated. The second temperature is the readout temperature, which is used to thermally excite the sample and gather data from the sample. The absorbed dosage is proportional to the output current obtained from the photomultiplier tube, and the output current is measured in microamps. [32] [33]

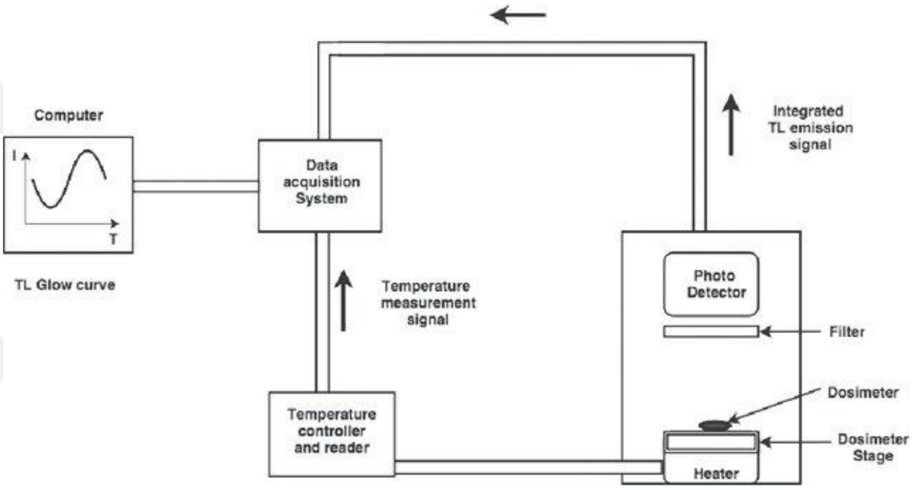


Figure 4.2 Schematic of TLD reader setup [33]

CHAPTER V

EXPERIMENTAL RESULTS

This experiment aims to study the dosimetric properties of polycarboxylate cement used in dental treatment. Three experimental studies were conducted: the dose-response, the heating rate, and the reproducibility. These studies depended on the TL glow curves resulting from reading the sample in different doses and heating rates.

5.1. Dose Response Study

In this experiment, the effect of the difference in the value of the radiation dose to which the sample is exposed to the thermoluminescence intensity at a constant heating rate is measured.

In this experiment, the sample is irradiated with different radiation dose values (ranging from 2.4 Gy to 4.6 KGy), and the sample is read at a constant heating rate of $1\ ℃/s$ for each of the radiation dose values.

The results indicate that TL peaks started to be recognized at $65\ ℃$ for a dose range of up to $144\ Gy$ and that there are four peaks in this range located at 65, 123, 204, and 340℃, as shown in Figure 5.1.

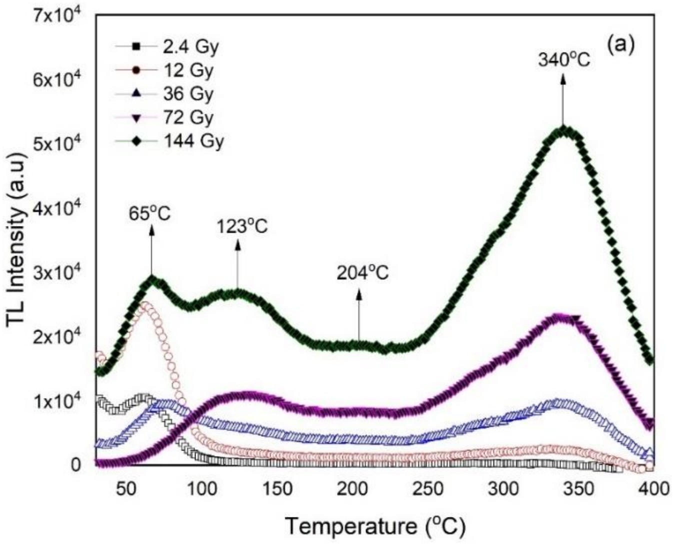


Figure 5.1 TL glow curve of polycarboxylate cement used in dental treatment, applied dose level 2.4 $Gy\ to$ 144 Gy on a constant heating rate of $\beta = 1\ ℃/s$

With increasing dose level, the results show that at lower temperatures (<100°C), the peaks disappear, and there are no new peaks, as shown in figure 5.2.

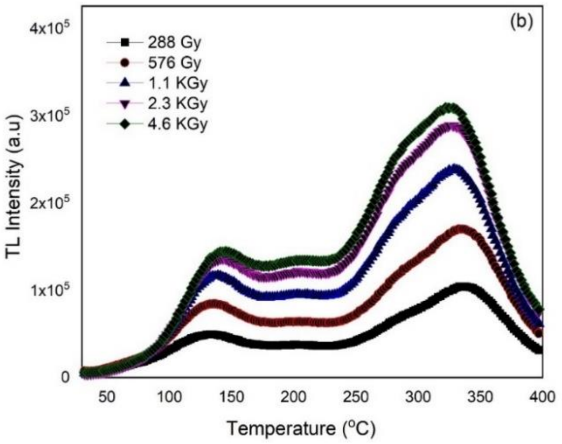


Figure 5.2 TL glow curve of polycarboxylate cement used in dental treatment, applied dose level 288 $Gy\ to$ 4.6 KGy on a constant heating rate of $\beta = 1\ ℃/s$

The effect of the applied dose on the TL peak temperature is depicted as shown in figure 5.3. The figure demonstrates that the locations of the thermoluminescence peaks are fixed at different temperatures depending on the applied dose, and there are no significant deviations in the locations of the peaks either at high temperatures or high values of radiation doses.

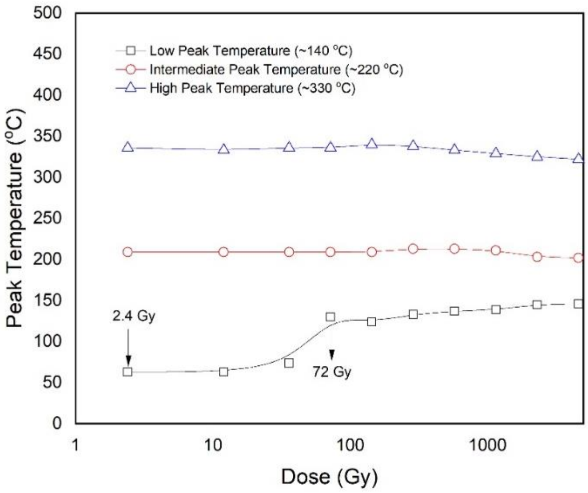
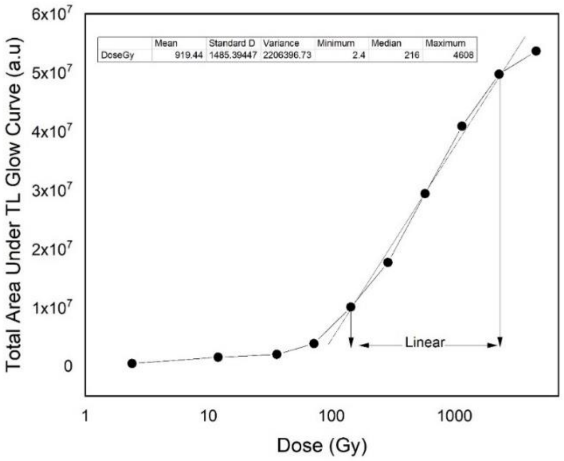


Figure 5.3 The effect of applied dose on TL peak temperature for peaks located around $140, 220, and$ 330 ℃.

The relationship between the dose value and the area under the TL glow curve is linear between 144 Gy and 2.3 KGy, as shown in the figure 5.4.



	Mean	Standard D	Variance	Minimum	Median	Maximum
DoseGy	919.44	1485.39447	2206396.73	2.4	216	4608

Figure 5.4 Variations of total area under TL glow curve as a function of applied dose.

The thermoluminescence peak intensity is a linear function of the applied dose between 144 Gy and 2.3 KGy as shown in figure 5.5.

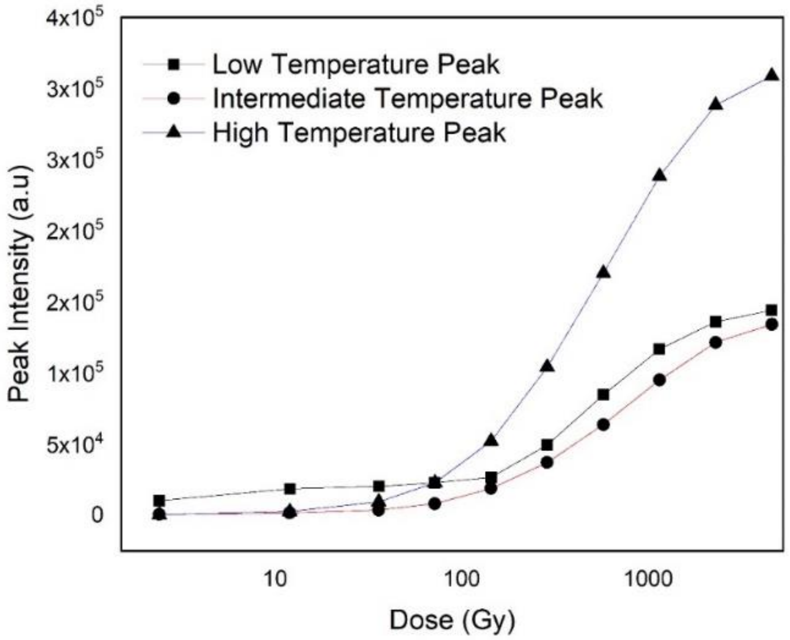


Figure 5.5 TL peak intensity as a function of dose, for peaks located around $140, 220, and\ 330$ ℃.

5.2.Heating Rate Study

The purpose of this experiment was to determine the effect of heating rate on thermoluminescence glow curves.

This process begins by irradiating the sample at 36 Gy, then reading at five different heating rates starting from 1 ℃/s to 5 ℃/s.

The increase in the heating rate does not affect the shape of the glow curve, as the figure 5.6 shows that there are no new or extra peaks.

In general, increasing the heating rate leads to an upward shift in the glow curve to high temperatures and a decrease in the intensity of thermoluminescence. And that is because of temperature lag due to the thermal contact of the heater and the sample.

Thus, the sample thickness has an important influence on temperature gradient and temperature lag especially at higher heating rates.

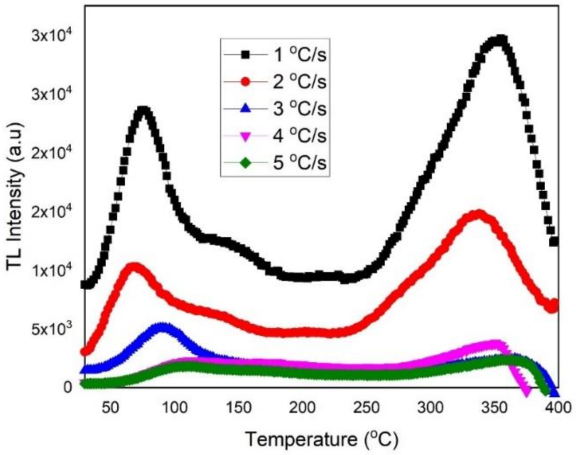


Figure 5.6 Variation of TL glow curve as a function of heating rate.

With the increase in the heating rate, the results indicate that the TL intensity decreases significantly for the peaks recognized at lower temperatures; in other words, as shown in the figure 5.7, the peak intensity at the temperature of 100 ℃ decreases considerably with the increase in the heating rate.

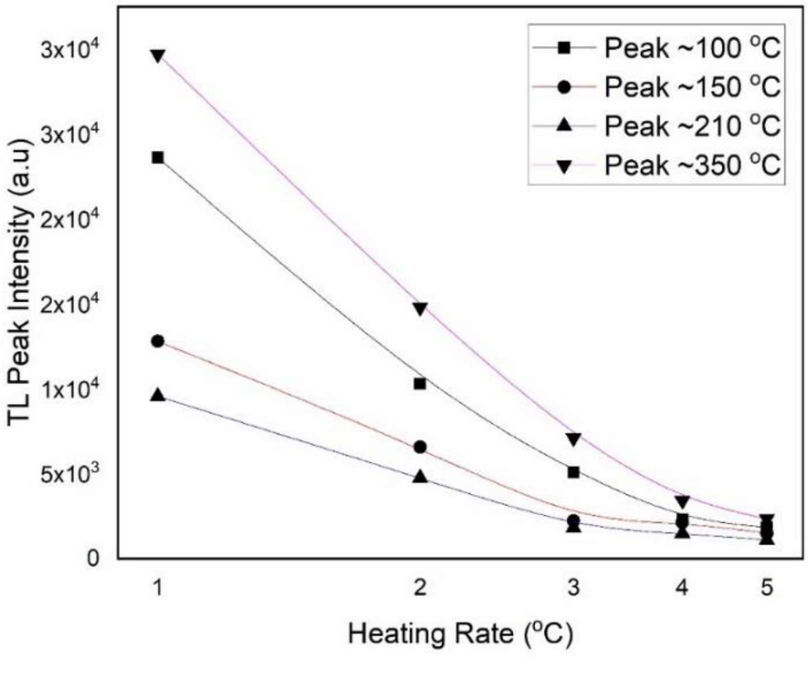


Figure 5.7 The effect of different heating rates on TL peak intensities, for peaks located around $100, 150, 210, and\ 350$ ℃.

Table 5.1 shows the TL intensities of four peaks at different temperatures was read at different heating rates ranging from 1 $℃/s$ to 5 $℃/s$.

Table 5.1 TL intensities of four peaks at different temperatures at different heating rates ranging from $\mathbf{1}$ $℃/\boldsymbol{s}$ to $\mathbf{5}$ $℃/\boldsymbol{s}$.

Heating Rate $℃/\boldsymbol{Sec}$	Peak 1 at $\mathbf{100}$ ℃	Peak 2 at $\mathbf{150}$ ℃	Peak 3 at $\mathbf{210}$ ℃	Peak 4 at $\mathbf{350}$ ℃
1	23620	12810	9564	29713
2	10301	6584	4777.5	14803
3	5095	2243	1830	7126
4	2308	2154	1489	3451
5	1856	1512	1110	2367

Increasing the heating rate, as previously stated, does not affect the peak or glow curve shape but instead causes the peaks to an upward shifting to higher temperatures. The figure 5.8 shows the effect of changing the heating rate on the temperature of the peaks. Through the table 5.2, we can detect the values of the upward shift of the peaks.

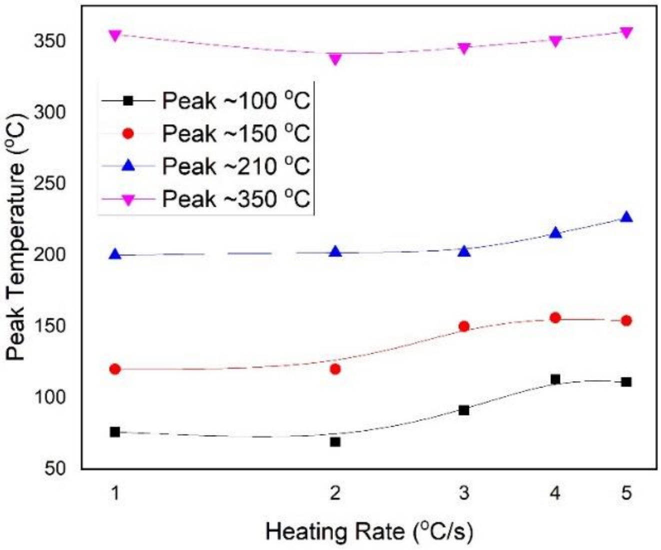


Figure 5.8 The effect of different heating rates on peak temperature, for peaks located around $100, 150, 210, and$ 350 ℃.

Table 5.2 TL peak temperatures for peaks located at temperatures $\mathbf{100}$, $\mathbf{150}$, $\mathbf{210}$, and $\mathbf{350}$ ℃.

Heating Rate ℃/$\boldsymbol{Sec}$	Peak 1 Temperature ℃	Peak 2 Temperature ℃	Peak 3 Temperature ℃	Peak 4 Temperature ℃
1	76	120	200	338
2	83	131	204	340
3	91	146	210	346
4	106	158	215	351
5	118	166	226	357

The experiment also showed that the area under the TL glow curve, which represents the relationship between the heating rate and the intensity of the glow curve, decreases as the heating rate increases and becoming nearly stable after $3\,℃/s$ and that is because the non-radiative recombination as shown in Figure 5.9.

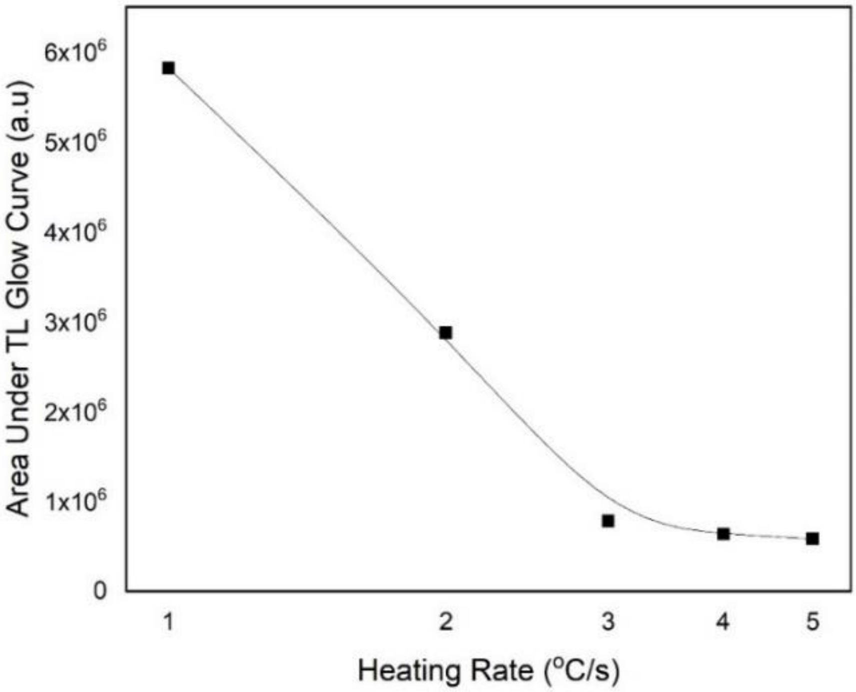


Figure 5.9 Total area under TL glow curve as a function of heating rate.

5.3. Reusability Study

One of the most important studies that have been examined during the experiment is reproducibility. The "cycle of measurement" is referred to as "reproducibility." In other words, it refers to a material's ability to produce thermoluminescence sequentially after many reads.

In this experiment, the sample was irradiated at $36\ Gy$ and read eight times in a row at a constant heating rate of $1\ ℃/s$.

Experience indicates no change in the glow curve shape, no new peaks recognized when reading the sample several times in a row at a constant heating rate, and there is no shift of the peaks at high temperatures as shown in figure 5.10.

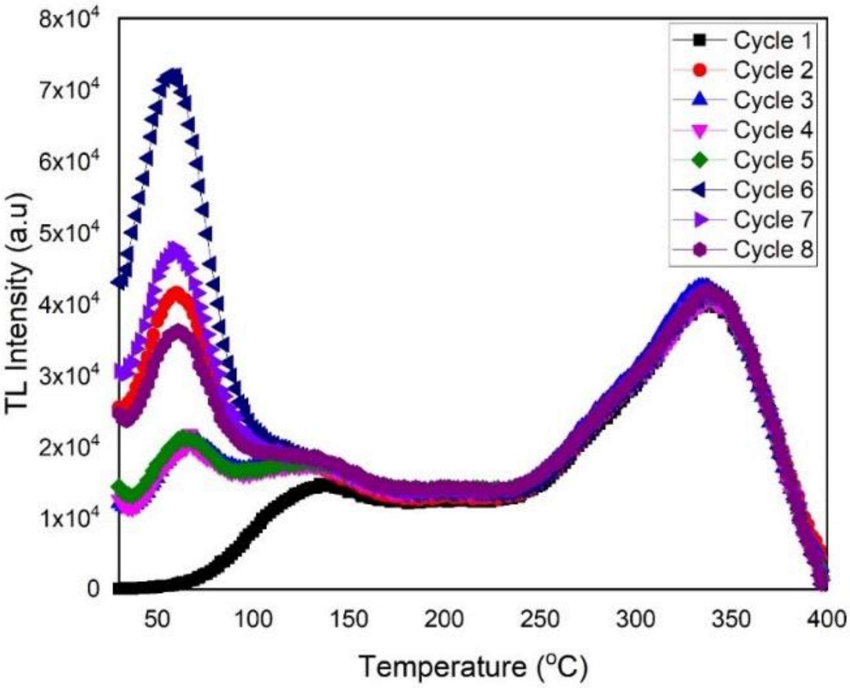


Figure 5.10 TL glow curve as a function of cycle of measurement.

Figure 5.11 shows that the intensity of the peaks at lower temperatures $\sim 65\ ℃$ is unstable compared to the intensity at higher temperatures (peaks at $200\ ℃$ and $355\ ℃$). Because: such emissions correspond to shallow traps in the sample where electrons are not stable where Shallow traps give rise to short-lived photoinduced absorption.

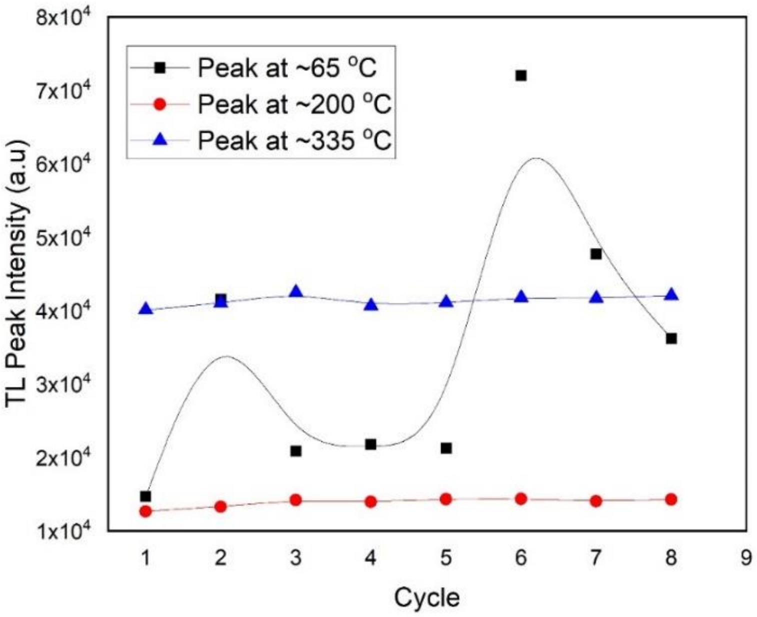


Figure 5.11 The effect of cycle of measurement on TL peak intensities, for peaks located around $65, 200, and\ 335$ ℃.

As previously mentioned, there is no shifting in position of peak temperatures. The figure 5.12 shows the effect of the cycle of measurement on peak temperatures for peaks located around 65 ℃, 200 ℃, and 335 ℃.

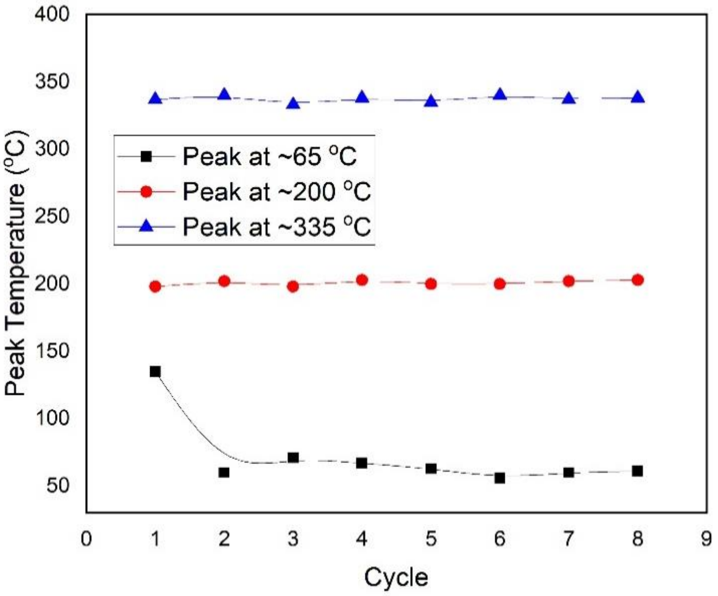


Figure 5.12 Peak temperature as a function of cycle of measurement, for peaks located around $65, 200, and\ 335$ ℃.

There is a recognized fluctuation in the area under the peaks for the low temperature peaks located between 30 *to* 150 ℃, with no changes mentioned for peaks of high temperatures located between 150 *to* 400 ℃ as shown in the figure 5.13.

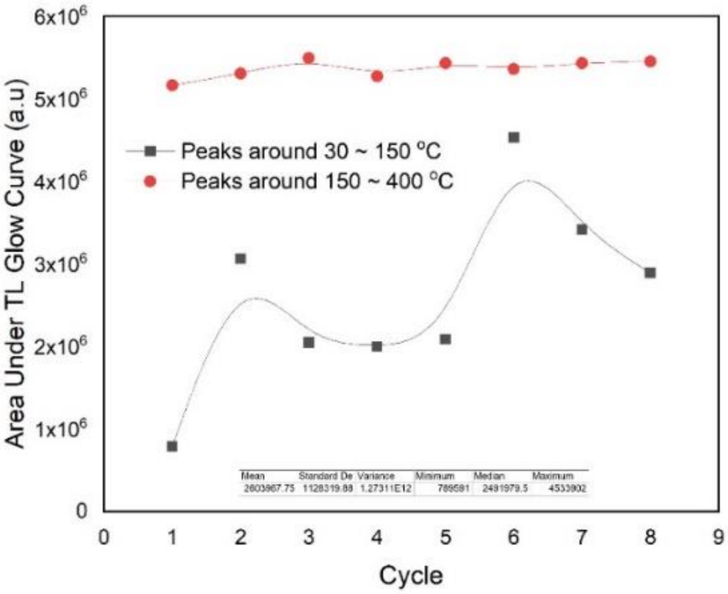


Figure 5.13 Variation of area under TL glow curve as a function of cycle of measurement.

CHAPTER VI
CONCLUSION

This research aims to study the dosimetric properties of polycarboxylate cement used in dental treatment.

Three experimental studies were conducted: the dose-response, the heating rate, and the reproducibility. These studies depended on the TL glow curves resulting from reading the sample in different doses and heating rates, as mentioned in chapter 5.

In the Dose-Response study, the effect of the difference in the value of the radiation dose to which the sample is exposed to the thermoluminescence intensity at a constant heating rate was measured. The results indicated that TL peaks were identified at approximately 65 ℃ for a dose range of up to $144\ Gy$ and that there are four peaks in this range located at 65, 123, 204, and $340\ Gy$. With increasing dose level ($288Gy$ up to $4.6KGy$), the results showed that at lower temperatures ($< 100°C$), the peaks disappear, and there are no new peaks. The locations of the thermoluminescence peaks were fixed at different temperatures depending on the applied dose. There were no significant deviations in the locations of the peaks either at high temperatures or high values of radiation doses. The area under the glow curve was linear between $144\ Gy$ and $2.3\ KGy$. The thermoluminescence intensity was a linear function of the applied dose between $144\ Gy$ and $2.3\ KGy$.

In the Heating Rate study, the effect of various heating rates on the TL glow curves was examined; the results indicated that the increase in the heating rate did not affect the shape of the glow curve by increasing the heating rate led to an upward shift in the glow curve to high temperatures and a decrease in the intensity of TL, also the peak intensity at the temperature of 100 ℃ decreases considerably with the increase in the heating rate. The experiment also showed that the area under the TL glow curve, which represents the relationship between the heating rate and the intensity of the glow curve, decreased as the heating rate increased and became stable after $3\ ℃/s$ that is because the non-radiative recombination.

In the Reusability study, the ability of the used material (polycarboxylate cement) to produce thermoluminescence sequentially after many reads were examined. The results indicated that there was no change in the glow curve, no new peaks recognized when reading the sample several times in a row at a constant heating rate, and there was no shift of the peaks at high temperatures, the intensity of the peaks at lower temperatures $\sim 65\ ℃$ was unstable compared to the intensity at higher temperatures (peaks at $200\ ℃$ and $355\ ℃$) because; such emissions correspond to shallow traps in the sample where electrons are not stable where Shallow traps give rise to short-lived photoinduced absorption, there was no shifting in the position of peak- temperatures, and There was a recognized fluctuation in the area under the peaks for the low-temperature peaks located between 30 to $150\ ℃$, with no changes mentioned for peaks of high temperatures located between 150 to $400\ ℃$.

Briefly, we can summarize; the sample has a clear TL glow curve with three main peaks of $140\ ℃$, $220\ ℃$ and $330\ ℃$. It has a wide linear dose-response between $144\ Gy$ and $2.3\ KGy$ and good reusability with eight cycles of measurement and a high sensitivity. And thus, we can say polycarboxylate cement Powder can be used as a potential dosimeter.

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CURRICULUM VITAE

Muhammed Halidoglu

M.Sc. Gaziantep University, Engineering Physics, 2022.

B.Sc. Alfurat University, General Physics, 2012.

H.Sc. Ali-Ibrahim High school, Scientific department, 2007.