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Factors influencing the thermal stability of HEMA polymer gel dosimeters for clinical radiotherapy

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Abstract

Thermal stability is an essential feature required for practical applicability of gel dosimeters in radiation therapy planning system (TPS). This study pioneers a comprehensive experimental study of various factors that influence thermal stability of Polymer Gel Dosimeters (PGDs) by investigating the impact of gelatin source and its weight fraction (WF), type and WF of antioxidant, presence of maltose as a disaccharide additive, and the storage time post manufacturing, on the thermal properties of 2-hydroxymethyl methacrylate (HEMA) PGD. Results show that in terms of fast gelation and gel strength, gelatin from bovine skin outperforms gelatin from cold water fish, withstanding temperature up to 11 °C above its melting temperature T_m, and tetrakis(hydroxymethyl)phosphonium chloride (THPC) outperforms ascorbic acid (AscA), and sample with maltose additive outperforms sample without maltose. The melting rate (R_m) increases with the temperature difference above T_m, and storage time post manufacturing, improves the thermal stability. These findings contribute to the identification of the causes of gel instability and propose the possible solutions that could lead to more accurate assessments and maintenance of dose distribution in three-dimension (3D), and to extend the shelf life of PGDs.

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Keywords: 2-hydroxymethyl methacrylate (HEMA), Melting temperature, Polymer gel dosimeter, Thermal stability

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1. Introduction

Cancer is one of the major causes of death in the world [1]. Radiation therapy is a clinical procedure in which cancerous cells are destroyed by using ionizing radiation. However, during the irradiation, the surrounding healthy tissues must be prevented from being overdosed [2]. Polymer gel dosimeters (PGDs) are dosimetry tools made from monomers, water and antioxidants, held within a gel matrix such as agarose, gelatin, gellan gum (GG), polyvinyl alcohol (PVA), Sephadex, chloral hydrate agar, among others, to measure ionizing radiation in three-dimensional (3D) distribution [3–9].

The necessity of using hydrogel for gel dosimeters arises from the failure of the earlier Fricke gel dosimeters to retain the 3D dose distribution for more than two hours, due to ionic diffusion, which resulted in blurred images and loss of spatial

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resolution [10]. Appleby *et al.* [11] incorporated ferrous sulfate solution into agarose hydrogel to serve as the gel matrix, to limit the mobility of ions and improve the dosimeters spatial stability [11]. However, due to the weakly cross-linked chains of the hydrogel, they are prone to melting during irradiation, long-duration scanning procedures, such as Magnetic Resonance Imaging (MRI), or during shelf storage. For example, MAGIC PGD melts at 25 °C, which makes it not suitable for use in high temperature conditions, such as thermal column reactors [12] or in geographical regions with higher ambient temperatures [13]. Satialgine was found to liquefy during experimental procedure [14], and 2-hydroxymethyl methacrylate (HEMA) PGD prepared by Hiroki *et al.* [8], with gellan gum as a gel matrix, also melted at relatively low temperature [15], which makes it non-applicable for practical usage. Several attempts were made to improve the melting temperature (T_m) of gel dosimeters, to enable them for long-time imaging, or withstand the temperature rise during exothermal polymerization reaction [16, 17], and prevent flaccidity during transportation. Abtahi *et al.* [13] improved the T_m of MAGIC PGD from 25 °C to 60 °C through the addition of agarose [13]; however, agarose is translucent [18] and does not favor optical evaluation of the PGD. Al-jarrah *et al.* [15] improved the T_m of genipin gel dosimeter from 23.2°C to 26.5 °C by increasing the weight fraction (WF) of gelatin [15], but this improvement is still below the ambient room temperature (T_R) of various parts of the world. Furthermore, some additives, such as inorganic salts, added to PGDs for sensitivity improvement, were reported to decrease the T_m of gel dosimeters [15, 19–21].

Various investigations were carried out by researchers to characterize HEMA PGDs, for their radiation sensitivity [7, 8, 16, 17, 22, 23], linear dose range, and temporal stability [2]. The sensitivity of HEMA PGD with maltose additive was also evaluated [2, 24], and the temperature independence of their dose response was studied [2], but a comprehensive evaluation of the factors influencing the thermal stability of PGDs could not be found in the literature.

This study aims to comprehensively study the factors that influence the T_m of PGDs.

The study covers the investigation for the impact of gelatin source and its WF, the type and WF of antioxidant, the presence of maltose as an additive, and the storage time after manufacturing, on the thermal properties of HEMA PGDs, and the study contributes to the identification of the causes for thermal instability of PGDs, which leads to sustainable solution to poor gel strength of gel dosimeters. This study, in combination with the previous ones on other characteristics of HEMA and non-HEMA PGDs, contributes to the establishment of a solid foundation for PGDs' practical applicability in TPS. The materials and methods used for this study are detailed in Section 2.

2. Materials and methods

2.1. Materials

The reagents used for this study are: gelatin from cold water fish and from bovine skin, HEMA, N,N'-methylene-bis-acrylamide (Bis), maltose, tetrakis(hydroxymethyl)phosphonium chloride (THPC), all from Sigma-Aldrich, Germany, and deionized water, produced locally in the laboratory. Reagents were weighed by using an electronic beam balance with an accuracy within 10^{-4} g, and heating and mixing were achieved by using a magnetic stirrer hot plate. A fumehood was used to remove unwanted gases during the sample preparation.

2.2. Samples preparation

Samples of HEMA PGDs with different formulations were prepared for various investigations. The samples were prepared under normal atmospheric conditions (normoxic) [25], similar to a previous study by Alhassan *et al.* [5], where the components were added in a sequential manner starting from deionized water, Bis, gelatin, HEMA, THPC or ascorbic acid (AscA), and maltose (where applicable). The prepared gel solutions were poured into Perspex cuvettes of volume 4.5 cm^3 and stored in a refrigerator, maintained at 4-6 °C for gelation.

2.3. Experimental procedures

Several techniques were used to study the thermal properties of the HEMA PGDs, as shown in Figure 1, specifically,

- observing the gel state of the prepared PGD by using the naked eye [26],
- Simple flow test [27],
- Hot water bath experiment [15],
- thermogravimetry analysis (TGA), and
- differential scanning calorimetry (DSC) [26, 28, 29].

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2.3.1. Simple flow test

The simple flow test was used to study the gel stability of PGDs as described by Kozicki *et al.* [27]. This method was selected as it closely simulates the clinical workflow where dosimeters are equilibrated to ambient T_R before irradiation or scanning, and where the melting or fluid flow is observed with the naked eye [13, 30].

Prior to the experiment, the T_R was regulated by using an air-conditioner, while doors and windows were kept closed [8]. The temperature was monitored by using two agreeing mercury-in-glass thermometers. When the temperature stabilized at the intended temperature, with a discrepancy within ± 0.5 °C, for at least 30 min, three or more samples from each HEMA PGD formulation were taken out of the refrigerator and tilted at an angle of 45°, and allowed to reach thermal equilibrium with the T_R. The samples were monitored for 360 min to observe any sign of melting or fluid flow on the surface of the cuvette. The T_R was initially set at 16 °C, and then repeated with progressively higher temperatures within 17-29 °C, at an interval of 2 °C in each case. The samples were observed with the naked eye to record the temperature at which melting or fluid flow was observed [31].

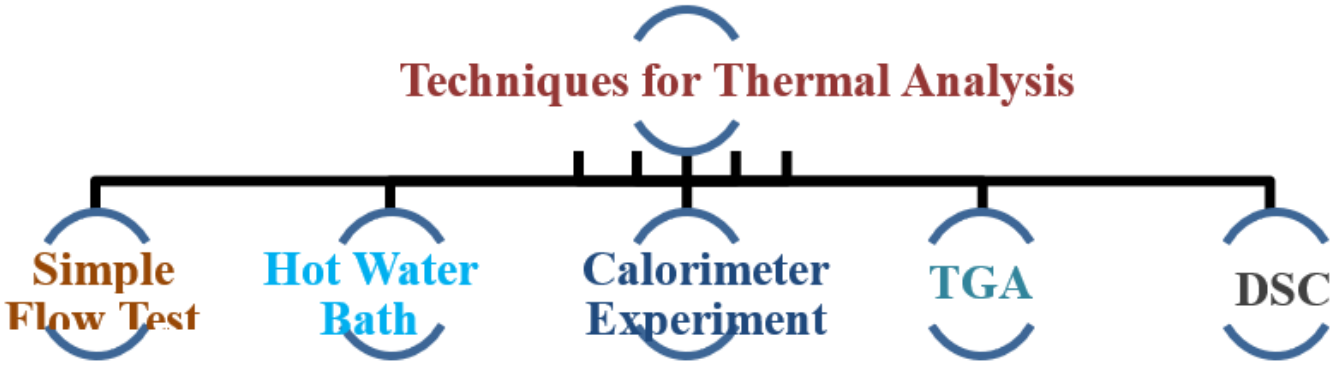


Figure 1: Techniques used for thermal properties evaluations.

2.3.2. Hot water bath experiment

The hot water bath experiment was described by Al-Jarrah *et al.* [15] and Bahrami *et al.* [30]. The technique makes use of a manual or digital electronic water bath. The PGD samples were tilted inside the water for monitoring, with inbuilt or immersed thermometers measuring the T_m. A magnetic stirrer hot plate heats the water while stirring continuously throughout the procedure to ensure uniformity. Alternatively, samples can be suspended inside the hot water, while the melting could be studied visually [24, 26].

2.3.3. Calorimeter experiment to determine Specific Heat Capacity (SHC)

SHC is the amount of energy (heat) required to raise the temperature of a unit mass of a sample by 1°C [32]. It reveals the thermal stability and heat resistance of substances. SHC of a sample can be calculated by using Equation (1).

$$Q = mc(T_2 - T_1), \tag{1}$$

where Q is the quantity of heat supplied, m is the mass of the sample, and T_1 and T_2 are the temperatures before and after the heat addition.

Calorimeter experiment is carried out according to Black's Principle, by mixing water at a higher temperature with gel solution at lower temperature, in a thermally isolated condition (calorimeter set). Black's Principle states that, in the absence of external heat loss, the heat lost by water equals the heat gained by the gel solution and calorimeter [32]. This relationship can be depicted in Equation (2).

$$m_w c_w(\theta_{c2} - \theta_w) = \left[m_c c_c + m_g c_g\right](\theta_{c2} - \theta_{c1}), \tag{2}$$

where m_w and c_w are the mass and the SHC of water (4.18 $Jg^{-1}K^{-1}$), c_c is the SHC of the glass calorimeter (0.84 $Jg^{-1}K^{-1}$), and c_g is the SHC of the gel solution.

2.3.4. TGA and DSC analyses

TGA evaluates mass loss as the temperature increases, while DSC provides insights into endothermic and exothermic processes during phase transitions [33]. The two procedures evaluate heat events in a sample, such as phase transitions, and reveal important thermal features, such as SHC at constant pressure (c_p), onset, inflection, mid, and end temperatures, and graphs displaying endothermic and exothermic processes [26, 34].

2.4. Evaluating the factors that influence the thermal properties of HEMA PGDs

Factors examined for their possible influence on the thermal behaviour of HEMAT PGD are summarized in Figure 2.

2.4.1. Effect of gelatin source on T_m

To evaluate the effect of gelatin source on T_m, two groups of HEMA PGDs were prepared: one with gelatin derived from cold water fish and the other with gelatin derived from bovine skin. In each group, four HEMA PGDs were prepared as described in Section 2.2. The gelatin WF was increased from 2 wt% to 8 wt%, HEMA, 6 wt%, Bis, 2.5 wt%, deionized water, 83.5-89.3 wt%, and AscA, 10 mM. The prepared gel solutions were transferred into 4.5 mL Perspex cuvettes and sealed with parafilm, then refrigerated for gelation at 4-6 °C. The T_m was assessed by using the simple flow test as described in Section 2.3.1.

2.4.2. Effect of gelatin WF on SHC

To study the effect of gelatin WF on the SHC of HEMA PGDs, a calorimeter experiment was carried out. HEMA PGD solutions of various gelatin WF, specifically, 2, 4, 6, and 8 wt%, were prepared. The mass of the calorimeter setup, including the lid and a glass stirring rod, was measured and recorded as m_c. A known mass of HEMA PGD solution was added to the calorimeter, and the new mass was recorded as m_{c2}. The mass of the gel solution, m_g, is thus the difference between m_{c2} and m_c. The initial temperature of the gel solution and calorimeter system was recorded as θ_{C1}. A known mass of hot water, m_w, at temperature θ_w was added to the calorimeter. The calorimeter was closed, and the mixture was stirred by using the glass rod through a hole in the lid. Once the mixture reached thermal equilibrium and the temperature stabilized, the final temperature was recorded as θ_{C2}. Assuming negligible heat loss,

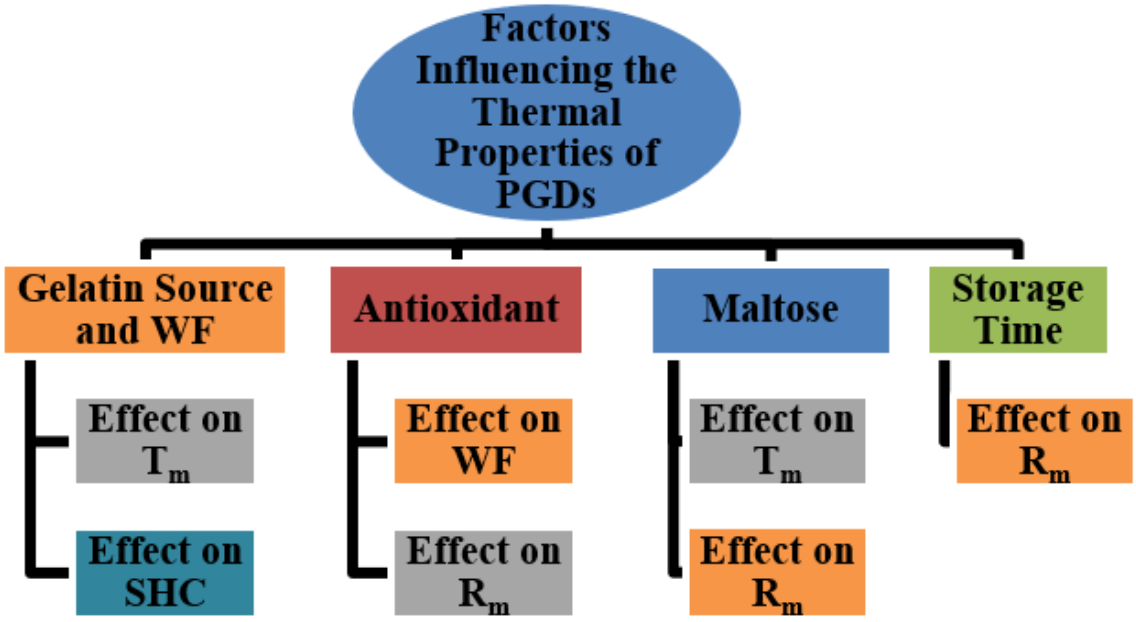


Figure 2: Factors influencing the thermal characteristics of HEMA PGD.

Black's Principle was applied to calculate the c_g of the gel. The procedure was repeated twice for each sample to determine the average c_g and the standard deviation (SD).

2.4.3. *Effect of gelatin WF and type of antioxidant on R_m*

Previously, Rabaeh *et al.* [35] studied the impact of gelatin WF and THPC concentration on the rate of PGD gelation. However, the effect of these factors on the R_m is more important, as it provides insights into the materials' ability to withstand higher or fluctuating temperatures without losing their 3D dose distribution during irradiation or scanning. To compare AscA and THPC in terms of their impact on the thermal properties of PGDs, two groups of HEMA PGDs were made: One with AscA and the other with THPC as an antioxidant. The formulations are similar to those in Section 2.2, with THPC now used in another PGD set.

A simple flow test was carried out. The data collection and evaluation were made in a novel approach developed here for a better understanding of the transition rate (melting rate [R_m]) of HEMA PGDs. This model resembles the graphical data presentation in TGA analysis, where a sample melts and loses weight with temperature, whereas in this new approach, the percentage of the number of PGD-filled cuvettes that melts over time replaces the weight loss in the TGA graph. The detailed procedure is that a set of N cuvettes containing the same HEMA PGD sample is exposed to a controlled T_R. At the initial time t_0, the probability (P) that all N vials are in the gel state is P = 1, meaning that 100 % of the sample is in gel form. At a later time, t_i, some number of vials (n) from the total N began melting at the surface. This indicates that $\left(\frac{n}{N}\right) \times 100$ % of the vials started melting, while $\left(1 - \frac{n}{N}\right) \times 100$ % of the vials remain in gel form.

The time at which all vials have completely melted is recorded as the point when P = 0, meaning 0% of the samples remain in gel form. The average time for dosimeters with varying gelatin WF to begin and complete melting at a specific temperature can be a determining factor in comparing the thermal stability of various formulations, which could reveal the impact of the type of antioxidant on the R_m of PGDs. This procedure was carried out and repeated for each of the two groups of HEMA PGDs (THPC-based and AscA-based) with various gelatin WFs across a temperature range of 16-29 °C.

2.4.4. *Effect of maltose on the T_m of HEMA PGD*

The effect of maltose concentration on the T_m of HEMA PGD was investigated by preparing samples with gelatin, 6 wt%, HEMA, 2.7 wt%, Bis, 2 wt%, AscA or THPC, 10 mM, and varying maltose concentrations ranging from 0 to 520 mM. The chosen gelatin WF was selected to ensure the samples would melt below 29 °C; otherwise, the simple flow test experiment could not be held. The simple flow test was carried out by progressively increasing the controlled T_R between 16-29 °C as described earlier. The temperature at which the sample melts is recorded.

2.4.5. *Effect of maltose on R_m of HEMA PGD*

PGDs with high T_m, or that remained in gel form up to 29 °C, cannot be examined by using a simple flow test experiment. Consequently, alternative methods such as hot water bath, TGA, and DSC were employed to further evaluate the T_m or R_m of the HEMAT PGD samples. Prior to the DSC procedure, TGA was carried out as a preliminary step to give an

Table 1: The input settings for DSC analysis on HEMAT PGDs.

S/No.	Parameter	Specification
1	Reference	Empty, 0 mg
2	Temperature Range	T_R-90 °C
3	Heating rate	20 °C min^{-1}
4	Atmospheric Condition	N_2

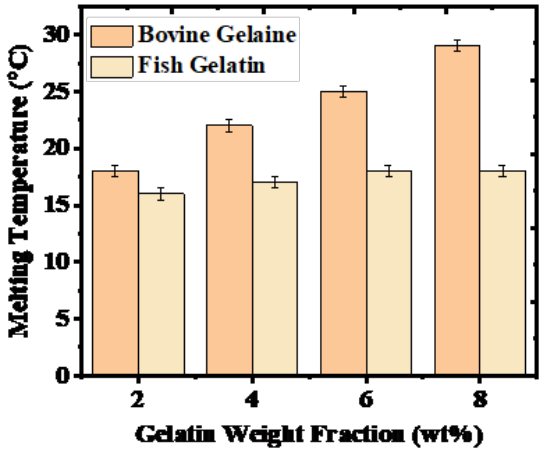


Figure 3: The variation of T_m with gelatin type and WF. The error bars represent the SD from three replicate measurements.

insight into the burning temperatures of the samples. The user input settings and specifications made for the DSC analysis are presented in Table 1.

2.4.6. *Effect of storage time on the T_m of HEMA PGD*

The thermal history of PGDs either during their preparation, cooling or storage processes is reported to have significantly influenced their dosimetry properties [23, 36]. In this study, the impact of storage time post-manufacture on the T_m of HEMA PGD was evaluated.

Four samples of HEMA PGDs were prepared and stored in a refrigerator, with the temperature within 4-6 °C. The effect of the storage time on the T_m was analyzed at various time intervals by using the simple flow test.

3. Results

3.1. *Effect of gelatin source on T_m*

Two groups of HEMA PGD samples, one prepared with gelatin derived from bovine skin and the other from gelatin derived from cold-water fish, were studied for their T_m as described in section 2.4.1. The T_m for each group, with gelatin WF of 2, 4, 6, and 8 wt%, is presented in Figure 3.

3.2. *Effect of gelatin WF on SHC of HEMA PGD*

Gelatin source and its WF are seen to have influenced the T_m of PGDs, in Section 3.1. Since SHC of a material body is

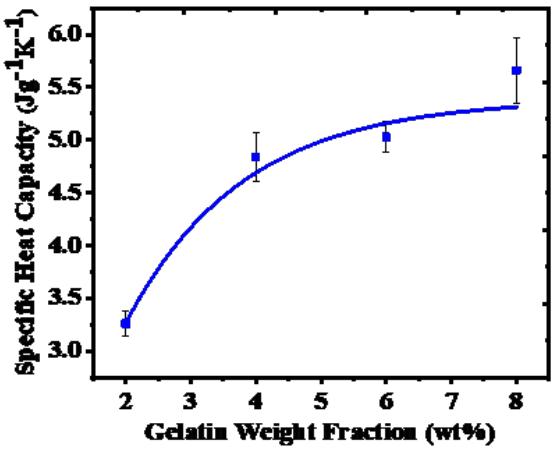


Figure 4: Relationship between gelatin WF and the SHC of HEMA PGD.

responsible for the amount of heat required to raise the temperature of its unit mass by 1 °C, and thus, reflects resistance to temperature rise, it is therefore a feature to characterize the thermal stability of materials. The SHC of four HEMA PGDs prepared with varying gelatin WFs, 2-8 wt%, was determined as described in Section 2.4.2. The graph of SHC against gelatin WF is plotted and fitted to an exponential asymptotic curve in Figure 4.

3.3. *Effect of antioxidant on R_m*

The rate at which PGD melts is an important parameter to characterize its thermal stability: The one with lower Rm is expected to retain the 3D structure for a longer time.

Two groups of PGDs were prepared with bovine skin gelatin, 2-8 wt%, with one of them containing THPC, and the other containing AscA as an antioxidant. The percentage of HEMA PGD-filled cuvettes that remains in a gel state at a given time, for various temperatures, was plotted as a function of the time taken for the samples to start and completely melt. The graphs for various temperatures are presented in Figure 5.

The average melting time (T_{ave}) for HEMA PGDs under study is plotted against the excessive temperature above their Tm as presented in Figure 6.

3.4. *Effect of maltose on the T_m of HEMA PGD*

The effect of maltose as an additive on the thermal properties of HEMA PGDs with AscA or THPC as antioxidants was examined, as described in Section 2.4.4. The T_m of the four HEMA PGDs with AscA containing different maltose concentrations (0 mM, 80 mM, 230 mM, and 520 mM) are presented in Figure 7.

3.5. *TGA and DSC analysis*

Since HEMA PGDs with THPC do not melt up to a temperature of 29 °C by using a simple flow test, and up to 60 °C by using a hot water bath experiment, the difference in T_m or any

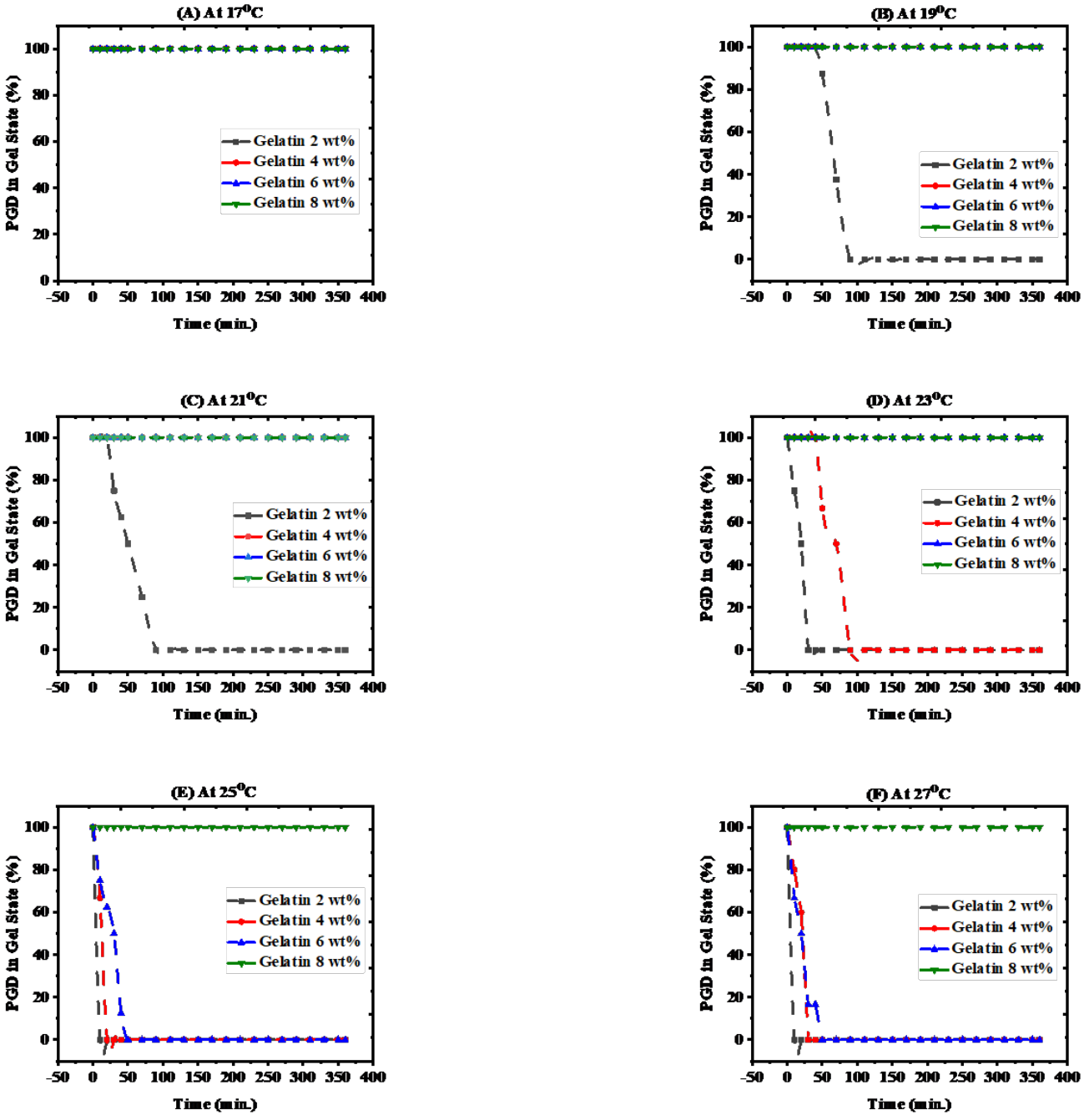


Figure 5: The R_m of HEMA PGDs with varying gelatin WF. (A) - (G) are for HEMA PGDs containing AscA as an antioxidant, while (H) is for HEMA PGDs containing THPC as an antioxidant.

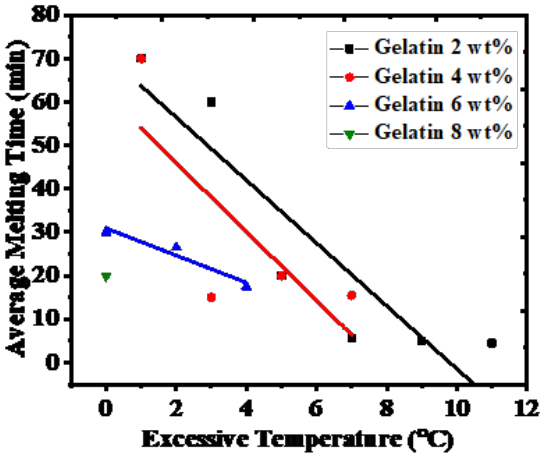


Figure 6: Relationship between T_{ave} and excessive temperature above T_m of HEMA PGD samples.

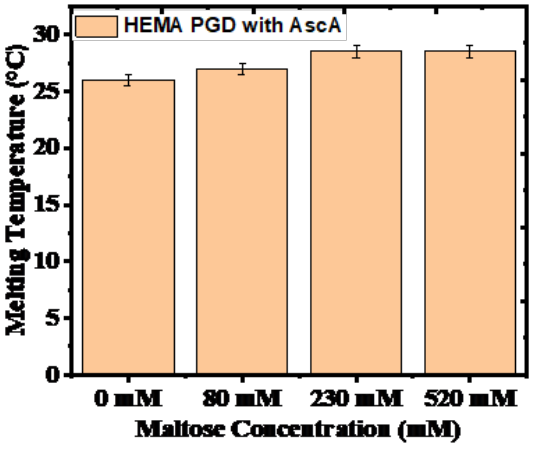


Figure 7: The improvement in T_m of HEMA PGDs with AscA due to presence of maltose additive.

other thermal property between PGDs with and without maltose could not be evaluated. This makes it difficult to further assess the influence of maltose on their thermal characteristics. However, TGA and DSC analyses were performed on two samples, one without maltose (maltose, 0 mM) and the other with maltose 80 mM, to provide a comparison between them [37].

The results from the TGA analysis are presented in Figure 8, with the numerical results presented in Table 2.

Table 2 shows that, for the HEMA PGD which does not contain maltose, the rise in temperature $\Delta X = 61.69$ °C resulted in the weight loss $\Delta Y = -71.00$ %. This implies that the rate of weight loss with respect to temperature is ≈ 1.15 mg °C^{-1}. On the other hand, for the HEMA PGD, which contains maltose 80 mM, the average weight loss per unit temperature rise is ≈ 1.12 mg °C^{-1}, which indicates that the sample with 80 mM maltose is thermally more stable than the sample which do not contain maltose.

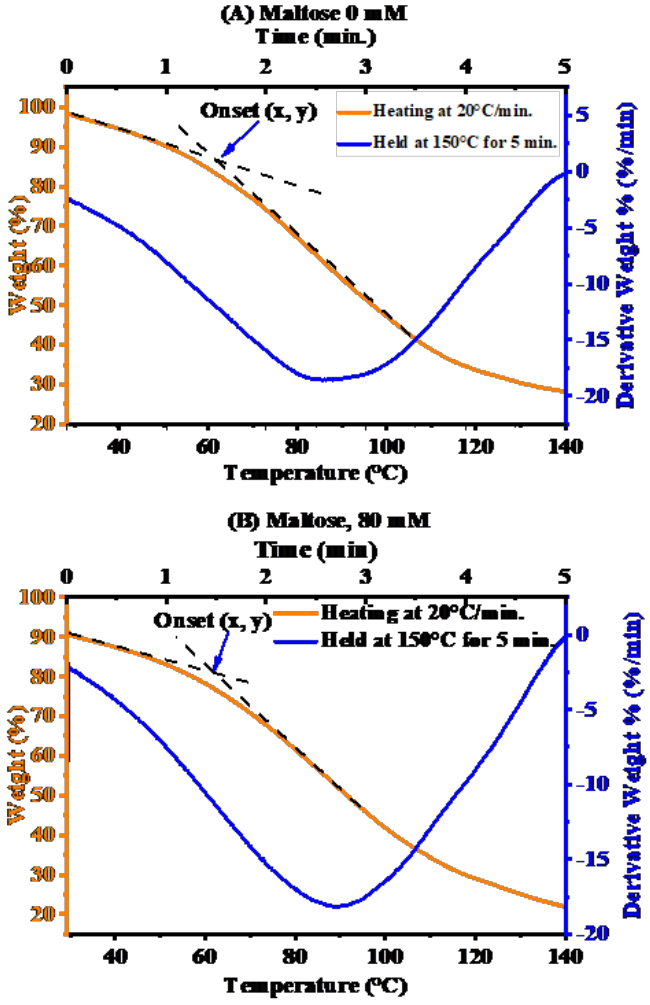
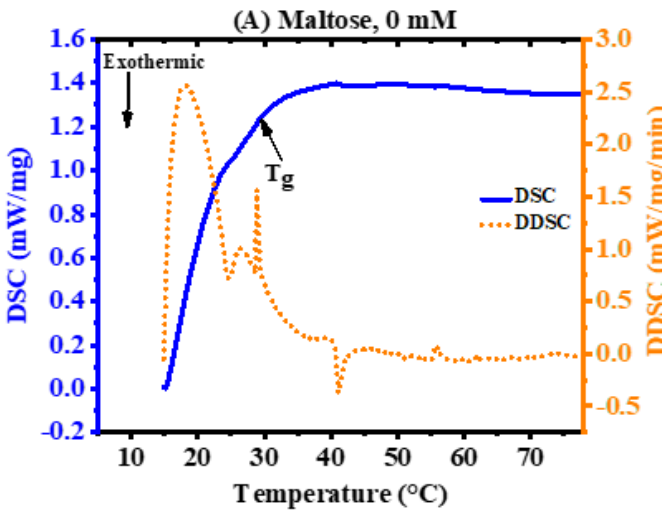


Figure 8: TGA analysis, showing the onset (x, y) values for HEMA PGDs: (A) without maltose, and (B) with maltose 80 mM.

Table 2: Summary of the findings, comparing the thermal properties of HEMA PGDs, with and without maltose, from the TGA analysis.

Variable	Maltose 0 mM	Maltose 80 mM
Original Weight	5.175 mg	5.591 mg
X_1	28.250°C	29.350 °C
X_2	140.500 °C	140.210°C
Y_1	99.075 %	91.364 %
Y_2	28.074 %	21.663 %
ΔY	71.001 %	69.701 %
Onset Y	86.105 %	79.286 %
Onset X	61.69 °C	62.35 °C

The thermal properties of the HEMA PGDs based on DSC analysis are presented in Figure 9.



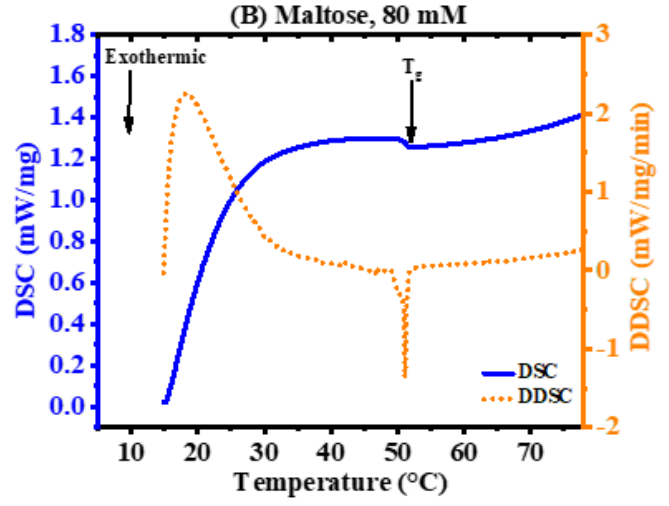
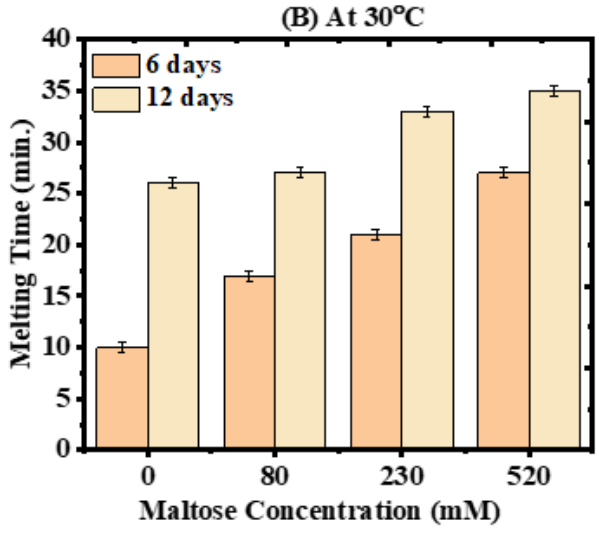


Figure 9: The DSC curves indicating the heat flow and the onset temperatures for HEMA PGDs with (A) maltose, 0 mM, and (B) maltose, 80 mM.



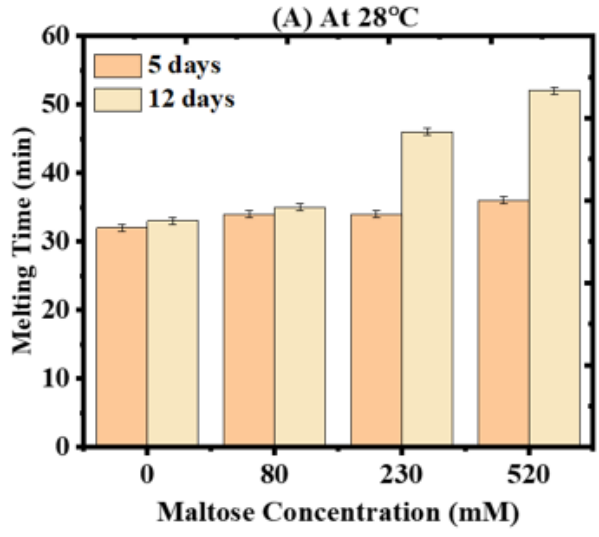


Figure 10: The melting times of HEMA PGDs with maltose concentrations 0-520 mM are presented at (A) 28 °C and (B) 30 °C.

3.6. Effect of storage time on the T_m of HEMA PGD

The effect of storage time post-manufacture on the T_m of HEMA PGD stored at a controlled temperature was studied. In this regard, four samples of HEMA PGDs with maltose concentrations ranging from 0 mM to 520 mM, and with AscA as an antioxidant, were investigated. The melting times of the samples were examined at various time intervals after manufacture. The times taken by the PGDs to melt at two different temperature conditions are presented in Figure 10.

4. Discussions

4.1. Effect of gelatin source on T_m

Figure 3 shows the variation of T_m with two types of gelatin, each at various gelatin WFs. The results indicate that the T_m of the PGDs made from fish gelatin increases from 16 °C to 18 °C as the gelatin WF increases from 2 to 8 wt%. This shows that there is an improvement in the T_m when the gelatin WF increases within this range. However, the maximum temperature that gelatin from cold water fish could withstand to remain in gel form is 18 °C, while HEMA PGDs made with gelatin from bovine skin show a T_m of 18 °C when 2 wt% of gelatin was used, and increases to 29 °C as the gelatin WF rises from 2 to 8 wt%.

These observations highlight two important points: (i) Increasing gelatin WF results in higher Tm, irrespective of the source of the gelatin. This improvement can be linked to the crosslinking density when the WF increases in a given mass or volume of gel recipe, and is related to a higher number of bonds between the polymer chains of the gelatin. Additionally, higher WF makes the gelation process faster, thus, it enhances the T_m [35]. (ii) PGDs made from bovine skin gelatin show higher T_m compared to PGDs made from fish gelatin at

all WF, within 2-8 wt%. This could be attributed to the difference in the gelatin type (source) [34]. Similar difference in T_m due to difference in the gelatin type was previously observed in gelatin from three different sources, namely, bovine skin, freshwater fish, and cold-water fish [38], which could be attributed to the difference in the composition of amino acid, and molecular weight distribution among the gelatin sources and species [38, 39]. Conclusively, gelatin source affects the T_m of PGDs, with gelatin from bovine skin outperforming gelatin from cold water fish.

4.2. *Effect of gelatin WF on SHC of HEMA PGD*

Figure 4 illustrates an increase in SHC with increasing gelatin WF. This indicates that a HEMA PGD with higher gelatin WF is less prone to melting at hot environments, and will be relatively more thermally stable under exothermic polymerization, MRI scanning, or storage environment, and can therefore, preserve radiation dose distribution in 3D for longer time. The relationship between gelatin WF and SHC in Figure 4 is fitted to an exponential asymptotic curve (R^2 = 0.9809), and the curve is defined mathematically by Equation 3.

$$y = a - bc^x, \tag{3}$$

where; y: is the SHC, x: is the gelatin WF, a: is the upper asymptote, b: is the range of y, and c: is the rate at which y increases with respect to x.

The curve in Figure 4 in combination with equation 3 hints that; an increase in gelatin WF, represented by x, results in an increase in the SHC, represented by y, and y tends to saturate at higher x. This increase in SHC is likely attributable to a denser polymer network and increased hydrogen bonding, which requires more energy to induce molecular motion. The result further reveals that the SHC of HEMA PGDs will not be increasing indefinitely as gelatin WF keeps on increasing, rather, it will reach a saturation limit, after which it either remain constant or drop, and this can be attributed to changes in the gel structure. Similar improvement in thermal stability due to higher gelatin WF, was previously observed in VIC dosimeter, evaluated by using DSC analysis. The result shows that VIC sample containing 5% gelatin has glass transition temperatures (T_g) of 33 °C, while sample containing 7.5% gelatin has T_g of 38 °C. The two samples also have different gelation time [26], which indicates different rate of gelation, impliedly, different R_m. This leads to the conclusion that higher gelatin WF influences and enhances thermal stability of PGDs. These findings are also in agreement with the findings of Al-jarrah *et al.* (2016), in genipin gel dosimeters, where an increase in the gelatin WF from 2% to 7% resulted in rising the T_m of the dosimeter from 23.2 °C to 26.5 °C [15].

4.3. *Effect of antioxidant on R_m*

Figure 5(A) shows that all HEMA PGDs prepared with AscA remain in a gel state at 17 °C for up to 360 min. Figure 5(B) shows that at 19 °C, the sample with 2 wt% gelatin started melting in approximately 50 min and completely melted by 90 min, with a total melting time of about 40 min. This sample has a T_m of 18 °C, as presented in Figure 3, while the samples with higher gelatin WF remained in a gel state. All these samples have a T_m above 19 °C as presented in Figure 3.

Figure 5(C) shows that, at 21 °C, the sample with 2 wt% gelatin started melting in approximately 30 min and completely melted by 90 min, while samples with higher gelatin WF remained in a gel state. Comparing the HEMA PGD sample with 2 wt% gelatin at 19 °C and at 21 °C, the time at which the melting started has reduced. Figure 5(D) shows that at 23 °C, the sample with 2 wt% gelatin started melting around 10 min and completely melted by 30 min, while the 4 wt% gelatin sample started melting at 50 min and completely melted at 90 min. The sample with 4 wt% gelatin has a T_m of 22 °C, as presented in Figure 3. Higher gelatin WF samples remained in a gel state.

Figure 5(E) shows that at 25 °C, the sample with 2 wt% gelatin melted almost immediately within the first 10 min, the 4 wt% gelatin sample began melting at 10 min and completely melted by 20 min, while the sample with 6 wt% gelatin started melting before 10 min and completely melted around 50 min. The 6 wt% sample has a T_m of 25 °C (Figure 3), but the sample with 8 wt% gelatin (T_m = 29 °C) remained in a gel state.

Figure 5(F) shows that at 27 °C, the sample with 2 wt% gelatin started and fully melted in less than 10 min. The 4 wt% gelatin sample started melting at 10 min and melted completely by 30 min, while the 6 wt% gelatin sample started melting around 5 min and fully melted in 48 min. Figure 5(G) shows that at 29 °C, the sample with 2 wt% gelatin started and completely melted within 8 min. The samples with 4 wt% and 6 wt% gelatin also melted within 30 min. However, the sample with 8 wt% gelatin started melting at around 10 min and fully melted by 30 min.

Figure 5(H) shows the results for HEMA PGD samples prepared with bovine gelatin and THPC as an antioxidant. All these samples remained in a gel state for up to 360 min at all the tested temperatures, thus only the graph for the highest temperature is presented. These results reveal four important things:

- PGDs can remain in a gel state at temperatures below their T_m for an extended period of time without deformation.

- For HEMA PGDs prepared with AscA, exposed to varying temperature conditions above their T_m, the samples with a lower T_m consistently exhibited a faster onset of melting, and a shorter time to complete liquefaction. Example can be seen in the sample with 2 wt% gelatin, which started melting at 50 min at 19 °C, 30 min at 21 °C, 10 min at 25 °C, and completely melted by 90 min at 19 °C, before 90 min at 21 °C, 30 min at 25 °C, and in less than 10 min at both 27 °C and 29 °C.

  A similar trend can be seen in the sample containing 4 wt% gelatin, as it began melting at 50 min at 23 °C, 10 min at 25°C, and less than 10 min at both 27 °C and 29 °C, and completely melted by 90 min at 23 °C, 30 min at 27 °C, and in less than 30 min at 29 °C. Similarly, other HEMA PGD samples with higher gelatin WF behave in the same manner

- The higher the temperature above a sample's T_m, the faster it melts.

Figure 6 shows that the T_{ave} decreases linearly as the excess temperature (ΔT) above their T_m increases. Where ($\Delta T = T - T_m$). For example, Figure 6 shows that T_{ave} decreases by (7.2 min, $R^2 = 0.8274$), (7.9 min, $R^2 = 0.5883$), and (3.1 min, $R^2 = 0.9394$), for every 1 °C difference between the T_m and the T_R, for HEMA PGDs with 2, 4 and 6 wt% gelatin, respectively.

Since T_{ave} determines the rate at which a sample melts, we postulated here that, for samples with the same SHC and thermal conductivity, under the same thermal conditions, the rate at which a given mass, m, or volume, v, of HEMA PGD melts ($\frac{dm}{dt}$) or ($\frac{dV}{dt}$), respectively, is proportional to ΔT. This relationship can be presented in Equation 4.

$$\frac{dm}{dt} \propto \Delta T \quad \text{or} \quad \frac{dV}{dt} \propto \Delta T. \tag{4}$$

The observation number (iv) is about the effect of THPC on gel stability: For PGDs prepared with bovine skin gelatin (WF 2-8 wt%) and containing THPC as an antioxidant, all samples remained in a gel state for 360 min. This suggests that THPC either contributes to improving the T_m of the PGDs or AscA lowers the T_m of the PGDs. The first possibility is supported by several studies, for example:

- In an investigation conducted by Rabaeh *et al.* [35] on the effect of gelatin WF from 2.5 to 5 %, and THPC concentrations from 5 mM to 30 mM, on the gelation time of NHMA-based PGDs, an increased in the concentration of THPC is reported to had accelerated the setting time (gelation time) at fixed T_R, thereby improving the T_m of the NHMA gel dosimeters [35, 40], and

- substituting AscA with THPC and increasing the gelatin WF from 5% to 7.5% in VIC-T dosimeters is reported to result in a reduced gelation time from several hours to just a few min. This observation prompted Jaszczak *et al.* to investigate the impact of THPC and gelatin WF on gelation time, the results of which revealed that, under the same temperature conditions, samples with higher gelatin WF (7.5%) form physical gels faster than those with lower gelatin WF (5%) [26].

Similarly, in this study, under the same temperature conditions, the sample with higher gelatin WF remains in a gel form longer than the sample with lower gelatin WF. Moreover, the samples containing THPC exhibit a higher heat resistance compared to the sample with AscA. These results provide an evidence for the influence of THPC in improving the T_m of PGDs, more especially, based on bovine skin gelatin.

4.4. Effect of maltose on the T_m of HEMA PGD

Figure 7 indicates that for HEMA PGDs with AscA as an antioxidant, and without maltose (maltose, 0 mM), the T_m is 26 °C, while the T_m for HEMA PGD with maltose 80 mM is 27 °C, and the T_m for both HEMA PGD with maltose 230 mM and 520 mM is 28 °C.

As shown in Figure 7, adding 80 mM maltose results in a 1 °C (3.85%) increase in T_m, while the addition of 230-520 mM maltose leads to a 2 °C (7.70%) improvement in T_m. This indicates a slight enhancement in the T_m of HEMA PGDs doped with maltose, although it contains AscA as an antioxidant.
For samples with THPC as an antioxidant, all samples remained in gel form for 360 min up to a T_R of 29 °C, which suggests that their T_m is above 29 °C. A hot water bath experiment was performed with increasing temperatures up to 60 °C, but no sign of melting was observed. This observation further indicates the role of THPC and maltose in improving the T_m of HEMA PGD formulations.

4.5. TGA and DSC analysis

Figure 9 shows the DSC or heat flow per unit mass, measured in mW/mg of a sample on the left vertical axis, and the differential DSC (DDSC), which represents the rate of heat flow per unit mass in a sample on the right vertical axis. Figure 9(A) shows that the Tg of the HEMA PGD without maltose (maltose, 0 mM) is 29.5 °C, with a delta C_p of 0.11 $Jg^{-1}K^{-1}$, while that of HEMA PGD with maltose 80 mM is 50.9 °C, with a delta C_p of 0.13 $Jg^{-1}K^{-1}$. This demonstrates that the HEMA PGD with 80 mM maltose requires more heat to raise its temperature by 1 °C or 1 K, and also requires a higher temperature to undergo the transition from a rigid to a more flexible state (glass transition). This suggests that the sample with maltose is less prone to deformation and physical changes as temperature rises, which indicates higher thermal stability.

The mechanism behind the improved thermal stability of Gelatin-based PGDs with the maltose additive can be explained based on the strengthening of the junction zones of gelatin, due to the presence of saccharides. The junction zones are regions within the collagen triple helix structure. When saccharides or polyols are added, they increase the proportion of protein that reverts to this conformation, which causes the junction zones to become smaller and more numerous. This leads to a more extensive gel network, thereby increasing rigidity and thermal stability [41].

The improvement in T_m with the addition of maltose is consistent with the improvement in T_m from 25 °C to 34 °C, observed when another saccharide, glucose, was added to genipin gel dosimeters [15, 42].

4.6. Effect of storage time on the T_m of HEMA PGD

Figure 10 shows the variation in melting time with respect to different maltose concentrations and varying numbers of days post-irradiation. In Figure 10(A), HEMA PGD samples with maltose concentrations of 0 mM, 80 mM, 230 mM, and 520 mM, examined 5 days, post-manufacture at 28 °C, melted at 32, 34, 34, and 36 min, respectively. After 12 days, they melted at 33, 35, 46, and 52 min, respectively. At a higher temperature of 30 °C, the PGDs evaluated 6 days after manufacture melted at 10, 17, 21, and 27 min, respectively, while after 12 days, they melted at 26, 27, 31, and 35 min, respectively. These results indicate that, in addition to the effect of maltose concentrations on improving the melting times of PGDs, the samples

take a longer time to melt at lower temperatures, and also show extended melting times at longer storage times.

The observed delay in melting time of PGDs due to longer storage times, which indicates increased stabilization with time, can be attributed to the gelation process of gelatin, which is found to be time-dependent [35, 43]. Independent research is recommended to investigate the physical and chemical phenomena behind this observation in the future.

5. Conclusion

Factors enhancing the thermal stability of HEMA PGDs were investigated. The gelatin source is found to impact the T_m of the HEMA PGDs, with bovine skin gelatin exhibiting higher T_m compared to gelatin from cold-water fish. Similarly, the gelatin WF improves the T_m in both PGDs, those with gelatin derived from bovine skin and gelatin derived from cold-water fish, and increases the SHC of the HEMA PGDs. Furthermore, the type of antioxidant, in combination with the gelatin WF, plays a role in enhancing the T_m and R_m, with THPC outperforming AscA in the enhancement of the thermal stability of the PGDs. Maltose was found to improve the thermal stability of PGDs by increasing both T_m and R_m, and also, the storage time post-manufacture contributes to increased rigidity and an enhanced thermal stability of the PGDs. Conclusively, HEMA PGDs with gelatin from bovine skin, THPC as an antioxidant, maltose as an additive, is proposed for improved thermal stability, without compromising dosimetric and radiological properties, and an independent research is recommended in the future to investigate the physical or chemical phenomena behind the improved thermal stability due to longer post manufacturing time, using a combined experimental approaches of DSC, Rheometry and Small-Angle X-ray Scattering (SAXS).

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Data Availability

The authors have not used external data for this manuscript.

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APPENDIX A.

List of Abbreviations

3D	Three dimension
AscA	Ascorbic acid
Bis	N,N′-methylene-bis-acrylamide
DDSC	Differential DSC
DSC	Differential scanning calorimetry
GG	Gellan gum
HEMA	2-hydroxymethyl methacrylate
Normoxic	Normal atmospheric condition
PGD	Polymer gel dosimeter
PVA	Polyvinyl alcohol
R_m	Melting rate
SHC	Specific heat capacity
T_{ave}	Average melting time
T_g	Glass transition temperatures
TGA	Thermogravimetric analysis
THPC	Tetrakis(hydroxymethyl)phosphonium chloride
T_m	Melting temperature
TPS	Therapy planning system
T_R	Room temperature
WF	Weight fraction