Polyvinyl Alcohol-based Hydrogel: A Systematic Literature Review on Thermal Properties by Differential Scanning Calorimetry

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Abstract. The purpose of this systematic literature review (SLR) study is to assess the importance of thermal properties and the depth of analysis on Polyvinyl alcohol (PVA)-based hydrogel. Google Scholar is used as a search for database sources using Publish or Perish software, which is related to PVA-based hydrogel's thermal properties from 2018 to 2020. Thermal properties are focused on the DSC thermogram characteristic. These results strongly indicate that PVA-based hydrogel's thermal properties are very important and considered for their potential application.

1. Introduction
PVA has been considered as one of the attractive synthetic polymers over a few decades as it possesses desirable properties such as non-toxicity, biodegradability, biocompatibility, water solubility, and chemical and thermal stability [1–7]. It has prepared by chemical modification like partial or complete hydrolysis of polyvinyl acetate [6, 7]. PVA (Figure 1.) holds an enormous number of hydroxyl (–OH) groups encourage a various reaction, like with new functional groups [8–10], cross-linking between the -OH groups and other polar molecules to form entanglements and aggregates [11–13]; Hence, It has highly hydrophilic and dispersible in water [11,12]. A semicrystalline hydrophilic, odorless, tasteless, and translucent white color granular, as PVA physical characteristics. Moreover, It has an excellent film-forming, emulsifying and adhesive property [14, 15].

In recent years, physical and mechanical properties of PVA has been improved by blending with various polymers [16-20], incorporation natural materials [21–24], organic molecules [25, 26] and nanoparticles [27]. Based on its excellent properties, PVA have led to various biomedical [28] and pharmaceutical [29, 30] applications including but not limited to: fibrous meshes and membranes for wound dressings [31,32], drug delivery systems [33–35], interventional radiology embolization particles [36], and hydrogels used in orthopedic medicine [37], contact lenses [38]. The wide range of applications for PVA has resulted in many investigations over the past four decades into both its fundamental thermal [39–46] and structural [47–49] properties as well as the crystallization behavior of PVA hydrogels, PVA nanofibers [50, 51], and PVA blends and composites [52, 53].
The techniques of thermal analysis provide important tools in order to characterize thermal properties of these materials. When a polymeric system absorbs heat, many physical processes and phase transitions take place [54]. In order to understand these physical and macromolecular changes, the tools of thermal analysis, namely thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), have been used by researchers [55, 56]. The techniques of thermal analysis have been used for the determination of the crystallinity of copolymers, which varies with incorporation of some additives [57]. The incorporation of nanoparticles in polymer-based hybrid materials is found to affect the thermal properties of this material [58, 59].

From thermal analysis, relaxation processes that occur in the polymeric material, such as α-relaxation and β-relaxation can be understood. The α-relaxation (glass transition) phenomenon is associated with side (functional) groups of polymers, whereas β-relaxation is associated with main chain molecular motion. Compatibility (complete mixing) of polymer blends can be determined using DSC curves. Miscible blend films exhibit one phase and a single glass transition temperature (T_g). Immiscible blends are phase-separated and exhibit multiple T_g’s, one for each component [59, 60]. The microstructural modifications can also be studied by estimating thermal parameters like glass transition temperature (T_g) and melting temperature (T_m) using DSC [61].

A systematic literature review (SLR) is defined as systematic and explicit methods to classify, select, and critically evaluate relevant primary research, and to extract and analyze data from several previous studies' [62]. An SLR can provide useful insights to both academics and practitioners [63]. An SLR should sensitive; because it aims to seek as many studies as possible hence reduced bias and to be effective for under review [64]. Moreover, SLR an important tool for synthesizing scientific evidence, enhancing the validity of the results of studies, and identifying areas of uncertainty where the study is needed [65].

This study aims to carry out a SLR analysis on thermal properties of PVA-based hydrogel by DSC, following the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) methodology [66]. DSC as a tool or instrumentation to deepen the study of its thermal properties. DSC has an important role in observing thermal properties as well as its excellent performance (namely T_m and T_g). However, in certain circumstances, the thermogram results cannot provide strong and accurate information regarding the T_g value. The dynamic mechanical-thermal analysis (DMTA) is here as an alternative solution for measuring the value of T_g. Thus, the sample preparation process here also greatly determines the quality of the thermogram data used as primary data in future research and report.
2. Method

Through a holistic and organized specification that adheres to standard protocols, SLRs provide readers with detailed knowledge of literature in a region [67–70]. SLRs also help to clarify current information gaps and to find areas for future study [71, 72]. Although, different authors propose different approaches for conducting the systematic literature review process, there are three main steps found to be shared in all of them [63]. This research covers these measures following Ortiz-Martínez et al. [62] suggested guidelines.

2.1. Protocol

Under the PRISMA process, the latest systematic review has been carried out. It consists of well-defined stages of analysis and the creation and definition of information source eligibility requirements, literature search strategy, literature selection process, and data synthesis based on selected literature [62, 66]. According to DSC results, this systematic approach involves defining and discussing PVA-based hydrogel of thermal properties through their parameters.

2.2. Eligibility criteria

To determine and limit the form of literature in this study, the determination of eligibility requirements will be chosen. Eligibility criteria consist of criteria for inclusion and exclusion. The inclusion criteria in the present review were: (1) articles that documented PVA-based hydrogel thermal properties; (2) articles published in 2018 to 2020; (3) reports that have detailed relevant details. In addition, the exclusion requirements were: (1) unquoted articles; (2) review articles; (3) chapter books and books; (4) patents; (5) works written in a language other than English; (6) PVA-based hydrogel articles without thermal analysis.

2.3. Search strategy and information source

In order to obtain the data generated by published papers in 2018-2020, a bibliographic search was carried out. In a systemic analysis with PRISMA, the database quest can be done with applications such as Publish or Perish (PoP). The PoP search technique uses keywords so information that is accurate and applicable to the topics agreed upon by all writers can be comprehensively contained. For the format of the keywords, the Boolean algorithm is used and we used "polyvinyl alcohol" AND "thermal properties" AND "thermal analysis" AND "differential scanning calorimetry" AND hydrogel while Google Scholar is the chosen data source and we obtain the meta-data type details (n = 641).

2.4. Study selection

Selection of data based on the PRISMA technique. The review team consisted of all writers who screened names, abstracts, and full texts of the works for possible inclusion separately. According to the inclusion and exclusion criteria, they were assessed by all authors in the section on the eligibility criteria. To handle the 641 works accumulated by one database, the Mendeley Version 1.19.4 program was crossed over. We chose the subject of the remaining works and the works associated with them. When the review team was unclear about whether or not a given work met the inclusion requirements, the complete works were downloaded for further examination. Besides, in search of potentially appropriate articles, the selected works' reference list was also examined; however, no additional works were found in this step. Finally, after the full-text eligibility assessment, a total of 13 papers were found to be suitable for the present systematic review. The article selection process has been explained in Figure 2.

2.5. Data collection

Because of this review phase, data extraction by the review team must be precise and unbiased. From the 13 papers included in the present systematic review, qualitative and quantitative data were extracted using a data extraction form built for use in the present analysis. Table 1 presents a portion of this data extraction form.
2.6. Data items
The review team performed data extraction on the basis of DSC data (namely T_m and T_g) of the 13 selected article variables for which data relevant to the publication of the article were searched, such as the first author, year, title, materials, and thermal parameters.

2.7. Synthesis of results
In this current systematic review, a synthesis of the findings based on the discussion of the empirical patterns in the data provided without meta-analysis lacked adequate statistical details. The search method with PoP resulted from some variable data such as patent, book, website, etc. It does not support the meta-analysis performed.

3. Results and Discussions

3.1. Article selection
Several stages of selection were passed based on the illustration in Figure 2. Of the 641 papers initially retrieved, 61 were chosen based on the title and abstract. Of these, 13 were included in the study, along with an additional study found by manually reviewing the reference lists of the articles chosen. Table 1.a to 1.b lists the 13 papers included. The dates of release range from 2018 to 2020. In the sample populations, there was no overlap.

3.2. PVA characteristic
The chemical stability and hydrophilic nature of PVA are great [73, 74]; It caused undergoes a rearrangement of chemical bonds when PVA is heated above 300°C, followed by the establishment of some form of linkages by hydrogen bonding with water molecules [75, 76]. Highly conjugated aromatic structures are formed during the heat treatment of PVA, transforming them into cross-linking agents at high temperatures [77].

Generally, PVA is known to decompose in two stages [59, 78]. In the first stage, which begins at 245°C, the formation of volatile products is followed by dehydration. The water molecule (H_2O) is removed from the polymer chain, and polyene structures are formed. These residues are mainly polymers with unsaturated structures in conjugation. In the second stage, the remaining residues of polyene are further degraded from 430 to 450°C to produce carbon and hydrocarbons.

3.3. Thermal properties by DSC thermogram
Measurements of differential scanning calorimetry can be used, in particular, to consider the thermal background and stability of polymer samples [79]. Two endothermic peaks related to the T_g and T_m transition are observed based on the thermograms [80]. As estimated from the extensions of the pre- and post-transition baselines, the T_g is calculated as a mid-point in the thermograms [20], [81]. The degree of polarity and essence of the material is explained by the T_g [80].
Figure 2. Flow diagram of articles selection process
Figure 3. DSC thermograms of PVA, K, and K/PVA [82]. Note, K is k-carrageenan.

The thermograms' cooling curve mentioned by Croitoru et al. [82] contains one characteristic, namely the large exothermic peak attributed to hydrogel network water crystallization (Figure 3). The melting temperature of the non-associated (free) water in the hydrogel is indicated by an endothermic peak for PVA at ~13°C and K at ~11°C. The sample with the highest degree of crystallinity and the highest thermal stability was K/PVA (1:6) at ~9°C [83].

Figure 4. DSC thermograms of PC2, PCC2, PCH2, and PCHC2 [84]
Table 1.a Summary of article results obtained with SLR method

| Authors            | Years | Titles                                                                 | Materials                                          | $T_g$ | $T_m$ |
|--------------------|-------|------------------------------------------------------------------------|----------------------------------------------------|-------|-------|
| Khaleghi, et al. [84] | 2018  | Synthesis and characterization of new honey incorporated double-network hydrogels based on poly(vinyl alcohol) and acylated chitosan | PVA/ CaCl2/ Chit-MA/ Honey                         | *     | *     |
| Kamoun, et al. [20]  | 2018  | In-situ UV-photopolymerized PVA-g-GMA hydrogels for biomedical applications: I. Synthesis, characterizations and grafting optimization | PVA-g-GMA                                          | *     |       |
| Martínez-Gómez, et al. [85] | 2018  | Characterization of poly-D-mannuronate and poly-L-guluronate block fractions from sodium alginate and preparation of hydrogels with poly(vinylalcohol) | MM/PVA and GG/PVA                                  | *     |       |
| Yang, et al. [27]    | 2018  | Polyvinyl alcohol/chitosan hydrogels with enhanced antioxidant and antibacterial properties induced by lignin nanoparticles | PVA/Ch/LNPs                                        | *     | *     |
| Chee, et al. [86]    | 2018  | Investigation of the effects of orientation on freeze/thawed polyvinyl alcohol hydrogel properties | PVA/CAF                                            | *     | *     |
| Tanriverdi, et al. [87] | 2018  | Preparation and In-vitro evaluation of melatonin loaded HA/PVA gel formulation | HA/PVA/M                                           |       | *     |
| Long, et al. [88]    | 2019  | Development of a long-term drug delivery system with levonorgestrel-loaded chitosan microspheres embedded in poly(vinyl alcohol) hydrogel | PVA/CS-LNG                                         | *     |       |
| Ullah, et al. [89]   | 2019  | Natural and synthetic materials based CMCh/PVA hydrogels for oxaliplatin delivery: Fabrication, characterization, In-vitro and In-vivo safety profiling | CMCh/PVA                                           | *     | *     |
| Freitas, et al. [90] | 2020  | Investigation on miscibility, thermal, crystallographic diffraction and dynamic-mechanical properties of poly(vinyl alcohol)/poly(vinylpyrrolidone)/zirconium phosphate nanocomposites | PVA/PVP/ZrP                                        | *     | *     |
### Table 1.b Summary of article results obtained with SLR method

| Authors                  | Years | Titles                                                                 | Materials          | $T_g$ | $T_m$ |
|--------------------------|-------|------------------------------------------------------------------------|--------------------|-------|-------|
| Zulfiqar, et al. [91]    | 2020  | Efficient removal of Pb(II) from aqueous solutions by using oil palm-bio-waste/MWCNTs reinforced PVA hydrogel composites: Kinetic, isotherm and thermodynamic modeling | OPB/PVA/MWCNTs     |       | *     |
| Reena, et al. [92]       | 2020  | Synthesis and characterization of cross-linked hydrogels using polyvinyl alcohol and polyvinyl pyrrolidone and their blend for water shut-off treatments | PVA/PVP            |       | *     |
| Croitoru, et al. [82]    | 2020  | Physically crosslinked poly (vinyl alcohol)/Kappa-carrageenan hydrogels: Structure and applications | PVA/CAR            |       | *     |
| Sirousazar and Khodamoradi. [93] | 2020  | Freeze-thawed humic acid/polyvinyl alcohol supramolecular hydrogel       | HA/PVA             |       | *     |
In general, the presence of a single peak $T_g$ for a polymeric blend in the DSC thermograms can be connected to good impermissibility and contact between the constituents [94]. However, in this case, a single value for $T_g$ has not been associated with the formation by hydrogen bonding of inter-polymer complexes since k-carrageenan shows no presence of this type of transformation. The $T_g$ may be attributed to a peak at ~75 °C for PVA hydrogel, following the literature's values [95]. The combined plasticizing effect of k-carrageenan and water induces a decrease in $T_g$ values for the K/PVA mixture relative to PVA. While in the 100-150 °C interval, broad endotherm peaks appear, which could be due to water vaporization [82].

Table 2. Formulation of samples for PC2, PCC2, PCH2, and PCHC2 [84]

| Formulation | PVA (%) | CaCl₂ (%) | Chit-Ma (%) | Honey (%) |
|-------------|---------|-----------|-------------|-----------|
| PCHC2       | 98.50   | 0.10      | 0.70        | 0.70      |
| PHC2        | 99.20   | 0.10      | 0.00        | 0.70      |
| PCC2        | 99.20   | 0.10      | 0.70        | 0.00      |
| PC2         | 99.90   | 0.10      | 0.00        | 0.00      |

Khaleghi et al. [84] successfully compared the effect of Chit-MA with honey using parameters of $T_m$ and degree of crystallinity. The DSC curves of PC2, PCC2, PHC2, and PCHC2 are shown in Figure 4; and, the formulation of the sample can be seen in Table 2. According to them, honey added to PVA increased the melting temperature and decreased the degree of crystallinity significantly compared to the addition of Chit-MA. This difference is possibly due to the mutual affinity of hydroxyl-rich surfaces between PVA and honey, leading to increased hydrogen bonds affecting the molecular mobility of the PVA chains and the matrix's crystallinity behaviour [84], [96].

![Figure 5. DSC thermograms of pure PVA (SH0) and typical HA/PVA supramolecular hydrogels loaded with 6 and 9 wt.% HA (i.e., SH6 and SH9) [93]](image)

$T_g$ is correlated with changes in the PVA amorphous region and is dependent on the mobility and free rotation of its chains, and $T_m$ is determined by the crystalline region transition [96]. In comparison with the PC2 study, the $T_g$ of both systems increased towards higher temperatures. Strong interactions between honey and Chit-MA with the PVA matrices that limited the free rotation of the PVA chains in the interfacial zone could be due to this. PCHC2 showed the highest $T_g$ compared to the other samples that could be due to Chit-MA and honey's synergic effect. Compared with the other samples, PCHC2 displayed the highest $T_g$, which may be attributed to the synergistic effect of Chit-MA and honey together.
In the latest report, Sirousazar and Khodamoradi [93] succeeded in observing the effect of humic nanoparticles (HA) loaded with PVA supramolecular hydrogels based on the \( T_g \) value. The increased melting point of PVA supramolecular hydrogels loaded with humic acid nanoparticles (HA) could be attributed to their increased gel fraction values and, because of the presence of HA, formed more crosslinking zones within the supramolecular hydrogels. Besides, samples showed broad and shoulder-like endothermic peaks around 40–55 °C, which may be due to the transition of samples to the glassy-rubbery phase. As the peaks were very weak and broad, the \( T_g \) obtained from DSC thermograms (Figure 5) would not be accurate and consistent for the samples examined. Therefore, they propose that the exact values of \( T_g \) should be recorded based on the results of the dynamic mechanical-thermal analysis (DMTA) as their solution.

4. Conclusion

PVA-based hydrogel has several thermal properties obtained from DSC measurements. In this SLR study, we found thermal properties parameters usually analyzed on PVA-based hydrogel, which is more focused on \( T_m \) and \( T_g \) values. However, not all of the data obtained have a good thermogram curve and can be analyzed. Finally, we recommend taking measurements using DMTA; it has a higher degree of accuracy for analyzing \( T_g \). We hope that this study will provide challenges and opportunities to deepen the analysis of thermal properties using DSC measurements, and the thermogram curve can be used as one of the primary data in future research.

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