



EFFECT OF ADDITIVES ON THE T_g , T_p AND GELATION DEGREE OF THE PVC PRODUCTS

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ABSTRACT

This paper aims to study the influence of additives on the thermal behaviour of polyvinyl chloride (PVC) products depending on using Differential Scanning Calorimetry (DSC). DSC was used to determine glass transition temperature (T_g), maximum processing temperature (T_p) and degree of gelation of PVC products. PVC products used in this paper include PVC powder standard, PVC drinking water pipe, PVC draining water pipe, USA PVC adhesive, commercial PVC adhesive and USA CPVC adhesive. The results show that higher T_g and processing temperature (T_p) are observed in PVC pipes. While, greater degree of gelation are seen in PVC adhesive.

Key words: PVC Pipes, PVC Adhesives, Degree of Gelation, Glass Transition Temperature, Processing Temperature.

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1. INTRODUCTION

Polyvinylchloride (PVC) products include pipes, windows, electrical insulator, adhesives, and other applications. Hard PVC (PVC-U) is used to produce water pipes [1]. 50% of water, wastewater, and gas pipes that are used in Europe are made of PVC-U [2,3]. PVC powder is mixed with additives (plasticizers) and solvent in order to produce PVC cement adhesives that represents flexible adhesives. In contrast, PVC powder is mixed with additives (stabilizers) with high temperature higher than 200°C using extruder to form different products like pipes or window frames. On cooling, a network consisting of entangled molecules and newly arranged, small crystalline domains, is obtained (Figure 1) [4].

Mechanical stability and fracture behaviour of PVC products are affected by the processing temperature (T_p) and the degree of gelation. Various test methods such as those involving solubility, hardness and other mechanical characteristics as well as optical microscopy are used to determine them. These methods often require time consuming sample preparation or need larger samples. For this reason, DSC has been used for many years to investigate the melting behaviour of PVC [5,6,7,8]. The degree of gelation is defined as the ratio of the amount of small, secondary crystallites (melting point below T_p) and the amount of large primary crystallites (melting points above T_p). The content of crystallites is

determined from the size of the corresponding enthalpies of fusion using DSC [4,10,11]. Little crystallites with a melting point around T_p are seen as melting gap in the DSC curves because of the influence of various recrystallization processes [9,12,13,14]. The degree of gelation considers an influential parameter that affects the stability and growth of crazes as well as the impact resistance. PVC products such as pipes that have a lower gelation degree are subjected to failure under dynamic loading due to drilling new connections and traffic loads [2,15, 16, 17,18] . X-ray diffraction is utilised to measure gelation [14,19].

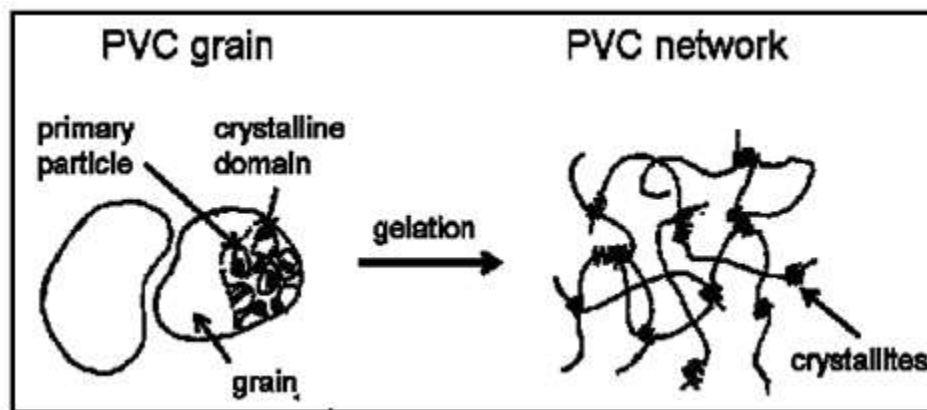


Figure 1: Schematic diagram illustrating the gelation process

DSC is now a well-established and simple test method for quality control. The ISO 18373 standard covers DSC methods for testing PVC pipes [9,20,21]. ISO 18373-1 describes the measurement of the processing temperature as the extrapolated peak temperature (B-onset). The ISO 18373-2 describes the measurement of the enthalpy of fusion of the small crystallites, the peak area A (the A endotherm). This is easily determined and is a measure of quality for the amount of small crystallites and hence indirectly for the degree of gelation.

These methods can also be used to test other PVC-U products. The following experiments describe the analysis of PVC products according to ISO 18373 Parts 1 and 2 to measure the maximum processing temperature and the crystallite enthalpy of fusion. As using DSC to determinate T_g of PVC products depending on ASTM D 3418 and E 1356 standard [22,23] .

In a previous study , the researchers used DSC to determine the quality of PVC pipes [24] . Other researchers used DSC to determine degree of gelation of U-PVC [3,25], glass transition temperature of plasticized PVC [26], and degree of gelation of U-PVC [27,28].

2. EXPERIMENTAL SECTION

2.1. Materials

- PVC powder standard: without any additives, made in Egypt.
- PVC drinking water Pipe: rigid PVC, made in Egypt, size (1/2 in).
- PVC draining water Pipe: rigid PVC, made in Kingdom of Saudi Arabia, size (2 in).
- USA PVC adhesive: flexible PVC (9002-86-2) consists of tetrahydrofuran (109-99-9), methyl ethyl ketone (78-93-3), cyclohexanone (108-94-1) and acetone (67-64-1), made in USA.
- Commercial PVC adhesive: flexible PVC (686-48-82-8) consists of tetrahydrofuran (109-99-9) , methyl ethyl ketone (78-93-3) and cyclohexanone (108-94-1) commercial making.
- USA CPVC adhesive: flexible CPVC (7114-04) consists of tetrahydrofuran (109-99-9) , methyl ethyl ketone (78-93-3) , cyclohexanone (108-94-1) and acetone (67-64-1), made in USA.

2.2. Differential Scanning Calorimetry (DSC)

DSC model SHIMADZU DSC-60 (made in japan) was used to study the thermal transitions of the PVC products (PVC Standard , PVC drinking water pipe , PVC draining water pipe , USA PVC adhesive , commercial PVC adhesive and USA CPVC adhesive). DSC samples were prepared by cutting small PVC pieces using a sharp knife, then they were sealed into aluminium hermetic pans. The weight of the DSC samples were (10 ±0.5 mg). The temperature program specified in ISO 18373 is the same for Part 1 and Part 2 was between room temperature and 300°C using heating rate 10°C/min.

2.3. Degree of Gelation

The data was analysed using the software attached to the DSC. Gelation was then calculated using Eq. (1) :

$$\text{Gelation Degree (\%)} = (A / A + B) \times 100 \% \quad (1)$$

Where region A is taken after the T_g to the fusion temperature where the plot inflects. While, region B is taken from the inflection to under 300°C [4,24,29] .

3. RESULTS AND DISCUSSIONS

Figures (2 , 3 , 4 , 5 , 6 , 7) show the DSC curves of PVC products. The corresponding temperatures for the A-onset (first peak) and the B-onset (second peak) are entered in the diagram. In Table 1 and Figure 8, T_g of PVC products is 86.73°C for PVC powder standard, 127.31°C for PVC drinking water pipe, 105.82°C for PVC draining water pipe and below R.T. for USA PVC adhesive, commercial PVC adhesive and USA CPVC adhesive.

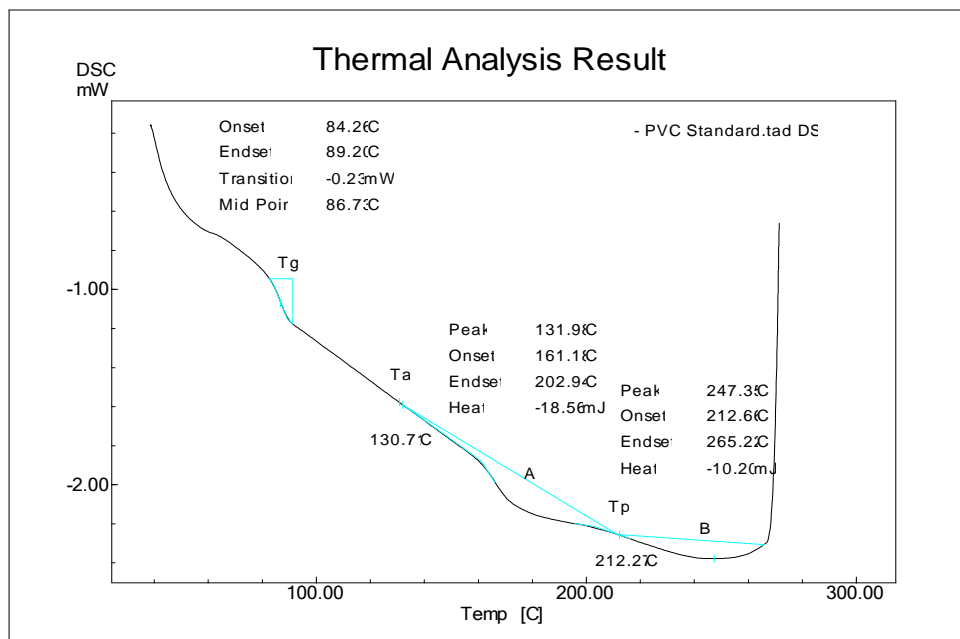


Figure 2 DSC curve of PVC powder standard

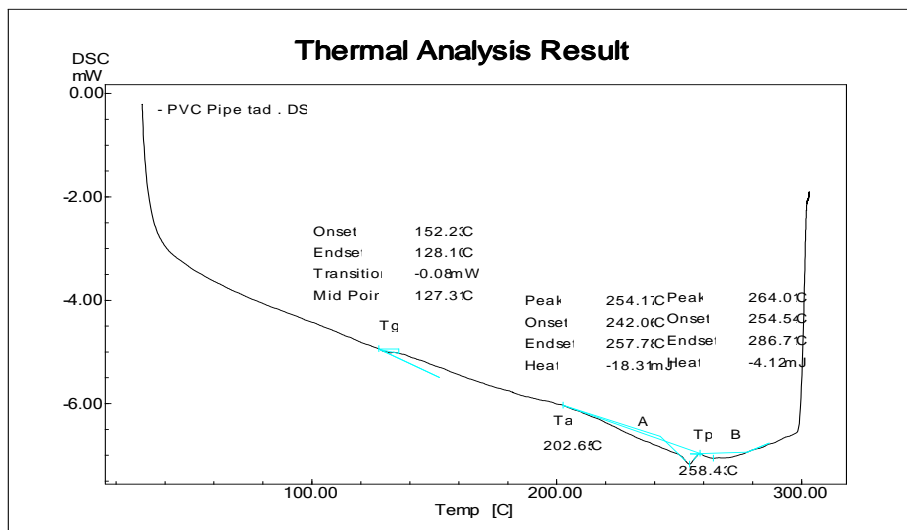


Figure 3 DSC curve of PVC drinking water pipe

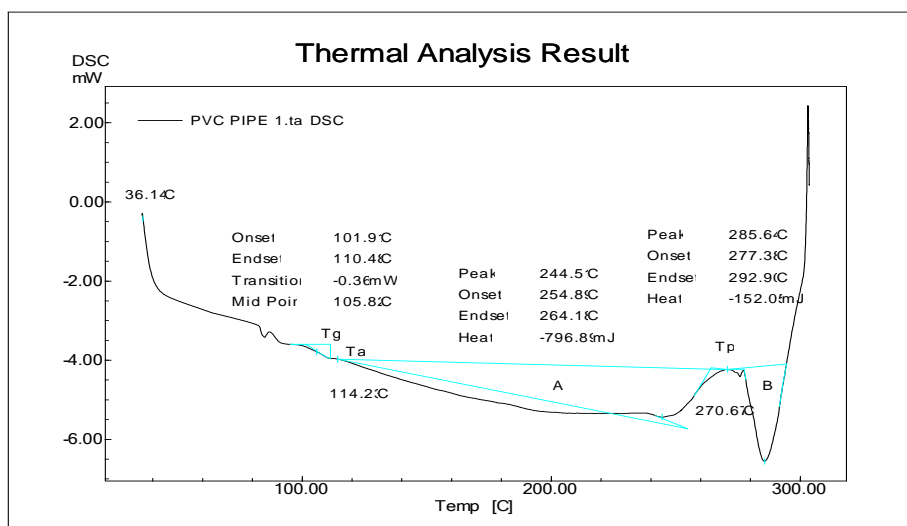


Figure 4 DSC curve of PVC draining water pipe

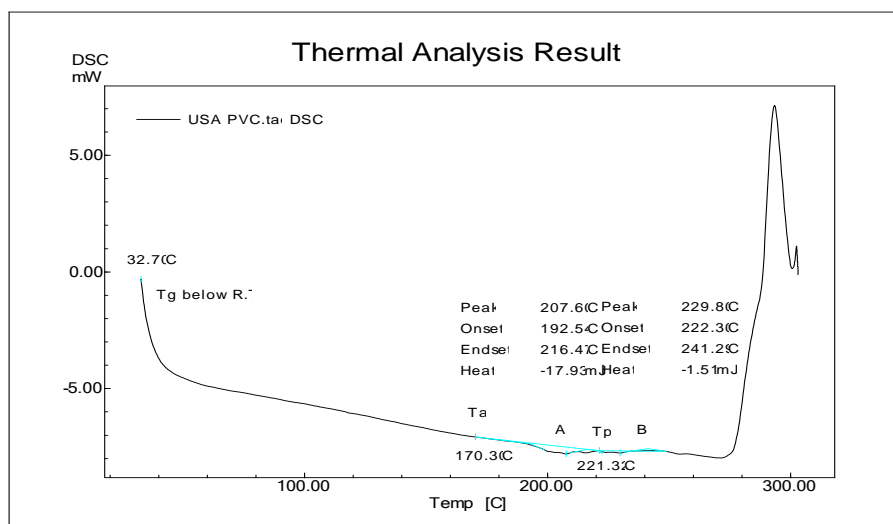


Figure 5 DSC curve of USA PVC adhesive

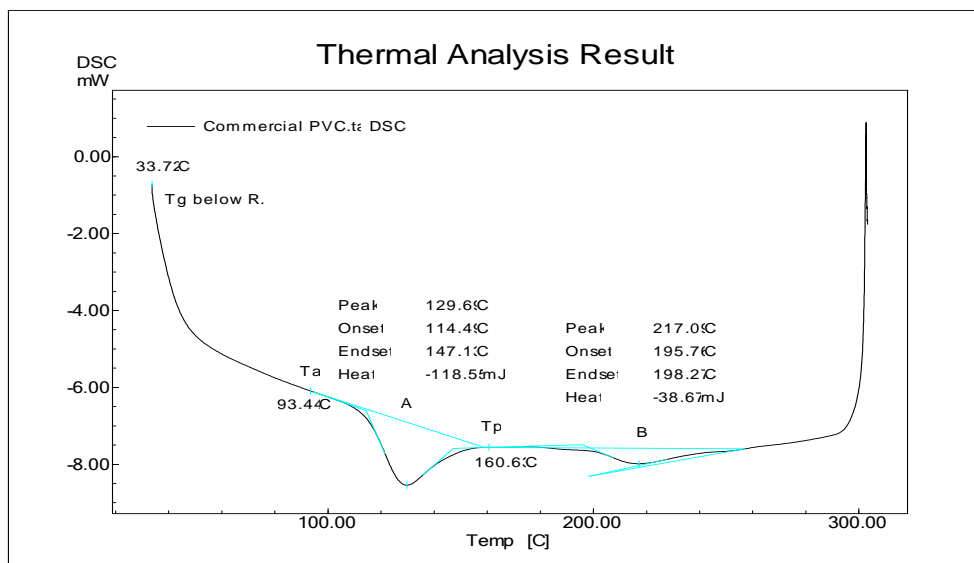


Figure 6: DSC curve of commercial PVC adhesive

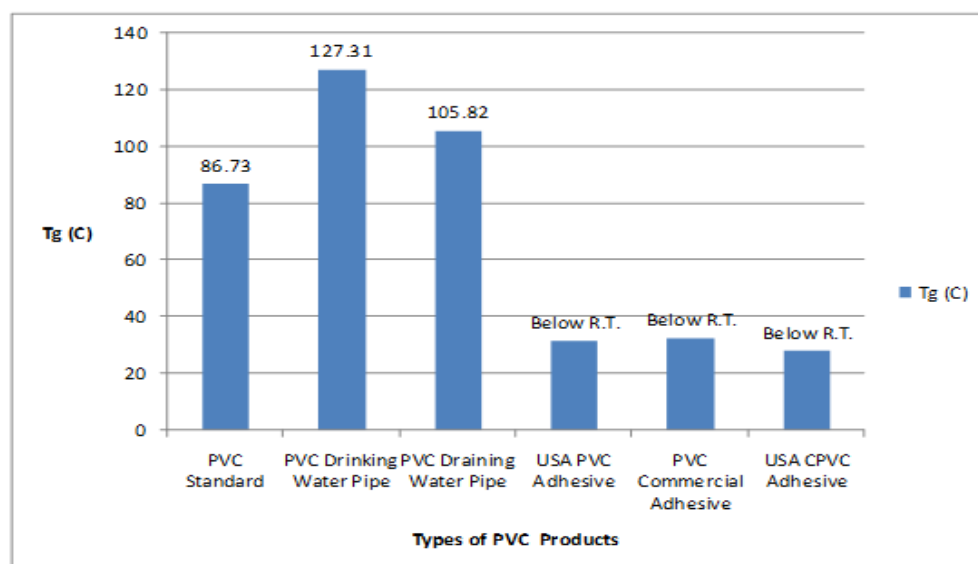


Table 1: T_g, T_a, T_p and degree of gelation of different PVC products .

Types of PVC	T _g (°C)	T _a (°C)	Area A (mJ)	T _p (°C)	Area B (mJ)	Degree of Gelation (%)
PVC powder Standard	86.73	130.71	18.56	212.27	10.2	64.53
PVC Drinking Water Pipe	127.31	202.65	18.31	258.43	4.12	81.63
PVC Draining Water Pipe	105.82	114.23	796.89	270.67	152.05	83.98
USA PVC Adhesive	Below R.T.	170.3	17.93	221.32	1.51	92.23
Commercial PVC Adhesive	Below R.T.	93.44	118.55	160.63	38.67	75.4
USA CPVC Adhesive	Below R.T.	127.5	143.69	244.85	1.55	98.93

Table 1 and Figure 9 display the maximum processing temperature (T_p) of PVC Products. The results show that the maximum processing temperature of (PVC powder standard at 212.27°C), (PVC drinking water pipe at 258.43°C), (PVC draining water pipe at 270.67°C), (USA PVC adhesive at 221.32°C), (commercial PVC adhesive at 160.63°C) and (USA CPVC adhesive 244.85°C) .

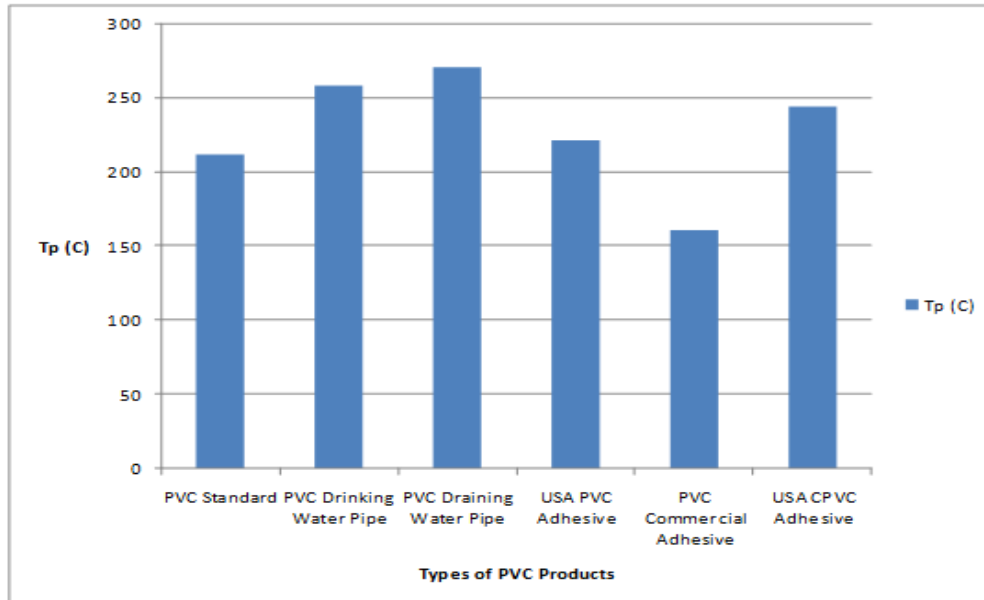


Figure 9: Maximum processing temperature of PVC products

The degree of gelation of PVC Products is presented in Table 1 and Figure 10. The results show that the degree of gelation (%) of (PVC powder standard about 64.53%), (PVC drinking water pipe about 81.63%), (PVC draining water pipe about 83.98%), (USA PVC adhesive about 92.23%), (commercial PVC adhesive about 75.4%) and (USA CPVC adhesive about 98.93%).

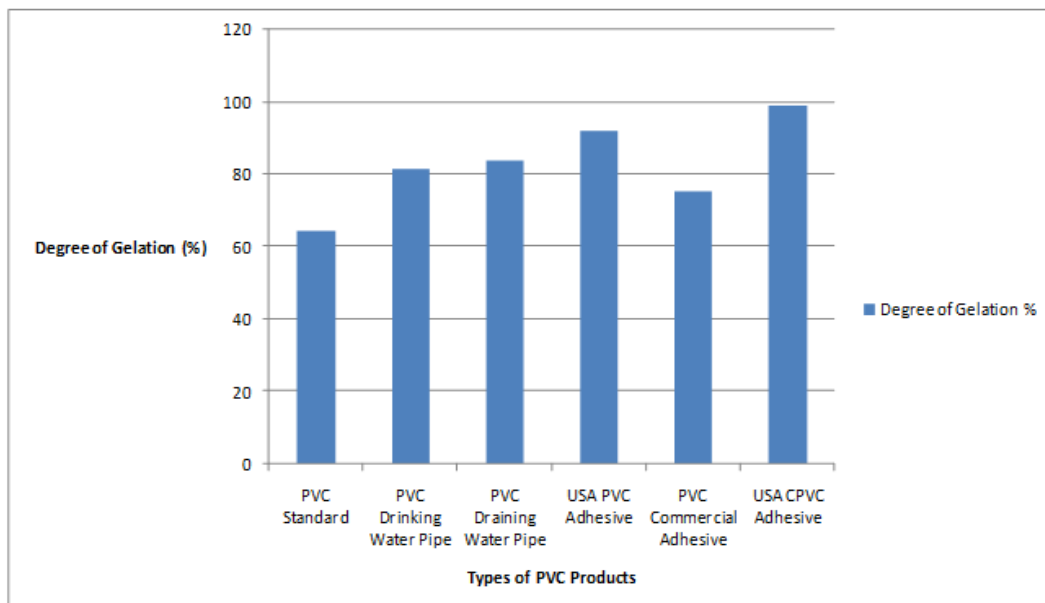


Figure 10 Degree of gelation of PVC products

PVC pipes show higher T_g and T_p than PVC powder standard. This is attributed to stabilizers additives were added into PVC raw materials that were used to produce PVC pipes, and these additives shift the T_g and T_p to higher temperature in comparison with T_g and T_p of PVC powder standard. PVC adhesives display greater degree of gelation and lower T_g as compared with PVC powder standard. This is because plasticizers additives were added to PVC adhesives leading to reduce the T_g and increase the degree of gelation [2,26,30].

4. CONCLUSIONS

PVC pipes with stabilizers display higher T_g and T_p than other PVC products. While, PVC adhesives with plasticizers show greater degree of gelation and lower T_g than other PVC products.

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