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Study Of Thermal Properties In Commonly Used Fabrics Using Differential Scanning Calorimetry (DSC) - A Forensic Perspicacity

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Abstract:- Ignition or heating behavior of six different types of cloth material samples of different manufacturers in India was examined by quantitative thermal analysis method i.e. 'differential scanning calorimetry (DSC)' to find out if this parameter could be used in differentiating between these cloth samples and comparison of their tendency of self heating possibility for forensic purpose. This study also focuses on giving a solution using the thermal analysis for analyzing the spontaneous fire accidents and for possible application in forensic study of fire cases. The results indicate that the transition heating temperature may serve as the useful criteria for differentiating different cloth samples from different sources. Moreover, thermogram of DSC curve can be used as fingerprint to identify cloth material and presence of external material which may cause the self ignition in fabric material. Thus, the cause of fire accident can be investigated.

Key words: Thermal Analysis, Fire Investigations, Forensic Science, Thermodynamics.

INTRODUCTION

Forensic Science laboratories used to work with little amount of exhibit/samples collected by investigation personal from crime scene to determine the type of material and its possible manufacturer for investigatory and evidence purpose, an example of it would be the characterization of polymers-plastics, cloth materials etc. Such materials recovered from crime scenes of high destruction, such as fires or explosives, are often not in a state of conductive to a chemical analysis. Fibers and cloth materials are useful for forensic purposes, as they tend to cling easily and provide useful characteristics for identification purposes. The present paper focuses on the most useful and effective solution for analyzing and evaluating the cause of spontaneous fires, assessing the fire using thermo analytical methods and the contemporary studies in this area for application in forensic fire accident examination.

In most of the Fire Accident cases self heating can be one of the cause. Self heating also often called as spontaneous combustion or spontaneous ignition. Self heating normally caused by exothermic chemical reactions or biological process. These are encountered especially in the fire accidents in industries and agricultural areas including forest fires. For the chemical reaction leading to a self heating process the theory of thermal explosion has been developed [1]. Among the methods that have been used in forensic fire investigation is Mackey test [2]. Which allows relatively rough statements and limited to certain classes of substances. To overcome and face such situation the forensic scientists are presently using thermal or thermo analytical methods [2-4]. From a forensic point of view thermal analysis, in particular Differential Scanning Calorimetry (DSC), is useful for characterizing polymers, fibers and highly self ignition or fire stimulating materials traces etc. [2-4]. Because in it the required sample size is relatively small and very little sample preparation is required which translate to fast analysis time.

DSC is a technique that measures heat flow into or out of a material as a function of time or temperature. In a DSC measurement, information about thermal and mechanical history (processing influences, crystallinity and curing, service temperature) is revealed by the first heating curve. For forensic comparison of chemically similar samples thermal history plays an important role because subsequent controlled cooling creates a "new" known specimen history, which gives same characteristic properties to all materials. In the present paper, peak melting temperature (temperature of peak maximum) and area and delta H of the samples are recorded from the first heating curve to study Fabric or cloth material.

MATERIAL AND METHODS:

Methods generally used to study self ignition are based on thermal analysis. Thermal analysis involves a large array of calorimetric methods, which usually require very small sample size and can also be used for unknown sample analysis. In the present study Heat flow Differential scanning calorimetry (DSC) of make PerkinElmer and model no Pyris-1 Instrument was used for examination of cloth/Fabric samples.

Differential Scanning Calorimetry (DSC):

Differential Scanning Calorimetry, or DSC, is a thermal analysis technique that looks at how a material's heat capacity (Cp) is changed by temperature. When a sample shows a transition its heat capacity changes and this is recorded as a change in

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the heat flow. This heat flow allows the detection of phase transitions such as melting, glass transitions, phase changes and cross linking. Sample compositional analysis and Effect of additives can also be examined by this method.

Semi-Crystalline material and their critical thermo-physical properties like; melting temperature, heat of melting, percent of crystallinity, Glass Transition temperature and processing temperature can be studied using DSC. In experimentation using DSC, the sample is put in crimped aluminum pan and the reference (usually an empty pan) sits on raised platform on the sensors. As heat is transferred through the sensor, the differential heat flow to the sample and reference is monitored by present area thermocouples. A thermocouple monitors sample temperature. A preheated purge gas (i.e. Nitrogen) is present to provide additional baseline stability as well as the desired sample/atmosphere interaction.

Polymers are potent source of fuel for fire and have inherent inflammability. Most Fabrics Ignite and continue to burn even after the source of ignition is removed. To utilize and improving the flame resistant clothing DSC is used for characterization of different fibers of the fabrics and textile. A typical DSC curve while heating Synthetic Fibers is shown in

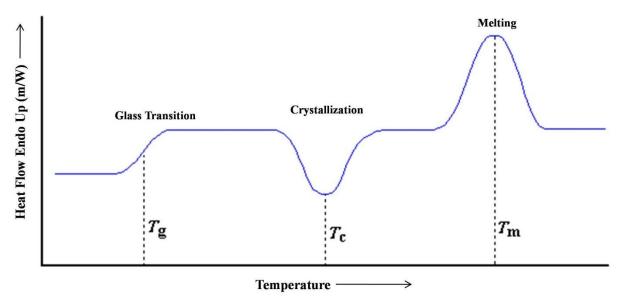


Figure-1: Illustration of different thermal events in a typical DSC curve

To understand the behavior of DSC curve and for its proper evaluation it is Important to know the various thermal properties and their correlation, these important formulas are as follows:

$$\Delta H = \int C_p \ dT$$

ΔH - the change of enthalpy between two states, Cp - specific heat capacity - the quantity of energy needed to change the temperature of 1g of material by 1°C at constant pressure and T - temperature

Here, Cp is hard to measure thus Heat flux calculations is used in place of Cp as it is correlated as follows:

$$C_p = \frac{Q}{\alpha \ m}$$

 $C_p = \frac{Q}{\alpha \ m}$ Q - heat flux - the quantity of heat transferred per unit time and mass

 α - heating rate and m- total mass of substance

$$\Delta H = \int \frac{Q}{\alpha \, m} \, dT = \frac{F}{\alpha \, m}$$

here F- represents the total area under transition peak.

In the present study six samples of commonly used Fabric or cloth material, manufactured and supplied in India, have been used i.e. Nylon, Wool, Rayon, Polyester, Teri cotton and cotton. These samples of cloths are analyzed over the temperature range from 25°C upto max 350°C. A heating rate of 15°C/min and 20°C/min was used with nitrogen atmosphere around the sample.

RESULTS AND DISCUSSIONS

Displayed in Figure 2(a) are the DSC results obtained on the first cloth material sample i.e. for Nylon. The plot shows the heat flow as a function of sample temperature and an endothermic response is oriented upwards. The cloth sample provides a substantial amount of characterization information with the DSC, a melting peak is obtained at 220.93 °C with a heat of melting of 97.92 J/g. Additional endothermic peaks are observed at 194 and 215 °C, and these are most likely due to processing or heat-setting. Similarly, for figure 2(b) DSC curve results show that the this sample i.e. wool cloth material has a melting peak at 130.13 °C, with a heat of melting of 197.98 J/g.

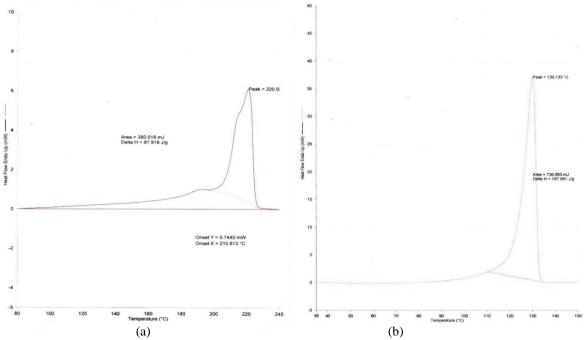


Figure-2. shows DSC curve obtained from experimental studies done using different type of cloth samples i.e. for (a) Nylon, (b) Wool.

In Figure 3 (a) DSC curve results show that the rayon cloth material has a melting peak at 250.66 $^{\circ}$ C, with a heat of melting of 78.68 J/g. Whereas, in Fig 3 (b) The pseudo-endothermic event observed at 96.44 $^{\circ}$ C in thermogram is due to a volume relaxation phenomenon occurring at the glass transition temperature (T_g) of the polyethylene Terephthalate (PET). This is the result of a physical aging process which gives rise to an ordering of the amorphous molecules when the material is stored for extended periods at temperatures below T_g . The melting temperature of polyester is 252.29 $^{\circ}$ C and the enthalpy required to melt this polyester fiber was 23.10 J/g.

DSC results obtained when Teri cotton and Cotton cloth fabric sample was loaded is displayed in figure 4 (a) and 4 (b). From these thermogram we can differentiate the properties of these fabric and observed a melting peak of tericotton at 109.44° C temperature with a heat of 98.31 J/g, whereas for Cotton sample its temperature of melting is 111.16° C with the heat of 120.23 J/g.

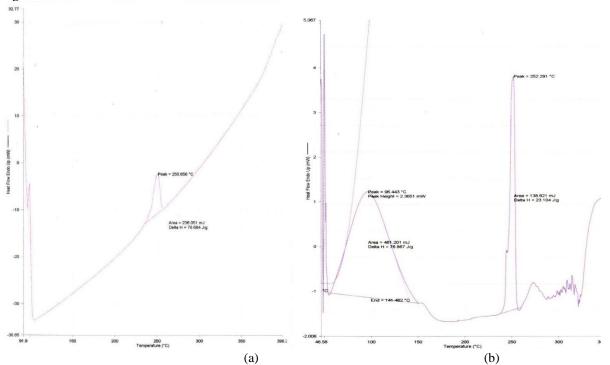


Figure-3. shows DSC curve obtained from experimental studies done using two different type of cloth samples i.e. for (a) Rayon, (b) Polyester

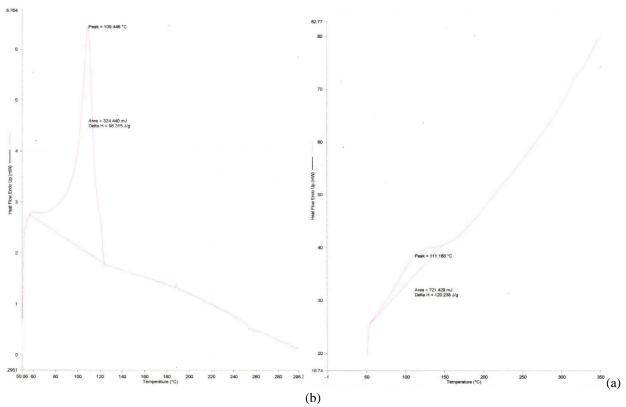
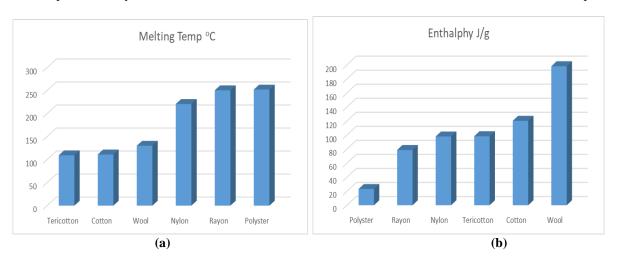


Figure-4. shows DSC curve obtained from experimental studies done using two different type of cloth samples i.e. for (a) Teri cotton, (b) Cotton.

| Table.1 | ·Analy | sis of | DSC | curve | and | details |
|---------|--------|--------|-----|-------|-----|---------|
| | | | | | | |

| S.no. | Cloth material | DSC curve properties | | | | | |
|-------|----------------|----------------------|------------------------|---------------|------------------------|--|--|
| | name | Area (mJ) | Peak Melting Temp (°C) | Delta H (J/g) | Heat Flow at Peak (mW) | | |
| 1. | Nylon | 380.018 | 220.93 | 97.918 | 6 | | |
| 2. | Wool | 739.063 | 130.133 | 197.981 | 37 | | |
| 3. | Rayon | 236.051 | 250.656 | 78.684 | -3 | | |
| 4. | Polyester | 138.621 | 252.291 | 23.104 | 3.75 | | |
| 5. | Teri cotton | 324.440 | 109.446 | 98.315 | 6.5 | | |
| 6. | Cotton | 721.429 | 111.168 | 120.238 | 40 | | |

From figure 2, 3 & 4 it is clear that different fabric material got burned by absorbing a particular amount of heat at particular temperature. From these DSC results for pure cloth material fiber it is clear that Self heating in these cloth material is rare at measured temperature ranges, as no any exothermic peaks are visible at given temperature range. For detailed analysis and comparative study of their thermal behavior, results for these cloth material is tabulated in Table 1 and plotted in Figure 5.



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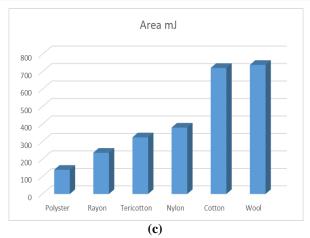


Figure-5. comparison of all cloth sample on the basis of their (a) melting point, (b) corresponding heat enthalpy and (c) area under peak to compare their tendency of ignition and heat energy required.

Identification of Fabric materials and comment on their tendency of spontaneous ignition:

As per comparative chart shown in Figure.5 we can conclude that Polyester needed minimum heat energy and Heat flux for transition due to which it generally tends to melt fast when external ignition is provided but its Transition melting temperature is maximum among samples which makes it very bad as a self heating cloth material. Whereas Cotton and wool is tends to burn at low temperature thus may catch fire or tends to self heating when surrounding temperature is slightly higher than 100°C but the heat enthalpy and Heat flux required to initiate transition is high as compared to other samples.

Thus from the present study we can conclude that the particular thermal characteristics from these cloth sample specimen can be used for recognizing the particular cloth material condition found at the fire accident cases. The observed thermal properties of these cloth samples are valuable for forensic purposes since they can provide a 'fingerprint' of the material which can aid in tracking down the particular manufacturer of any fabric yarn like for shirt, curtain, carpet and bed sheet etc..It can be seen in Table 1 and Figure 5 (c) that for fabric of a different materials the values of peak melting temperature are different.

As compared to other cloth materials discussed here, high cellulose content makes cotton particularly most liable to catch fire through external ignition. Therefore, while investigating fire accident cases we should check around the possibility of presence of sparks, fire, naked lights and lit cigarettes traces etc. In addition to external ignition, cotton and other cloth materials are also liable to thermal, chemical and microbial self-heating/spontaneous combustion. Self-heating/spontaneous combustion arises as a result of moisture, fats/oils, due to the action of acids, such as nitric or sulfuric acid, and through contact with oxidizing agents and with goods with a tendency to self-heating. Thus, if from cloth samples received from fire accident crime scene these type of traces of self-heating material is resent then it can be checked by DSC curve results comparison. As it may help in assessing to investigate the possibility of an ignition.

CONCLUSION

This study clearly shows that most of the cloth material i.e. fabric used for daily wear cloth, home decor and for furniture are have very rare tendency of self heating as per experimental results shown in this paper. But in the presence of external ignition stimulation material these material may tends to caught by fire. Whenever, there is a case of fire accident or burned body etc. the collection of cloth pieces found at that place and from burned or buried body can leads to important clue regarding investigation cause of accident or murder. As examination of such cloth material using DSC can be used as fingerprint for cloth material and presence of external material can be identified due to which self ignition or deliberately ignition was caused.

In the present study we have used pure cloth material samples of six different fabric to investigate their self heating tendency. As a future extension of this work lab prepared samples of cloth materials soaked in flammable material in burned, half burned and unburned state can be tested. This study will help to clearly differentiate between fingerprint region of cloth material and its thermal behavior in presence of external material and their traces.

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