Analysis of Toner Powder Using Different Techniques

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Abstract: In the present article, analysis of toner powder used in the printer has been undertaken. Toner is basically mixture with thermoplastic base material. Thermal analysis of toner is undertaken with differential scanning calorimetry (DSC), glass transition happens at or near 71°C. The annealing of the prepared sample at three different temperatures-25°C,50°C, 100°C, introduces variation in the crystallite size of the toner particle, studied in the XRD characterisation. UV spectroscopy graph shows the absorption of light in the UV range of the toner powder sample. FTIR spectroscopy confirms the functional information of the toner powder indicating presence of compounds like alkenes, aliphatic ethers and oximes.

***Index terms*:** Toner powder, XRD, UV spectroscopy, FTIR, DSC.

I. Introduction

Toners are black, fine powders which are the mixture of thermoplastic base that contain pigments, UV stabilizers, polymer pigment, magnetite, and other additives Most of the high-speed digital printers use toner particles which are mixed with magnetic carrier particles. Magnetic carrier particles can be either Ferrite or Magnetite.[1]. Magnetite is one of the main components of toner powder which helps in tribocharging property of the toner particles.

With increasing demands that are being placed on higher image quality and lower cost for printing, improvements in printer capability and reliability are being continuously explored The image quality and cost benefits, which once was only possible with offset printing, are being increasingly realized with new digital printers.[2] For better performance research is concentrated on the enhanced performance of toner particles, so it becomes imperative to understand complete characterisation of the toner.

It is an imaging technology that takes a digital file and prints the output using photoreceptor, light source, electrostatic principle and toner as well. Toner particles move between the components through static electricity. [3] Printing devices nowadays uses a laser beam which after reflection on a polygon mirror, itches the optical photosensitive roller to cast the image. The charged toner particles are electrostatically attracted to the oppositely charged optical photosensitive roller. Each toner particle is designed in such a way to hold a specific charge and they travel with extreme precision. A stronger transfer charge attracts toner particles from the drum and then onto the paper. Pressure roller and fuser roller create an environment of heat and pressure for proper fusion of toner particles onto the paper to make the image permanent. [4]. The toner powder sample was studied using X-Ray diffraction, UV spectroscopy and FTIR spectroscopy.[5] [6][7][8][9].

II. Materials

Toner powder (Styrene Acrylate copolymer)

III. Characterizations

Crystal structure of the toner powder sample was analysed using an X ray powder diffractometer (Brukers X ray Diffraction D8 – discovered instrument) at room temperature with Cu K-α radiation $(\lambda = 1.5418\ \text{Å})$ Further, the toner sample was annealed at 50°C (and at 100°C) crushed and XRD pattern was observed. The crystallite size of the toner sample powder was calculated. UV spectroscopy (200- 800 nm) and FTIR Spectroscopy was performed to find out functional groups. Thermal characterisation was investigated using DSC at three different laboratories, IIT, ICT and Ramanathan Research Laboratory -Ruia College.

IV. Results and discussion:

X ray Diffraction (at 25°C-figure 1)

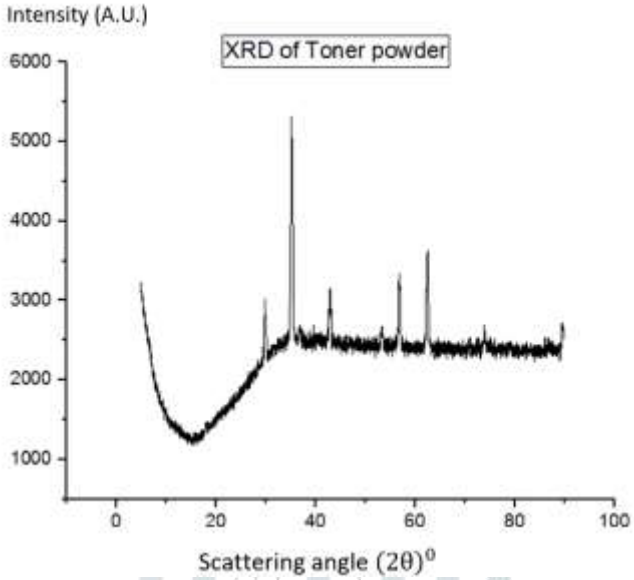

Figure 1: XRD pattern for toner powder sample at room temperature 25°C

The X-ray diffraction pattern confirms the formation of pure phase without any trace of impurity, also it well matches with the JCPDS card no 19-0629. The relatively broad peaks in the XRD pattern indicate the ultrafine nature and average small crystallite size [1]. The crystallite size 'D' of the toner powder was derived from Debye Scherrer equation:

Equation 1: Debye Scherrer equation: $\mathbf{D} = \frac{0.9\,\lambda}{\beta \cos\theta}$

Equation 2: Interplanar spacing: $\mathbf{d} = \frac{\lambda}{2\sin\theta}$

Further, the toner sample was annealed at 50°C (and at 100^0C) and crushed and the XRD pattern (figure 2 and 3) were studied. The crystallite size was once again calculated using Equation 1 and the interplanar spacing was calculated using Equation 2.

Table 1-Mean diameter and Interplanar spacing from XRD

Temperature (^{0}C)	Process	Mean diameter 'D'(nm)	Interplanar spacing 'd'(nm)
25	No action	0.355	0.1014
50	Annealing	0.465	0.1807
100	Annealing	0.403	0.1811

From the comparative figures in table-1, we observed that there is an increase in the interplanar spacing and the increase in the mean diameter when the sample is annealed at 50°C and at 100°C as compared to that of room temperature. With respect to change in temperature from 25^0C to 50^0C, from the calculation it seems that there is 31% increment in the crystallite size and 78 % increment in the interplanar spacing. With respect to change in temperature from 25^0C to 100^0C, calculations show that, there is 13.5 % increment in the crystallite size and 78.6 % increment in the interplanar spacing. It does point to the fact that crystallite size has first increased from 25^0C to 50^0C and then decreased by more than 13 % with reference to 50^0C size calculated, maintaining interplanar spacing. The increase in the crystallite size for 50° annealed sample indicates the increase in lattice strain of the sample.

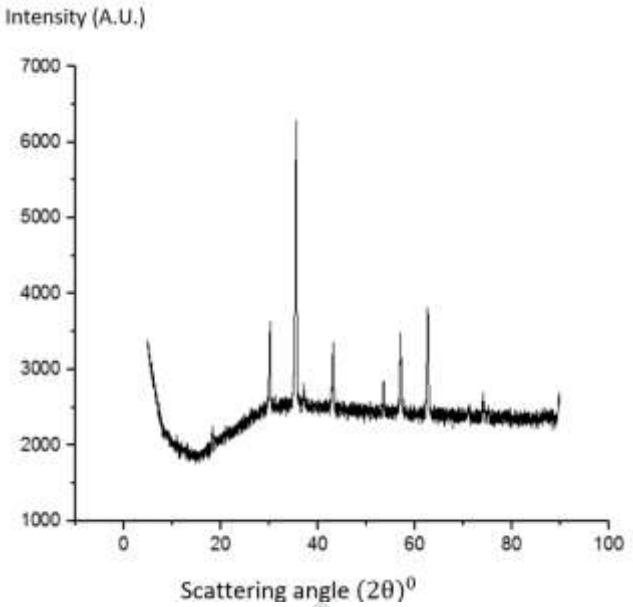

Figure 2: XRD pattern for toner powder sample annealed at 50°C

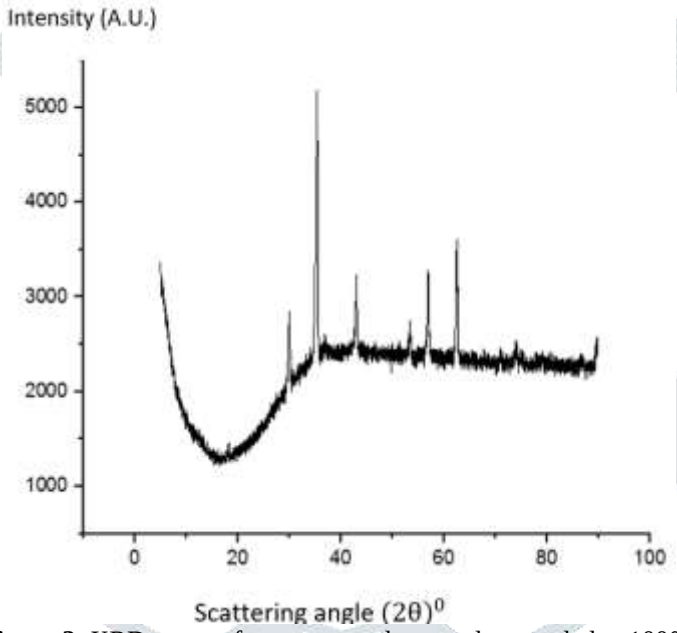

Figure 3: XRD pattern for toner powder sample annealed at 100°C

UV-Vis spectroscopy

UV spectroscopy graph shows the absorption of light in the UV range of the toner powder sample. In the region from 400 nm to 800 nm (figure 4), no peaks are observed which indicates that there is no absorption in the visible region of the spectrum. A sharp peak is observed at 221 nm with an absorption value of 3.52 (figure 5) This shows the presence of α, β- unsaturated ketones and an electronic transition: $\pi \longrightarrow \pi^*$ [10] UV spectroscopy showed the absorption of light in the UV region. Most of the absorption of light was observed in the 200-300nm range. There no transitions in the visible part of the spectrum. In the UV spectrum, there is a sharp peak at 221nm.

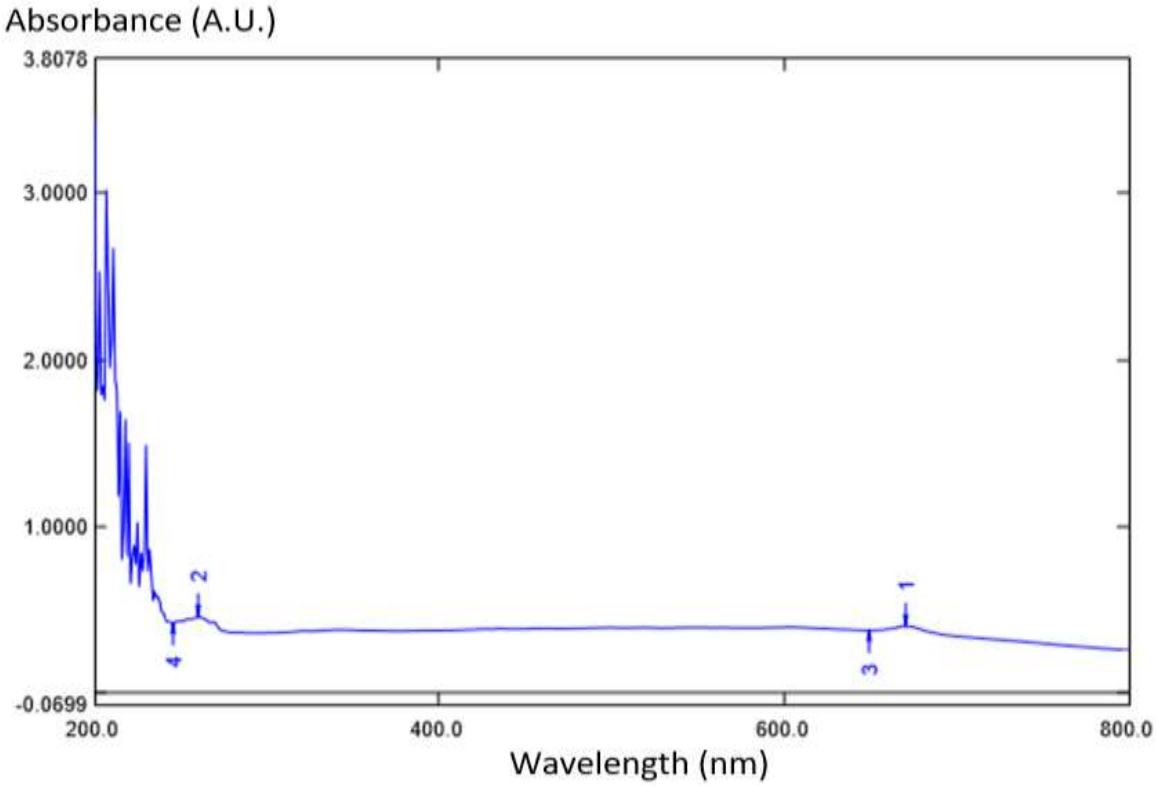

Figure 4: UV-Vis spectrum of toner sample powder at RT (wavelength-200nm-800nm)

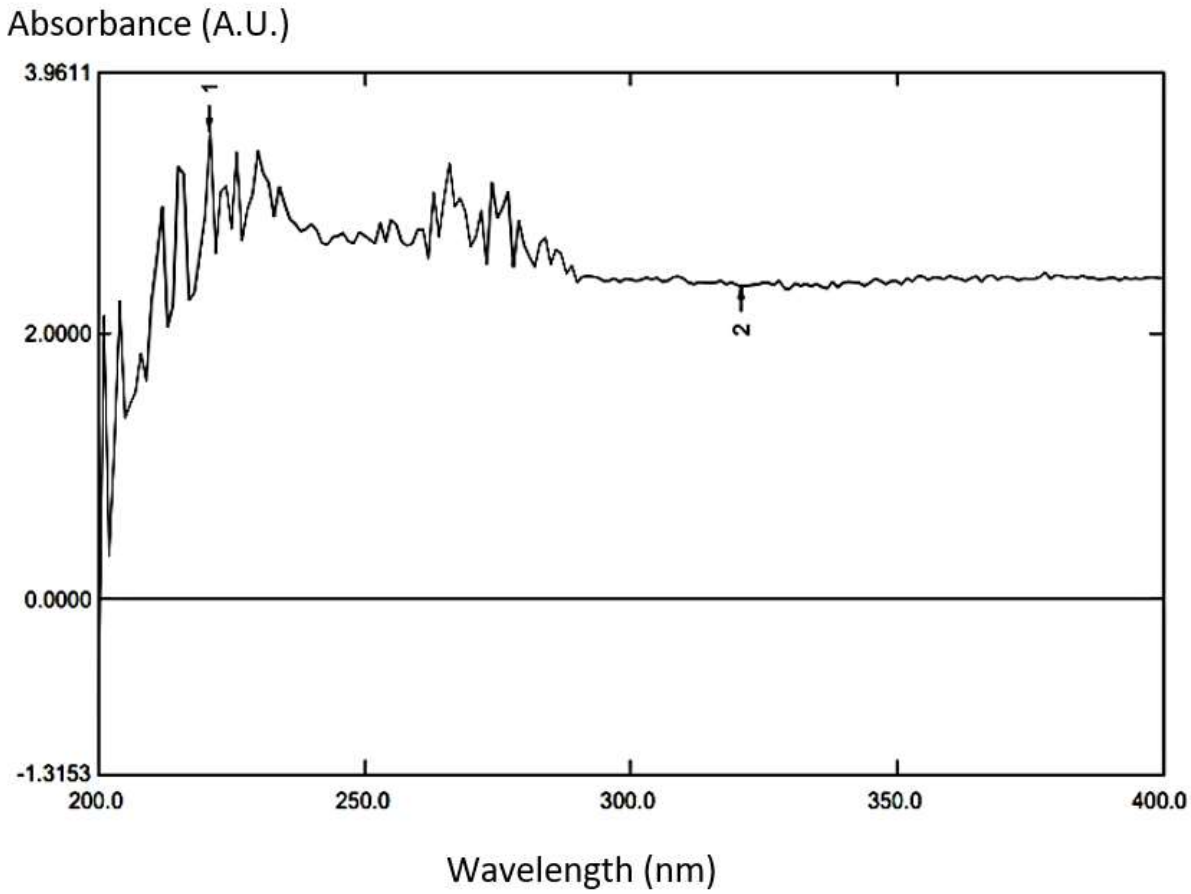

Figure 5: UV spectrum of the toner sample at RT (wavelength-200nm-400nm)

FTIR

FTIR spectroscopy of the toner powder sample was done and the following graph was obtained.

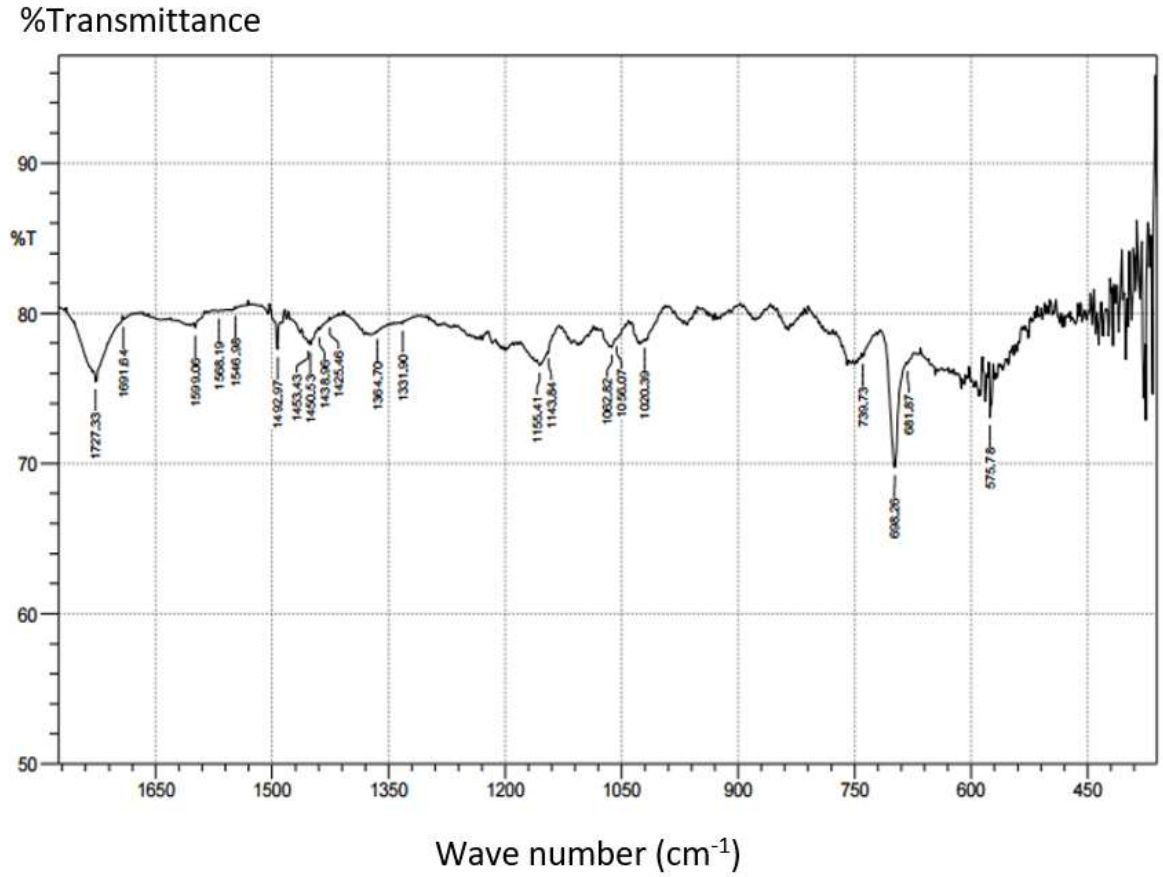

Figure 6: FTIR spectroscopy of the toner sample powder.

Table 2- Wave number and corresponding bond types, from FTIR characterisation.

Wave number(1/cm)	Bond type	Functional group
698.26	C=C	Alkenes
1143.84	C-O	Aliphatic ether
1691.64	C=N	Oxime

Prominent dips were observed (figure 6), which indicate typical bond types and functional groups, (table 2). With reference to functional groups indicated by FTIR characterization graph (figure 6), the presence of compounds like alkenes, aliphatic ethers and oximes, indicates that the toner powder is combustible. This is the reason that temperature plays an important role for a toner sample as it can be a potential hazard due to its combustible nature.[9]

Differential Scanning Calorimetry

Thermal characterisation was performed at ICT-laboratory, Mumbai. With reference to heating run of the toner sample (figure 7), two endothermic peaks are observed (at 42^0C and 72.38^0C). First peak represents moisture removal hence insignificant. Second endothermic peak represents the melting process which involves a phase change, the energy is absorbed by the intermolecular bonds which lead to loosening and breaking of these bonds. The second endothermic peak at 72.38^0C represents 10.367 mJ involved in the process. The energy absorbed by the toner powder is used in phase change at the temperature 72.38^0C. As the temperature rises further, it is clear that the toner powder releases energy. he general rising nature of the curve indicates that the material is under constant heat exchange behaviour. The enthalpy of fusion is 1.7728 J/g. Heat flow difference between the sample and the reference is positive because the heat flow to the sample is higher than that of the reference.

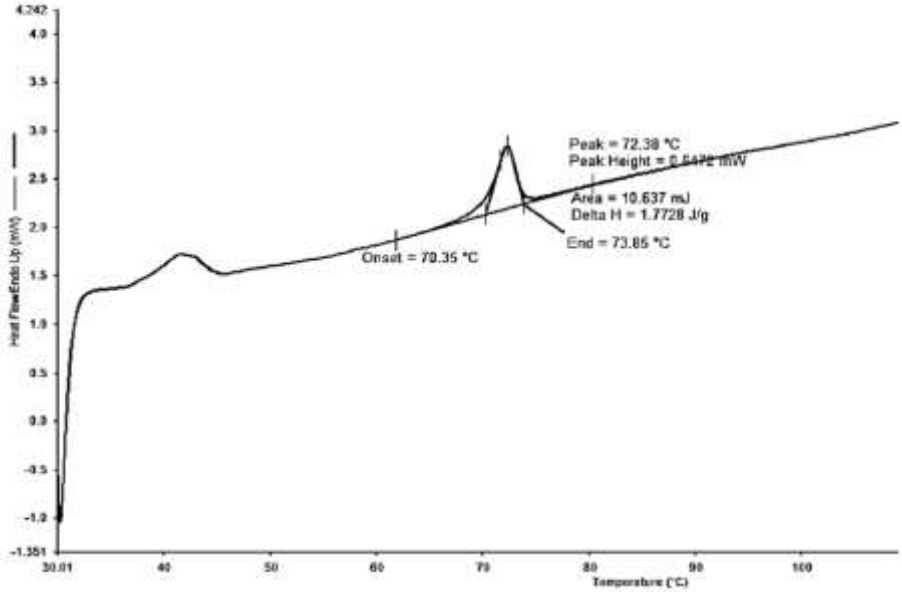

Figure 7: Heating run of the toner sample (ICT laboratory, Mumbai)

In the cooling run_(figure 8) represents exothermic peak and shows the crystallization process. During crystallization, the toner powder loses its random chain arrangement, intermolecular bonds get formed and the powder becomes ordered. The enthalpy change involved is negative 0.5627J/g and the energy involved in the crystallization process is -3.376 mJ. The onset temperature is 37.79^0 C which concludes at 34.40^0 C.

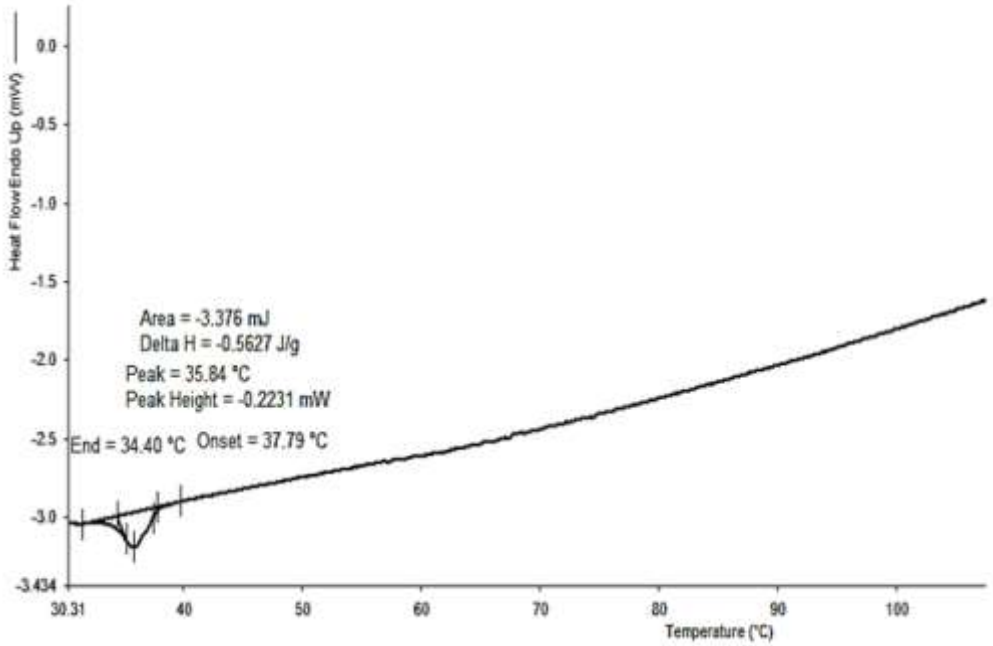

Figure 8: Cooling run of the toner sample (ICT laboratory, Mumbai)

The toner particle was characterized second time at IIT-Bombay (DSC Q20 V24.11 Build 124 instrument). In the heating run of the sample (figure 9), one endothermic peak is observed at 71.82^0 C. The heat flow involved is -0.6779 W/g. From 28^0 C to 70^0 C, there is a first stable baseline with mean heat flow of -0.4 W/g , with clear break of endothermic peak and shift in the baseline observed thereon from 72^0 C till 150^0 C, second baseline happens with mean heat flow of -0.55 W/g. The onset temperature of the endothermic peak is 69.98^0 C and the end set temperature is 71.91^0 C. with clear break in the base line, indicates a glass transition process. The toner material transit from a solid state into a glassy state showing partial melting here. Slopes of the first baseline is negative 0.0021 w/g/^{0}C and second baseline negative 0.0009 w/g/^{0}C pointing that smaller amount of heat is utilised in second baseline phase of the heating run.

The toner sample was further thermal characterized (3rd) at P.S. Ramanathan Advanced Instrumentation Centre, Ruia College (figure 10). The graph indicates an endothermic peak at 70.40^0C. The onset temperature is 69.65^0 C and the end set temperature is 70.87^0C. From 40^0C to 50^0C, we get the value of slope as 0.002 mW/^{0}C and from 80^0C to 90^0C, the value of slope is 0.013 mW/^{0}C. This too indicates a glass transition process.

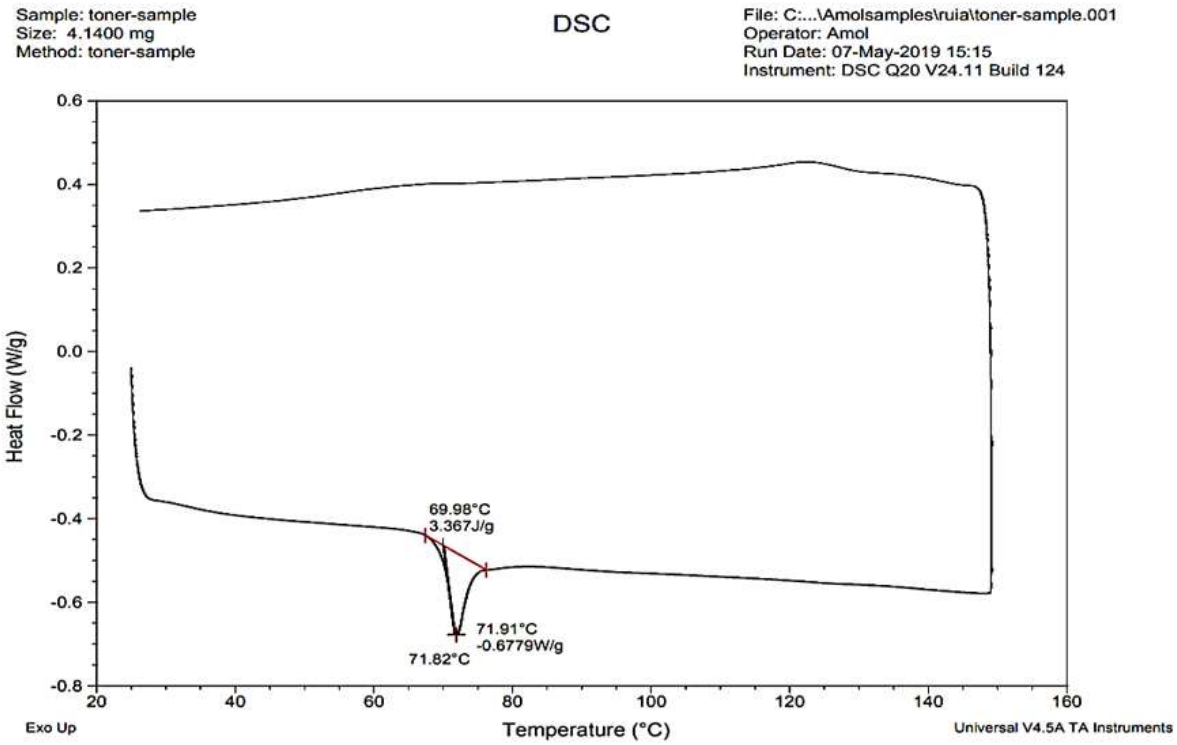

Figure 9: Heating run of the toner sample (IIT-B laboratory)

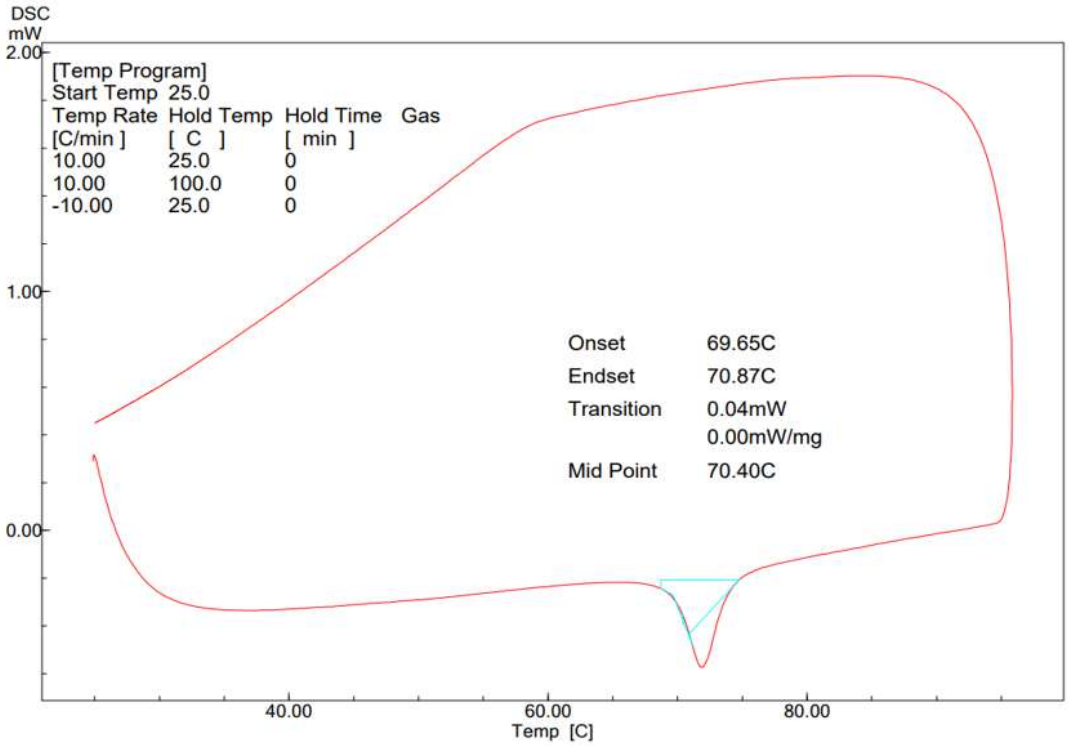

Figure 10: DSC graph of toner sample (RRL-Ruia)

On comparison of the three DSC characterisations, performed at three different laboratories, brought out common feature that glass transition happens at or near 71^0 C. Enthalpy change found to be slightly different, reason may be different instrumentation at two laboratories. Thermal analysis of the toner sample gives the glass transition temperature, heat flow on the stable baselines, the endothermic temperature peaks for heating run, enthalpy change involved in the transition process.

Conclusion:

The toner sample powder is annealed at 50^0C and 100^0C, crushed and XRD analysed, there was a change in crystallite size of the particle. FTIR spectroscopy showed the presence of halo compounds, alkenes, carboxylic acid, aliphatic ketones etc. UV visible spectroscopy shows the presence of ketones. Using Differential Scanning Calorimetry, thermal behaviour of the toner sample is investigated, which pointed to the glassy phase transition.

References:

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