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STUDY OF THERMAL PROPERTY MATRIX VIA DSC FOR REINFORCED CALCIUM
CARBONATE IN DIFFERENT RATIO IN HIGH DENSITY POLYETHYLENE

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ABSTRACT

Differential scanning calorimetry (DSC) is monitoring heat effects associated with phase transition and chemical reactions as a temperature function. In a DSC the difference in heat flow to the reference and on sample at the same conditions, the temperature value is recorded.

DSC is widely used for examining plastic materials to determine their thermal transitions in material and crystallization behavior

In present experiment Particular size of calcium carbonate is reinforced in high density polyethylene in different weight ratio.

The objective of present experiment to study the effect of thermal properties of reinforced blended material as compare to neat material.

Keywords- *Calcium Carbonate, HDPE, Differential scanning calorimetry, DSC.*

I. INTRODUCTION

Semi crystalline polymer do not exhibit melting point sharply as defined transition from solid to liquid state occurring at discrete temperature. Polyethylene undergoes a transition from the semi crystalline to the molten state that takes place over temperature range that can span from less than 10°C up to 70°C. As it passes through this transition the semi crystalline morphology gradually takes on more of the characteristics of the amorphous state at the expense of crystalline regions. The melting range broad because it consists series of overlapping melting points that correspond to the melting of lamellae of various thickness. The thicker lamellae have higher melting point. A dispersion of lamellar thickness is a natural consequence of entanglement and chain branching that divides chain backbone into a series of discrete crystallizable sequences with a distribution of length. Further broadening environment of the distribution of crystallite sizes and hence the melting range occurs. When crystallization occurs over a range of temperature as the sample cools.

Differential scanning calorimetry monitors the heat effect associated with phase transition as a function of temperature. In DSC the heat flow rate difference into the sample and reference is measured as a function of temperature. While the sample is subjected to a controlled temperature program and reference material such as alumina an inert material. The temperature on both sample and reference are increased at constant rate. The basic principle involved is that when sample undergoes physical transformation more (or less) heat will need to flow on it and then reference is maintained both at the same temperature.

In DSC whole process was carried out under environment of nitrogen gas in order to prevent any chance of thermal degradation. The samples are heated beyond melting point @10°C/min and kept at temperature for 5 minutes to remove its thermal history and then cooled down to the room temperature at kept at that temperature for 2 min. The cooled sample was again heated @10°C/min to study the effect of crystallization finally get the melting point T_m & enthalpy of heating ΔH J/g. Glass transition temperature is reversible change of the amorphous region of a polymer material from (or to) a viscous condition to (or from) a hard, relatively brittle one. The temperature range at which the glass transition takes place.

The endothermic transition upon heating from crystalline solid to the liquid state is called melting point. For crystallization time, melting temperature is an one step process while crystallization involves crystals growth and nucleation. Nucleation is dependent on cooling rate whereas the melting is unaffected if material contains other impurities or amorphous material, it will lead to a lowering of melting enthalpy. An amorphous content of material will give rise to a glass transition temperature.

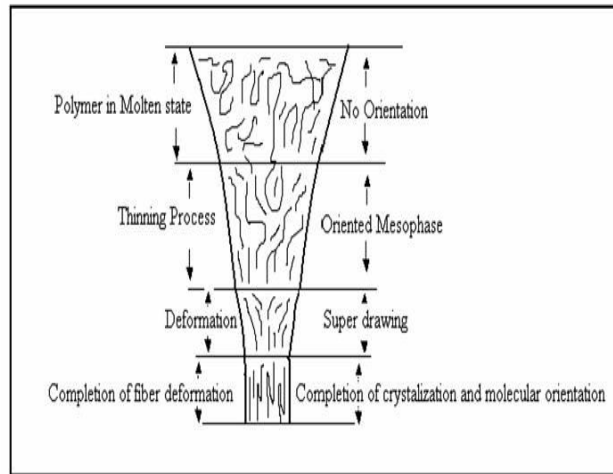


Fig 1 Spin line step wise structure for development of fiber structure at high temperature

II. EXPERIMENT

Crystallization behaviors of the nano composites were investigated with a differential scanning

Calorimeter, Toshvin DSC-60 Shimadzu makes. The differential scanning calorimeter is calibrated using indium with sample weight of 1 mg. All processes were carried out in a purge nitrogen gas. The samples were heated to 150°C and held for 5 min in the molten state to remove the influence of thermal history. The melt sample is then subsequently quenched at a rate of 10°C/min to reach the specific temperature and kept for 1Hrs at that temperature. When the crystallization process was completed, the samples were heated to 300 C at a rate of 10 °C/min to measure the melting temperature.

In this experiment, HDPE used for this work is Relene Grade. M60075 of Reliance Industries Ltd. [Density: 0.94 gm/cc; MFI: 8-10 gm/10 minute]. The nano-filler used in this work is calcium carbonate, purchase from local market in Maharashtra. The Calcium carbonate (coated) used is having average particle size 9-11 nm, Grade OMYACARB 2T –SA of Omya Malaysia SDN BHD Malaysia.

$$\% \text{ Crystallinity} = (\Delta H_{\text{obs}} / \Delta H_f) * 100$$

Where ΔH_f = heat of fusion for polyethylene 293.6 J/g

Following are the Graphs for HDPE material and blended material

Fig 2: For HDPE material

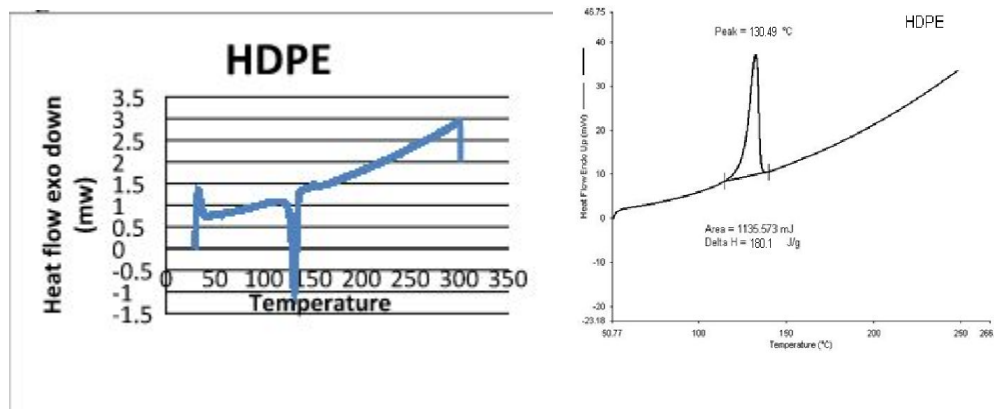


Fig 3: For blended material HDCC (HD+5% CC)

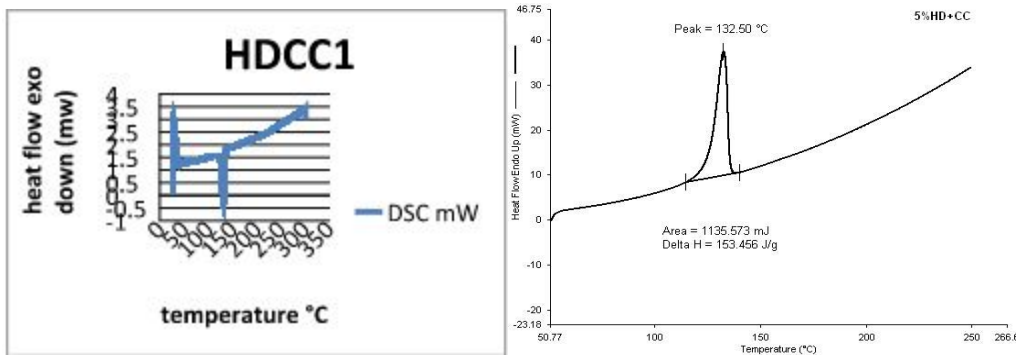


Fig 4: For blended material HDCC (HD+10% CC)

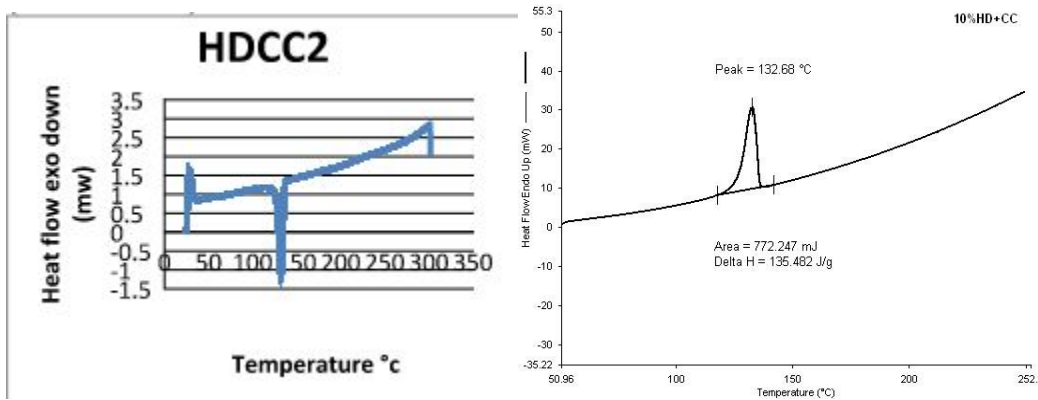


Fig 5: For blended material HDCC (HD+15% CC)

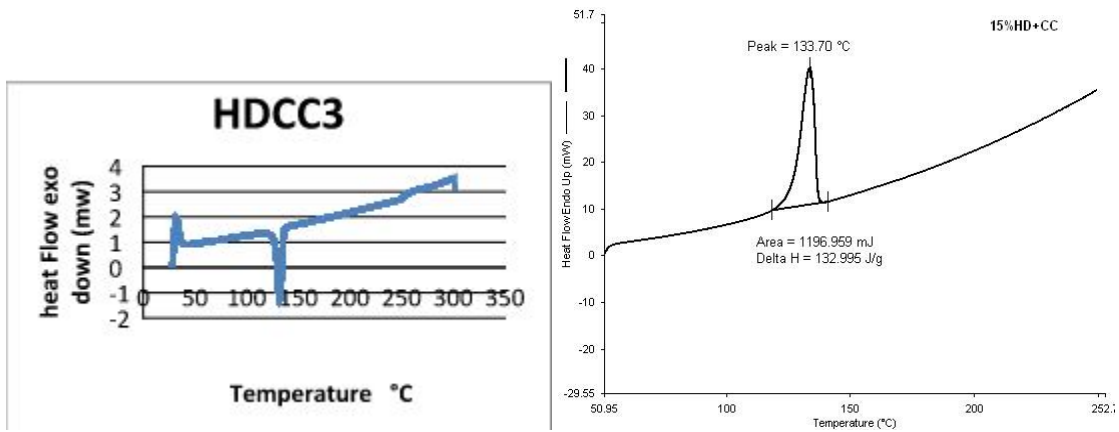


Fig 6 : For blended material HDCC (HD+20% CC)

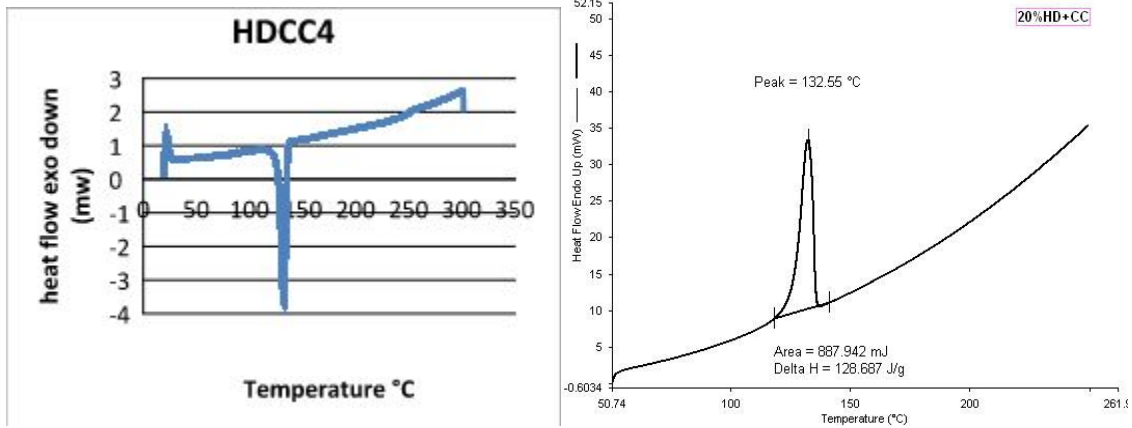


Fig 7: For blended material HDCC (HD+25% CC)

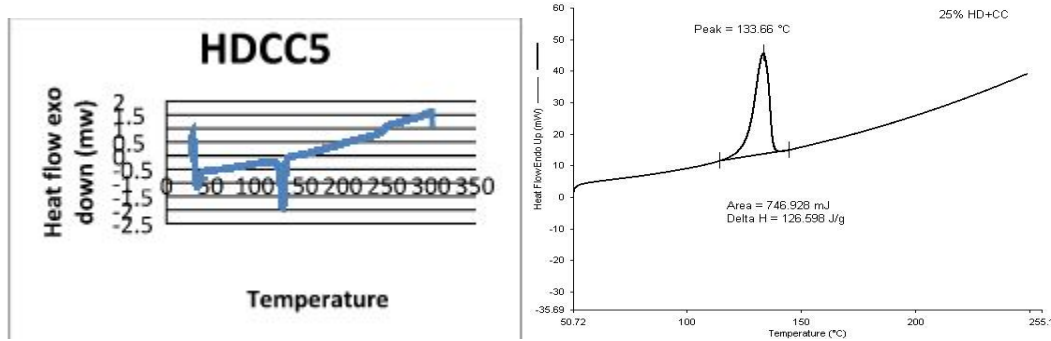


Fig 8: For blended material HDCC (HD+30% CC)

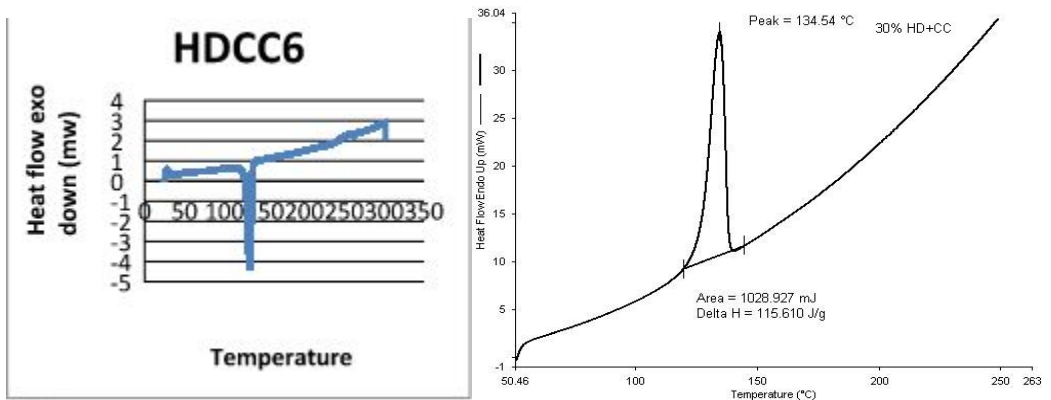


Fig 9: For blended material HDCC (HD+35% CC)

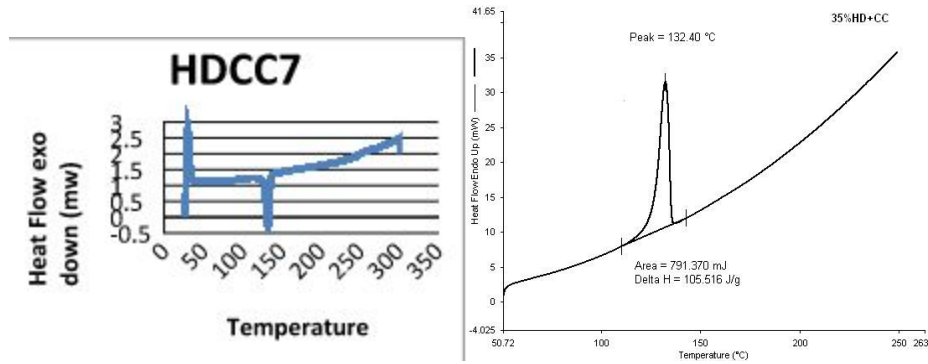
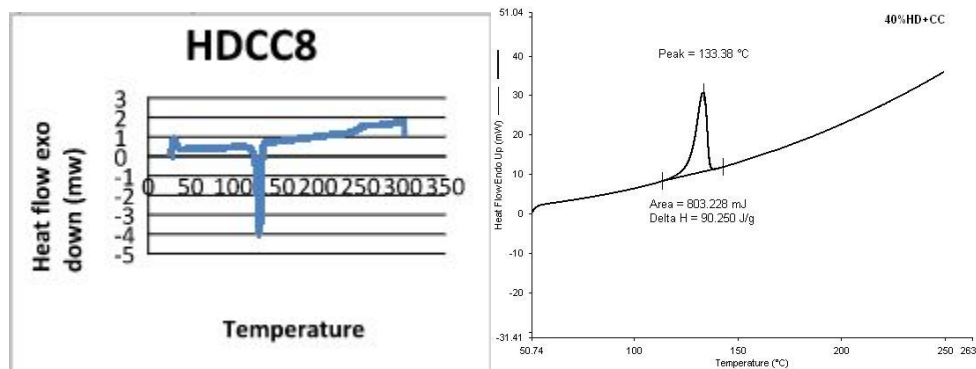


Fig 10: For blended material HDCC8 (HD+40% CC)



III. RESULTS & CONCLUSION

Table 1 : Result summary

S. No.	Material	Tg °C	Melt onset temperature °C	Melt endset temperature °C	Melt peak temperature °C	ΔH	% Crystallinity	Decomposition temperature °C
1	HDPE	36.9	128.83	135.27	130.49	180.1	61.3	151.41
2	HDCC1 (HD+5% CC)	35.55	128.89	135.14	132.5	153.456	52.26	225.14
3	HDCC2 (HD+10% CC)	33.45	127.79	135.87	132.68	135.482	46.14	241.58
4	HDCC3 (HD+15% CC)	36.36	128.08	135.42	133.7	132.995	45.29	248.22
5	HDCC4 (HD+20% CC)	34.96	128.17	135.59	132.55	128.687	43.83	247.47

6	HDCC5 (HD+25% CC)	33.61	127.37	134.88	133.66	126.598	43.11	248.0
7	HDCC6 (HD+30% CC)	38.75	128.08	135.64	134.54	115.610	39.37	251.89
8	HDCC7 (HD+35% CC)	35.7	128.65	136.34	132.4	105.516	35.93	249.4
9	HDCC8 (HD+40% CC)	33.34	127.13	134.48	133.38	90.250	30.73	250.0

Due to presence of filler calcium carbonate Tg step height is decrease as the filler content is increases but show no effect in Tg value. There is no drastic change in melting point of blended material as compare to the neat material. Change in heat of fusion(ΔH) is observed due presence of filler (Calcium carbonate) in decreasing trend with increase in filler content , that effect the crystallinity of material.

As the crystallinity of the material Decrease as the filler content increases shows the amorphous content is increases as the filler content increases ,due to this mechanical properties of the blended material effected as compare to neat material . Mechanical properties of blended material are under study.

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