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RESEARCH ARTICLE

Study of XRD property matrix by reinforcement of calcium carbonate in different ratio in High density polyethylene

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ABSTRACT

Polymer exist both in crystalline and amorphous form, polymer crystallinity is one of the most important property of all polymer. Crystallinity of the material is the Presence of amount of crystalline region in polymer with respect to amorphous region, Crystallinity influences most of the polymer properties, some of these are hardness , modulus, stiffness and tensile. X-ray powder diffraction (XRD) is a most commonly used analytical technique for phase identification of a crystalline material and can provide information for the unit cell dimensions. The analyzed material is finely homogenized, ground and average bulk composition is to be determined. X-ray diffraction is also used to measure the nature of polymer and extent of crystallinity present in the polymeric material. Calcium carbonate is used as inorganic filler reinforcement with HDPE as base material with coupling agent, proportionally with eight different proportions. The addition of 5%, 10%, 15%, 20%, 25%, 30%, 35% and 40% on weight fraction basis mixed with HDPE, with the help of an extrusion machine. The resulting mixture characterized by unique microstructure which is responsible for their properties. The reinforcement is stiffer and stronger, than the basic polymer. It increases its modules and strength with change of crystallinity. The objective of the present investigation is to find out the change in microstructure of the composites, and compared with neat material.

Keyword: Calcium Carbonate, HDPE, Filler Material, crystallinity, X-ray diffraction.

INTRODUCTION

In X- ray powder Diffraction, if powder are placed in the path of monochromatic X - ray beam, Diffraction is occur from the planes in those crystallites that are oriented at the angle to fulfill the Bragg condition (Cullity and Stock, 2001a; 2001b; Suryanarayana and Norton, 1998).

In powder Diffraction, Powder is composed of many finely & small ground crystals known as crystallites and these crystallites assumed to be oriented randomly to one another (Smart and Moore, 2012). Crystallites solid consist of regular array of atoms, Ion or molecules with inter atomic spacing, and the wavelength of the incident light has to be on the same order as the spacing of the atom (Ladd and Palmer, 1998)

According to Bragg law,

$$\lambda = 2d \sin\theta$$

for cubic system

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

$$d_{hkl} = \frac{A}{\sqrt{h^2 + k^2 + l^2}}$$

$$\rightarrow \quad \lambda = \frac{2a\sin\theta}{\sqrt{h^2 + k^2 + l^2}}$$

$$\rightarrow \sin^2\theta = \frac{\lambda^2}{4a^2} (h^2 + k^2 + l^2)$$

Table 1: Possible Values of h k l

Hkl	$h^2 + k^2 + l^2$
100	1
110	2
111	3
200	4
210	5
211	6
220	8
221	9
300	9

MATERIALS AND METHODS

Experiment

In powder diffraction, the powder sealed in the glass capillary and is diffracted the X-ray beam to produce the cones of diffracted beam, these cones intersect a strip of photo graphic film located in the camera (cylindrical) to produce a characteristic sets of arcs on the film . The film can be removed and examined, with the use of radius of the camera and distance along the film from the center, 2θ Bragg angle and then the d-spacing for each reflection can be calculated.

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.

This HDPE material and calcium carbonate used is the blended in twin screw extruder at desire temperature, speed and pressure parameter combinations, then this blended granules are moulded into testing specimens with the aid of injection moulding machine at stipulated moulding parameter (Temperature, speed & pressure). The combination used for this experiment is HDPE: CC in the ratio of mentioned in tabular form Table 2.

Table 2: composition details

Specimen code	HDPE %	Coated $CaCo_3$ %
HDCC-1	95	5
HDCC-2	90	10
HDCC-3	85	15
HDCC-4	80	20
HDCC-5	75	25
HDCC-6	70	30
HDCC-7	65	35
HDCC-8	60	40

RESULT AND DISCUSSION

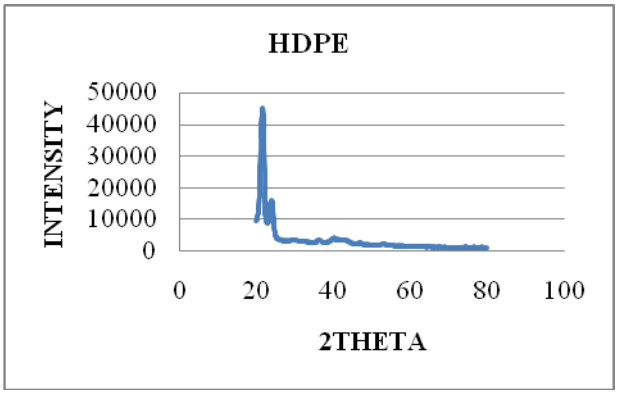
Results based on observation shows change on the angle of plane from 111 to 211 between virgin HDCC and different compositions of HDPE and calcium Carbonate. from these observation it conclude that as angle of plane is changes and number of peaks is increase as compare to neat material, there is change in crystallinity of material changes when Calcium carbonate is mixed with HDPE, but there is drastic change on angle of diffraction 2θ values it implies that basic structure of neat material is not changed only angle of plane is changes that effect the crystallinity of the material.

Table 3: Calculated value of h k l

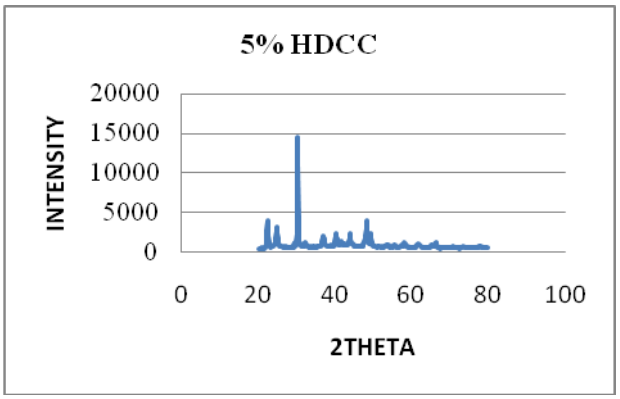
	2θ	Θ	sinθ	$Sin^2θ$	Ratio	Ratio *3	$h^2k^2l^2$	INDEX
HDCC8	21.79	10.9	0.189	0.0357	1	-	-	--
	29.75	14.9	0.257	0.0659	1.85	5.55	6	211
	48.65	24.3	0.412	0.1696	2.57	7.77	8	220
HDCC7	21.59	10.8	0.187	0.035	1	--	--	
	29.55	14.8	0.255	0.065	1.85	5.6	6	211
	47.46	23.7	0.40	0.16	2.5	7.5	8	220
HDCC6	21.69	10.84	0.188	0.035	1	--	--	--
	29.55	14.78	0.255	0.0652	1.86	5.6	6	211
	47.56	23.78	0.403	0.163	2.5	7.5	8	220
HDCC5	21.59	10.795	0.1873	0.0351	1	--	--	--
	29.55	14.775	0.255	0.065	1.85	5.6	6	211
	47.65	23.83	0.4039	0.163	2.51	7.5	8	220
HDCC 4	22.28	11.14	0.1932	0.037	1	--	--	--
	30.14	15.07	0.259	0.0675	1.82	5.5	6	211
	48.24	24.12	0.4086	0.167	2.5	7.5	8	220
HDCC3	22.28	11.29	0.196	0.0384	1	--	--	--
	30.24	15.12	0.261	0.068	1.8	5.4	5	210
	48.34	24.17	0.41	0.167	2.5	7.5	8	220
HDCC2	22.18	11.06	0.19	0.037	1	--	--	--
	30.04	15.02	0.259	0.067	1.8	5.43	5	210
	48.24	24.12	0.41	0.17	2.5	7.5	8	220
HDCC1	22.28	11.14	0.19	0.037	1	--	--	--
	30.14	15.07	0.259	0.0675	1.82	5.5	6	211
	48.24	24.12	40.86	0.17	2.5	7.5	8	220
HDPE	21.59	10.8	0.187	0.0351	1	-	-	
	22.39	11.2	0.194	0.0377	1.07	3.22	3	111

Table 4 : Shows change on the angle of plane from 111 to 211 between virgin HDCC and different compositions of HDPE and calcium Carbonate

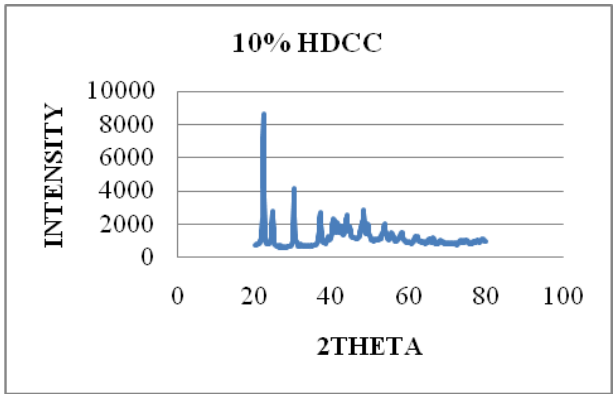
Sample code	Lambda	a	2θ	Index
HDPE	1.5406Å	3.872 Å	21.59	111
HDCC1	1.5406Å	3.872 Å	30.14	211/220
HDCC2	1.5406Å	3.872 Å	22.18	210/220
HDCC3	1.5406Å	3.872 Å	30.24	210/220
HDCC4	1.5406Å	3.872 Å	30.14	211/220
HDCC5	1.5406Å	3.872 Å	29.55	211/220
HDCC 6	1.5406Å	3.872 Å	29.55	211/220
HDCC 7	1.5406Å	3.872 Å	29.55	211/220
HDCC 8	1.5406Å	3.872 Å	29.75	211/220



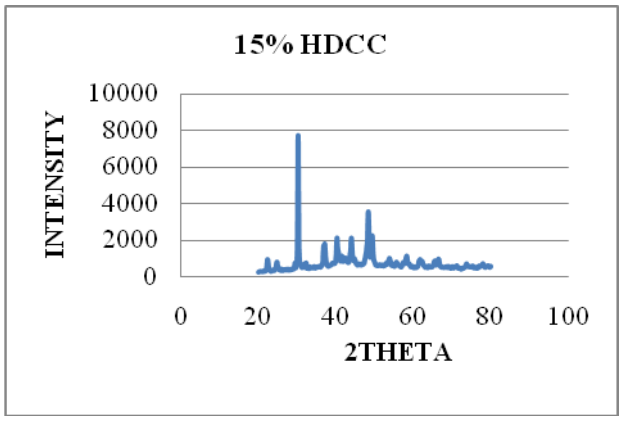
For HDPE
2θ = 21.59 at maximum intensity
Lambda : 1.5406Å
a = 3.872 Å



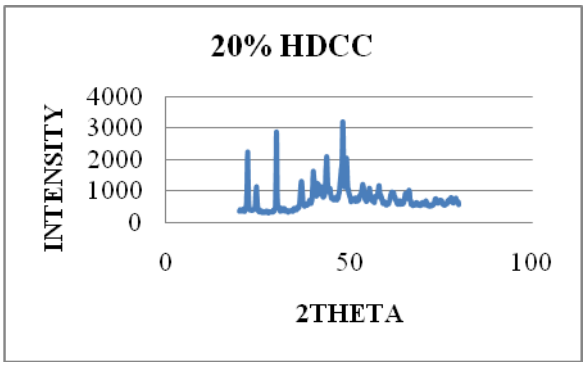
For 5% HDCC
2θ = 30.14 at maximum intensity
Lambda : 1.5406Å
a = 3.872 Å



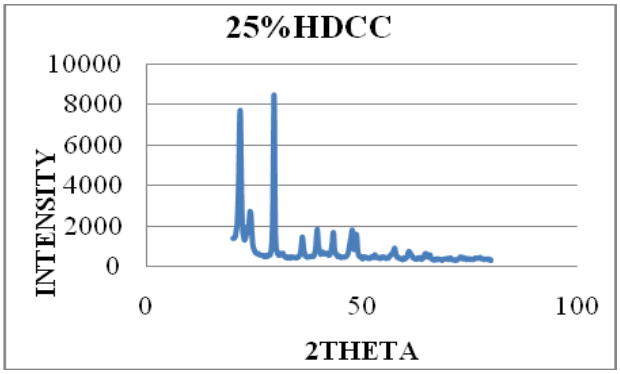
For 10% HDCC
2θ = 22.18 at maximum intensity
Lambda : 1.5406Å
a = 3.872 Å



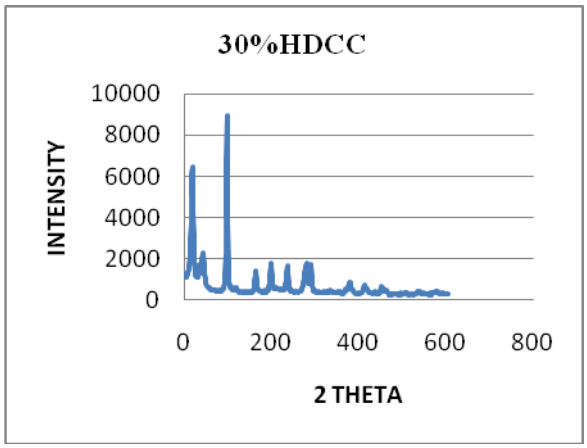
For 15% HDCC
2θ = 30.24 at maximum intensity
Lambda : 1.5406Å
a = 3.872 Å



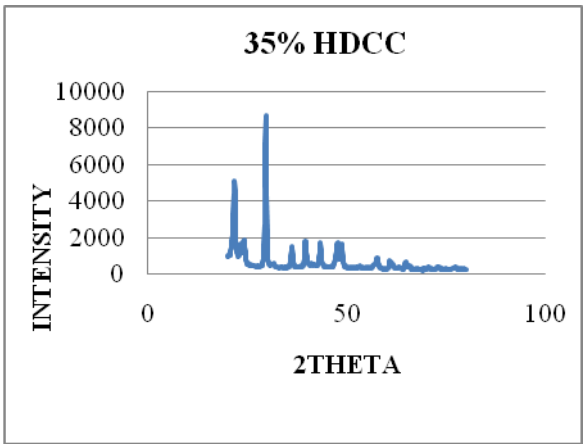
For 20% HDCC
2θ = 30.14 at maximum intensity
Lambda : 1.5406Å
a = 3.872 Å



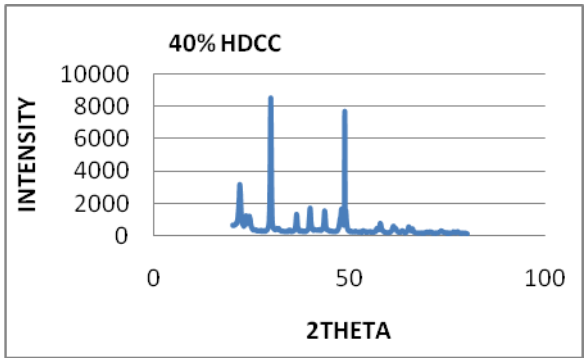
For 25% HDCC
2θ = 29.55 at maximum intensity
Lambda : 1.5406Å
a = 3.872 Å



For 30% HDCC
2θ = 29.55 at maximum intensity
Lambda : 1.5406Å
a = 3.872 Å



For 35% HDCC
2θ = 29.55 at maximum intensity
Lambda : 1.5406Å
a = 3.872 Å



For 40% HDCC
2θ = 29.75 at maximum intensity
Lambda : 1.5406Å
a = 3.872 Å

Due to change in crystallinity of the material shows the change in the mechanical properties of the blended material. This is under study.

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