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Deconvolution of X-ray Diffraction Spectrum of Polypropylene

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Abstract

Deconvolution methods have been used to evaluate degree of crystallinity and particle size from diffractogram of polypropylene(PP). The deconvolution method is used to separate the various peaks from the total XRD spectrum and it is successful in separating the crystalline, amorphous and background regions of a diffractogram. The degree of crystallinity of PP is calculated to be 49%. The particle size of crystallites in different crystalline phase is calculated using Scherrer equation.

Keywords: *X-ray diffractogram (XRD), Deconvolution method, degree of crystallinity, Particle size, Polypropylene*

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INTRODUCTION

X-ray diffractogram of semi-crystalline polymers consist both amorphous and crystalline regions besides the background region. The background spectrum is due to additive/ stabilizers present in polymer. Therefore efforts have been made to separate the three regions. A base line was constructed using computer technique, which separate the background, crystalline and amorphous contributions. Area under the sharp XRD peaks correspond to the contribution of crystalline region, while the broad hallow represent amorphous domain of the polymer. The spectral area corresponding to all the three regions are calculated by double integration methods. The estimation of degree of crystallinity (doc) by XRD method is initially proposed by Nara and Komiya [1]. Later the method is applied to evaluate degree of crystallinity of starches derived from medicinal plants by Wang et al. [2]. The method is as follows:

If A_c represent area under crystalline peaks and A_a is area under amorphous region, the degree of crystallinity(X_c) is given by:

$$X_c = A_c /(A_c + A_a) \qquad (1)$$

This method requires resolution of XRD peaks. Hence deconvolution of XRD peaks is made. Sugimoto et al. have used deconvolution methods to separate contribution from different functional groups contributing to the total infrared spectrum of polymers [3]. Oxidized polyethylene gives infrared absorption bands in the region of 1700–1800 cm^{-1} [4]. Sugimoto et al. have resolved the FTIR spectrum to be due to three functional groups. They are (i) 1713 cm^{-1} band due to carboxylic acid groups (ii) 1738 cm^{-1} band due to carboxylic ester group and (iii) 1779 cm^{-1} band due to carboxylic anhydride group [3]. All the three groups will give a broad absorption band in the 1700–1800 cm^{-1} region. The deconvolution method is applied and peaks corresponding to region have been successfully resolved by Horiguchi et al. [4]. These authors have used Lorentz or Cauchy distribution function in the following form

$$f(\nu) = 1/ \Pi\Delta_0 \; \{ 1/ [1+ (\nu - \nu_{nc'}/\Delta \nu)]\} \qquad (2)$$

where, ν is wave number in cm^{-1}, $\nu_{nc'}$ is wave number of peak nc', and Δ_0 is the peak width of half height.

Geleski et al. have used deconvolution technique to identify different crystalline phases of polyamide 6 (PA6) has shown the sharp XRD peak at 2θ angles of 19–20^0 and 23–24^0 corresponding to α_I and α_{II} crystalline phases. Geleski have utilized deconvolution method to resolve the contribution of each phase [5]. The deconvolution method enabled to separate the crystalline contribution of α_I phase corresponding to (200) reflection; α_{II} phase due to (002) reflection; (200) reflection giving γ-phase; (020) plane due to γ-phase and a higher order peak due to amorphous content of PA6. These authors have also used Gaussian distribution function to reconstruct the XRD spectrum having following type

$$I = I_0 \exp [-(H-H_0)^2 / (\Delta H)^2] \quad (3)$$

where, $\Delta H = ½ \ln 2\, \Delta H_{1/2} = 0.6\, \Delta H_{1/2}$, $\Delta H_{1/2}$ is the width of half peak height, I_0 is the Peak amplitude.
The method of calculating I_0 and $\Delta H_{1/2}$ are as described by Sanjeeva Rao [7].

EXPERIMENTAL

Polypropylene in the form of powder is used in the present studies. X-ray diffractogram are recorded in Braker D8 diffractometer for powder samples of PP. X-rays produced using a sealed tube having a wavelength of 0.154 nm (K_α radiation).

RESULTS AND DISCUSSION

X-ray diffractogram of Polypropylene is as shown in Figure 1. The diffractogram possess various peaks corresponding to different (hkl) planes of polypropylene. They are listed in Table 1.

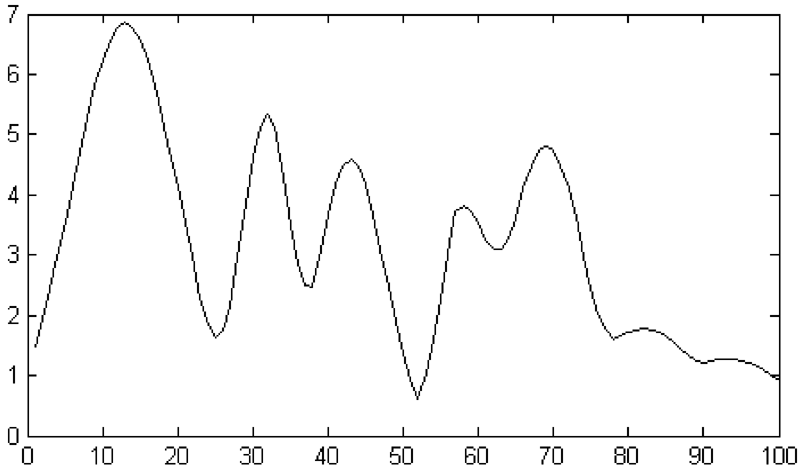

Fig. 1: *XRD Spectrum of Polypropylene.*

Table 1: *Various Peak Positions of XRD Spectrum of Polypropylene and their (hkl) Planes.*

Sl.No	Peak position at (2θ)	(hkl) plane
1	13.7	110
2	16.7	040
3	18.2	130
4	20.8	111
5	21.7	131
6	24.0	-
7	25.2	-

PP is reported to exhibit different crystalline forms namely α, β, γ-phases [8]. These crystalline phases arise due to different (hkl) planes corresponding to the PP [9]. Out of these peaks Casewell et al. have identified planes (peaks 1–5) corresponding to α-phase of PP [9]. The remaining two diffraction peaks at a 2θ angle of 21.7^0 and 25.2^0 were not identified. The authors have used deconvolution methods to separate the contribution of each peak from the total XRD spectrum of PP using Eq. (2). As such the resolved peaks are as shown in Figures 2–7. The parameters, used to simulate the individual peaks are as listed in Table 2. Using the parameters it is possible to resolve the area under crystalline, amorphous and background regions. Then using Eq. (2) the degree of crystallinity (X_c) is calculated. For polypropylene the Xc value is around 0.49 which is in the range of values reported in literature [10].

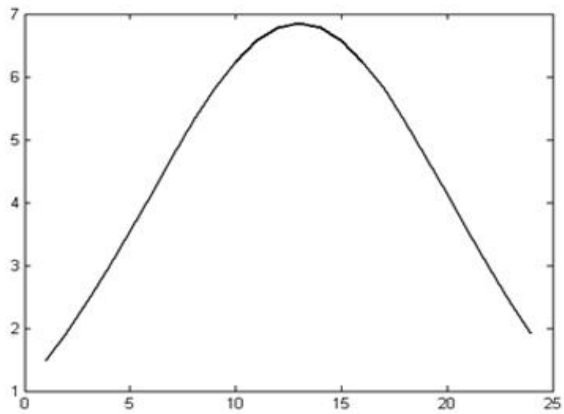

Fig. 2: Deconvolution of Peak I.

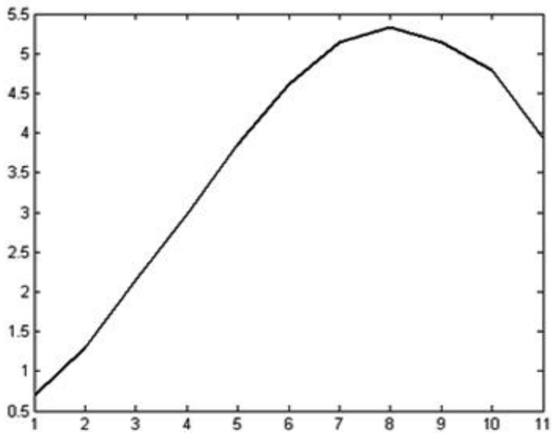

Fig. 3: Deconvolution of Peak II.

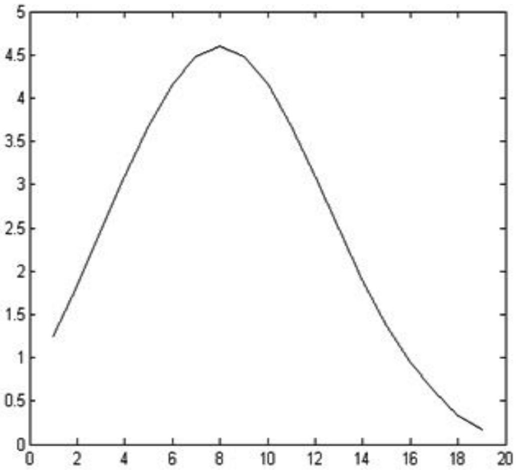

Fig. 4: Deconvolution of Peak III.

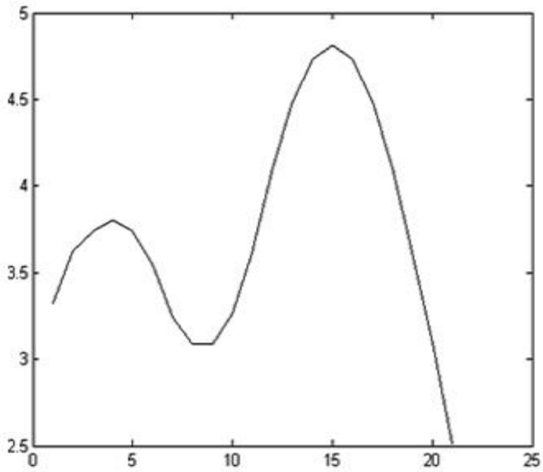

Fig. 5: Deconvolution of Peak IV.

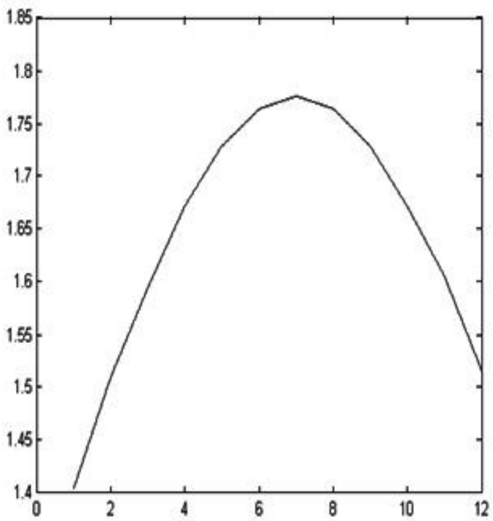

Fig. 6: Deconvolution of peak V.

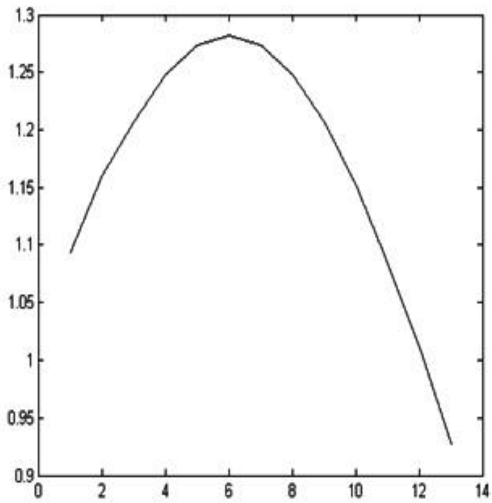

Fig. 7: Deconvolution of peak VI.

The particle size of crystallite is calculated using Scherrer equation for the crystalline peaks (1). The crystallite size corresponding α-phase is found to more than that of β and γ -phases.

Table 2: *Parameters used to simulate the XRD peaks.*

Sl.No	Peak position(2θ)	Peak amplitude	Peak width	Centre of Peak (2θ) degrees
1	13.7	7.0	0.8	13.7
2	16.7	5.8	0.4	16.7
3	18.2	4.85	6.5	18.2
4	20.8	3.95	0.6	20.8
5	21.7	5.0	0.6	21.7
6	24.0	1.8	1.0	24.0
7	25.2	1.30	1.0	25.2

CONCLUSION

Deconvolution methods are very useful in calculation of degree of crystallinity, particle size in semicrystalline polymer using X-ray diffraction method. The degree of crystallinity is estimated around 49% particle size of α-phase is more than β and γ-phases.

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