Structural properties different between two types of PE subjected to heat treatment

May Abdul Sattar Mohammed Najeeb, Kareem.A.Jasim and Dr. Nabil.N.Rammo
Collage of Education / pure science, Physics Department, University of Baghdad/ Ibn Al-Haitham collage, Baghdad / Iraq.

Abstract. In this study two modifications of polyethylene white (Pure one) and blue which is prepared with CaCo3 stabilized from polyethylene's tunnel have been studied by (XRD) subject to different temperatures at heat treatment. Both specimens show difference in the scattered X-ray intensity of the basal planes (110) and (200) with changing of the heat treatment. The degree of crystallinity of both modification of polyethylene (PE) has been studied. It has been looked that the white type is better than the blue type.

1. Introduction
Polymers have been selected for several reasons, including the use of common engineering materials for small, high performance parts and it offers many of the manufactures. Moreover, published research describing its structural characteristics appears to be under-represented because of its importance. Polymers are generally used in manufacturing and engineering [1, 2].

The analysis of X-ray powder diffraction patterns of samples of polyethylene (PE) revealed, in addition to traditional crystalline and amorphous components (amorphous phase) characterized by a diffuse scattering peak at the Kα diffraction angle of 80º (Cu – Kα radiation) [3, 4, 5].

In understanding of the degree of crystallinity for a polymer is important as crystallinity affects physical properties such as storage modules, permeability, density and melting temperature. While most of these manifestations of crystallinity can be determined a direct measure of degree of crystallinity provides a fundamental property from which these other physical properties can be predicted [6].

The aim of the present studies is to find the intensity of the crystalline, the intensity of amorphous and the degree of crystallinity at a variable of heat treatment for samples white PE and blue PE.

2. Experimental
We used two sets (Twelve pieces) for samples from different sources of PE manufactured with different origins, the first set (six pieces) is a white PE and the second set (six pieces) is also manufactured blue PE. Two specimens of polymeric materials were prepared. These materials were prepared by casting. These specimens were cut with rectangular dimensions (2X3) cm. Both samples were treated at (25, 70, 90, 110, 120, melted) °C for each set separately in (Carbolite furnace) for 30 minutes. The crystallization of the material was determined by x-ray technique, where 2θ angle (10 – 80) degree. We're using to determine the starchier preparing.
3. Results and discussion

Some categories of materials shrink when they are exposed to thermal treatment if the area of the specimen weren't constrained during the treatment and free – area annealing allows more freedom for the molecular structure to be rearranged and may rise a shrinkage when the heat treatment rising; In this work, these samples from this kind of materials. The maximum shrinkage when the sample melted (Liquid phase).

The alignment of the goniometer was periodically checked by recording the data for the standards (quartz and NaCl in the measurements in the reflection mode and a pentaerythritol single crystal in the measurements in the transmission mode). Three X – ray diffraction patterns measured for each sample were statistically processed with the use of the Microcal Origin 6.0 program to determine the average interplanar spacing's $d_{hkl}$ and were then indexed with the use of the Powtool program.

To estimate the degree of crystallinity, we determined the integrated intensity of crystalline by add all numerical intensities in the range of peak, the total sum then will be the value of crystalline peak. Then we get integrated of amorphous region take back – ground intensities at start and end selected a above. The degree of crystallinity $X_c$ of the samples was determined as:

$$X_c = [1 + (I_c / I_a)]^{-1} \times 100\%$$ (1)

Where $I_c$ is the crystalline intensity, $I_a$ is the amorphous intensity and $X_c$ is the degree of crystalline [7], as shown in Table (1 and 2).

**Table 1.** The intensities of crystalline and amorphous for white and blue PE.

| T (°C) | Ic(White PE)(Count/Sec) | Ia(White PE)(Count/Sec) | Ic(Blue PE)(Count/Sec) | Ia(Blue PE)(Count/Sec) |
|-------|------------------------|-------------------------|------------------------|------------------------|
| 25    | 138606                 | 37593.6                 | 31006                  | 10731.6                |
| 70    | 250678                 | 76430.2                 | 34356                  | 13887.5                |
| 90    | 119202                 | 43718.4                 | 51106                  | 23501.3                |
| 110   | 213060                 | 79171.4                 | 30558                  | 13112                  |
| 120   | 220834                 | 93913.6                 | 25502                  | 13684                  |
| melted| 302516                 | 133892                  | 39694                  | 27766.9                |

**Table 2.** The degree of crystalline in different temperatures to each.

| T (°C) | Xc % White PE | Xc % Blue PE |
|-------|---------------|--------------|
| 25    | 78.66         | 74.29        |
| 70    | 76.60         | 71.21        |
| 90    | 73.16         | 68.50        |
| 110   | 72.91         | 67.08        |
| 120   | 70.16         | 66.50        |
| melted| 69.27         | 58.80        |

The accuracy of the determination was 5%.
The integrated intensity of the reflection (110) at the 2θ angle of ~ 20.5° was taken as the crystalline component of PE, because peaks at large angles make an insignificant contribution to the scattering samples [8, 9, 10, 11, 12, 13].

By using the above equation, the degree of crystallinity of the untreated sample and annealing samples at free area in the temperatures (25, 70, 90, 110, 120, melted)°C did cause change in the degree of crystallinity. At 25 °C the maximum degree of crystallinity was obtained; it amounted to 78.66% and the minimum degree of crystallinity was obtained; it amounted to 69.27% for White PE. While at 25 °C the maximum degree of the crystallinity was obtained; it amounted to 74.29% and the minimum degree of the crystallinity was obtained; it amounted to 58.8% for blue PE. The results of the degree of the crystallinity investigation in the two types are illustrated in Figures A (1, 2, 3, 4, 5, and 6) for white PE and Figures B (7, 8, 9, 10, 11, and 12) for blue PE.

We see that the angles of the intensity peaks are 21°.9°, 23°.5°, 36°.3° turn to a pure PE, but the angles 29°.8°, 36°.5°, 39°.5°, 43°.3°, 47°.8° goes to the call site phase. The reason of decreasing in the reflection peak (100) is the relaxation of the crystalline regions that found in the amorphous regions, this caused to increase the amorphous regions.
**Figure A.** The intensity vs Bragg's angle (2θ) of different temperatures for White PE.

**Figure B.** The intensity vs. Bragg's angle (2θ) of different temperatures for Blue PE.
Generally in white PE, the degree of crystallinity decreased about 5% until the sample in melt temperature. This means that the polymer kept the semi crystalline property until above of melt temperature. But in blue PE, the degree of crystallinity decreased about 15% until the sample in melt temperature. This means that the callisite affected on the semi crystalline property until the above of melt temperature. ‘Figure (C)’ shows the degree of the crystallites vs. temperature for white and blue PE.

![Figure C](image)

**Figure C.** degree of crystallinity vs different temperatures for (White & Blue) PE.

### 4. Conclusion
This method appeared more accuracy to find the degree of crystallinity with different heat treatment for white and blue PE which seen that the degree of crystallinity for a blue PE is lower than the degree of crystallinity for white PE because the callisite effected in the un arrangement for the crystalline regions. This caused to increase the amorphous regions with increasing the heat treatment in the heat cycle.

Note that the white and blue polymorphisms of A- B showed that the heat treatment has effect on the formation of the material as the material began to dissolve as the temperature increased in a different way to the material at 25°C. We also notice a change in the position of the integrated intensity of the reflection (110) indicated that the material began to respond to the heat treatment and received a change in the composition of the material.

### References

[1] Polymer engineering and science, 1969, 9(5).
[2] Colloid. polymer Sci 265:209, 1987.
[3] Jian S., binsong at el. Determination of degree of crystallinity of nylon 1212 by WAXD. *Chinese journal of polymer science*, 2004, 22(5):491 -496.
[4] Fresenius J. Anal Chem, 1995, 353:778 – 784.
[5] Original Russian text , Ya-A. Lebedev .....etc, 2010, 55(4): 657-652.
[6] Polymer physics edition. *Journal of polymer science*, 1983, 21: 1133-1145.
[7] Polymer, 1978, 19.
[8] Handbook of Instrumental Techniques for Analytical Chimestry Ed. Frank settle Ch, 1997, 50.
[9] KAKUDO M. and RULLMAN. *Journal of polymer science*, 1960, xlv: 91-101.
[10] Kolodka E., Wang W. Szhu J. and Hamielec A. *J.Appl. polym. Sci*, 2004, 92, 307.
[11] Moly K.A. et al. *European polymer journal*, 2005, 41: 1410-1419 .
[12] Brandon Rawlings. *Crystallinity in polymers*, 2009, 12.
[13] Mulla S . M . , at el . Use of the X – Ray Diffraction Technique for polymer Characterization and studying the Effect of the Optical Accessories. *ARAI*, 2012, 6.