Synthesis and Characterization of Some New Polymers for Isatin

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Abstract: This work included synthesis of several new polymers which derived form I satin by condensation of Isatin and different polymers (poly acryloyl chloride, PVC, PMMA, poly acrylo nitril in the presence triethyl amine as catalyst in THF gave new polymers structure confirmation of all polymers were proved using FT-IR, ¹H-NMR and UV spectroscopy, thermal analysis TG for some polymers confirmed their thermal stabilizes .Also the mechanical properties (hardness and compression) were measured and it was found to have high compressibility and hardness.

Keywords: Isatin, Poly methyl meth acryl ate

1. Introduction

Poly imides have became one of the most important and versatile classes of high performance polymer due to excellent mechanical and thermal properties. Poly imides are step polymers or condensation polymers derived from both aliphatic or aromatic dianhydrides and Diamines, or their derivatives (acid chlorides and amides. The Poly imides contain either hetro cyclicimide linkage or open imide linkage in Aherepeating unites. Poly imides are a class of representative high- performance polymers that have been widely used in flexible displays, poly imides involving aromatic and heterocyclic rings in the main chains are well known as heeal-resistant organic materials. In this study some poly imides bases of substitute Isatin, which 2, 3dioxiudde has been recently found to oxhibit endogamous activity in mammals. It is reported to exhibit broacl spectrum chem. other apeutic properties such as antibacterial, antifungal, antiHIV, antiviral, anticonvulsant, antitubercular and anticancer.

2. Material and Methods

Melting point was determined in Gallen Kamp melting point apparatus and was uncorrected. FT-IR spectra were recorded on Shimadzu FT-IR 8400 Fourier Transform Infrared spectrophotometer as KBR disc. ¹ H-NMR and ¹³ C-NMR spectra were recorded on Bruker specrospin Ultra shield magnets 300 MHz instrument using tetramethyl Silane (TMS) as an internal standard and DMSO-d₆ as a solvent in Ahl-Albate University in Jordan.

3. Synthesis of Poly Amides

(0.01mol) of poly a crylol chloride (or any polymers) was added to absolution of Isatin (0.01mol) in (10ml) of dimethyl form amide (DMF) in the presence of (1ml) of tritely amine (Et_3N). The mixtures were refluxed for (8hrs.). After cooling the solvent was removed to afford Avery viscous solution. Melting points, Yield data are listed in table (1).

| polymer | | | | | | | | |
|---------|-----------|-----------|------------|--------------|--|--|--|--|
| Comp. | Color | Softening | % | Solvent used | | | | |
| No | Coloi | Softening | Conversion | in reaction | | | | |
| 1 | Orange | 178-182 | 60 | THF | | | | |
| 2 | Brawn | 140-142 | 72 | THF | | | | |
| 3 | Yellow | 190-198 | 65 | DMF | | | | |
| 4 | Off white | 230-236 | 70 | DMF | | | | |
| 5 | Brawn | 167-173 | 55 | DMF | | | | |

 Table 1: The physical properties for Isatin with deferent

| | Tuble 2.1 1 In Spectral data for an product compounds | | | | | | | | |
|------------|---|---------------------------------------|---------------------------------|--------------------------|---------------------------------|----------|--|--|--|
| Com. No | γ (C-H) cm ⁻¹ aliph. | γ (C-H) cm ⁻¹ arom. | γ (C-N) cm ⁻¹ | γ (C=O) cm ⁻¹ | γ (C=C) cm ⁻¹ | Others | | | |
| 1 | 2780-2980 | 3026 | 1373 | 1740 | 1535 | | | | |
| 2 | 2890-2930 | 3039-3190 | 1420 | 1760 | 1580 | | | | |
| 3 | 2870-2890 | 3170 | 1415 | 1755 | 1612 | | | | |
| 4 | 2933-2893 | 3111 | 1462 | 1735 | 1697 | C-O 1201 | | | |
| 5 | 2930-2970 | 3077 | 1460 | 1770 | 1590 | | | | |

Table 2: FT-IR Spectral data for all product compounds

Table 3: Mechanical properties [(Hardness (shore D) and compression test (D Brazillian)]

| Comp. No | Hardness | d (mm) | t (mm) | P (N) | Brazillian test δ=2p/πdt |
|-------------|----------|--------|--------|-------|-----------------------------|
| 1 | 57.1 | 13.05 | 4.13 | 100 | 1.182 |
| 2 | 49.91 | 13.05 | 3 | 140 | 2.278 |
| 3 | 46.6 | 13.05 | 3.69 | 290 | 3.83 |
| 4 | 74.2 | 13.05 | 3.12 | 210 | 3.28 |
| 5 | 59.7 | 13.05 | 3.89 | 430 | 5.39 |



Figure 1: The Scale harder shore D

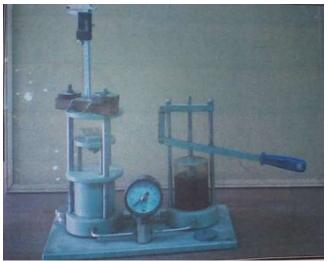
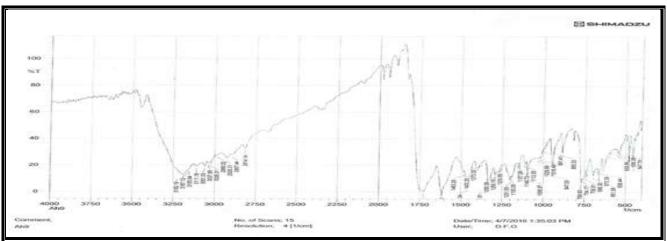


Figure 2: The Compression test instrument

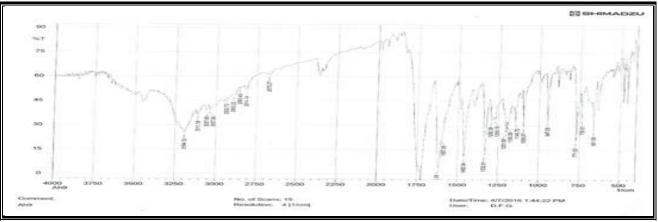


Finger 3: The FT-IR spectrum of compound [2]

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Finger 4: The FT-IR spectrum of compound [3]

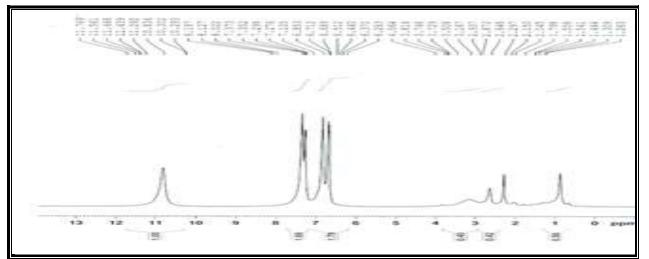


Figure 5: The ¹H-NMR of compound [1]

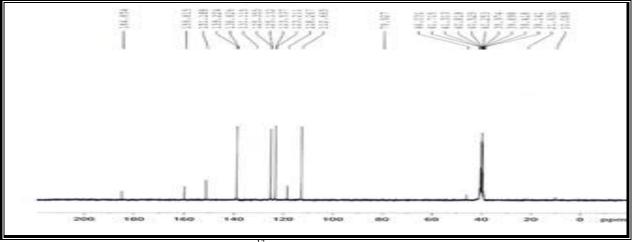
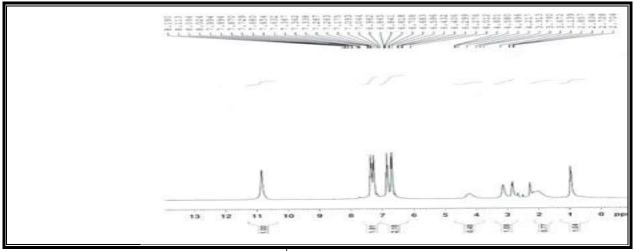


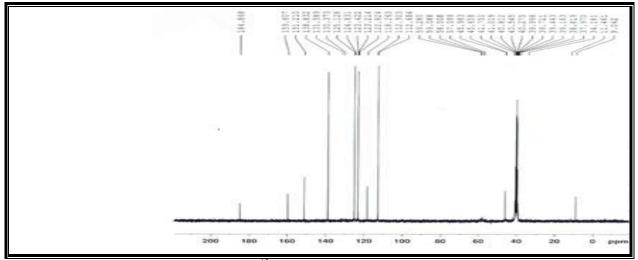
Figure 6: The ¹³C-NMR spectrum of compound [1]

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Finger 8: The ¹³C-NMR spectrum of compound [2]

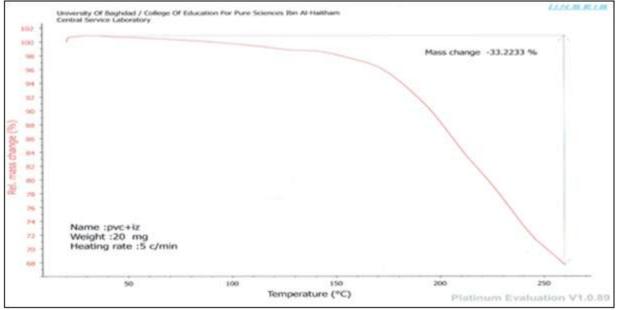


Figure 9: The TG of compound [2]

4. Results and Discussion

(such as poly acryloyl chloyide) in the presence of triethylamine (Et_3N) as a catalyst $^{[7]}. \ As is shown below;$

New polyimide containing heterocyclic moiety was synthesized by the reaction of Isatin with different polymers

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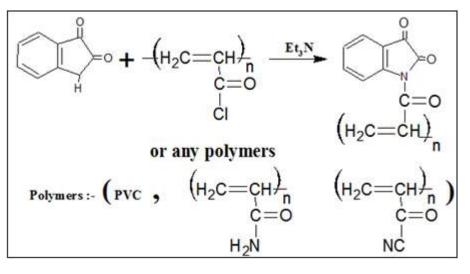
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The products were corroborated by measuring the PH value, which was found ranging between (6 -7.5) Structures confirmation of all prepared polymers were proved using FT-IR, ¹H-NMR, ¹³C-NMR. Spectroscopy and thermal analysis (TG) of some of the prepared polymers.

FT-IR Spectrum of compound [2] showed the following characteristic features: significant bands at (3039-3190) cm⁻¹, (2890-2930) cm⁻¹, (1420) cm⁻¹ and (1760) cm⁻¹ was attributed

at stretching vibrations of V (C-H) aromatic, V (C-H) aliphatic (C-N) and (C=O) respectively as shown in Table (2) as shown in figure (3) and (4).

¹H-NMR spectral data of compound (1) and (2) showed the signal at (1.3-3.5) pmm was attributed to (CH_2) proton and multiple signals at (6.2-8.1) ppm due to aromatic protons and singlet signal at (3.8) ppm due to



(-CH) proton as shown in figure (5) and (7), While ${}^{13}C$ -NMR Spectrum of compound (1) and (2) showed the signal at (184, 159) ppm for carbonyl group (C=O), while the signal at (112-151) ppm for aromatic carbons while the signal at (58, 79) ppm for carbon of group (C-N) as shown in fig (6) and (8). Thermal properties of some of the prepared polymers were studied using thermal gravimetric (TG).Thermal stability of polymer is consider one of the most important properties of macromolecules particularly those polymers which are designed to be used at high temperatures .Poly imides and related polymers are of such types of thermally stable polymers ^[9]. Thermal analysis of compound [2] showed moderate stability at 300C° as shown in fig (9). The mechanical properties of polymeric materials they might be improper by modifying the composition of the polymer. The compression resistance of the materials depends on the matrix properties, reinforcement properties, and strength of binding across interface and volume fraction of reinforcements and quality of voids and the direction of the fibers as shown in fig (2), while Hardness Testing is measured by determining the depth or project area of an indentation produced by a standard inventor the higher the hardness of the material, (¹¹⁾ the shallower the indentation for given load and the smaller the projected area as shown in fig (1) the results recorded in Table (3).

References

- T. B. Mustafa, "Synthesis and modification of some Hetro cyclic compound with potential Biological Activity Coupled on poly (Maleic Anhydride- Methyl Methacrylate", J.of American Science, 6 (8), p. (512-524) 2010.
- [2] E. O. Al-Temimi, S. J. Al-Bayati, , "Synthesis and polymerization of polyacryl Imide and one of Their

Derivatives Then curing the product with alkyl halide", Baghdad Science. J., 7 (1), P- (631-640) 2010.

- [3] Z. Tabatabaei Yazdi, S. M. –Alaei, "Poly (ether-imide) and related sepiolite nanocomposites: investigation of physical, thermal, and mechanical properties", Poly. Adv. Technol, 26, p (308-314) (2015), .
- [4] X. Tian, X. Jiang, H. Lu, D. Huang, (2012), "Synthesis and characterization of soluble and transparent. Copolyimides with controlled glass transition temperature", Indian J. of chemical Technology, 19, p. (271-277).
- [5] Q. Jin, K. Horie and 1- Mita, "Polyimides with Al: cyclic Diamines (Syntheses and Thermal properties), J. of polymer science, part A: Polymer chemistry, 31, p. (2345-2351). (1993),
- [6] S. J. Kaammas; M. A. Mahmoud; S. A. Yousif, Z. R. Abadul Mageed, "Synthesis and Reduction of some Mannich bases for substitute Indol-2, 3-dione", Tikrit J. of pure science, 19 (1), p (67-74). (2013),
- [7] R.M.Solverstein, F.X.Webster, D.J.Kiemle, "Spectrometric identification of organic compounds, "7th Ed., John wiley and Sons, Inc, (2005).
- [8] P.Crews, J. Rodriguez⁴M.Jaspars, "Organic structure analysis", Oxford University press, New York, (1998).
- [9] G.A.A.Al-Farajy, M.Sc Thesis, college of Science, University of Bagdad, (2003).
- [10] H.H.Thanon, ph.b, Thesis, Department of Applied physics, The school of Applied Science, University of Technology, (2006).
- [11] Z.G.Salih, MSo. Thesis, Department of Applied Sciences University of Technology, (2006).

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