



Synthesis and characterization of some new *N*-substituted phthalimide

Entesar O Al Tamimi*, Muna I Khalaf, Raghad A Abd Al-Jabar

Department of Chemistry, College of Science, University of Baghdad, Iraq

Abstract The present work involves preparation of *N*-substituted phthalimide derivatives. The Performing of this research includes three steps: The first step of this work was includes preparation of potassium phthalimide. The second step involves preparation of *N*-alkyl phthalimide by reacting of phthalimide salte with (benzylchloride, PVC, adipoyl chloride, and Aryloyl chloride) respectively. The third step includes polymerization of unsaturated prepared compounds by using (BPO) as initiator with heating, and reaction of another mole of phthalimide salt with *N*-Adipoyl mono chloride phthalimide. All the prepared compounds in this work were characterized by recording their melting points, softening point, infrared (FT-IR) spectra, and ¹H-NMR for some of them.

Keywords adepoyl chloride, phthalimides substituted, polymerization

Introduction

Cyclic imides reveal weak acidity of the O=C-NH-C=O group, Cyclic imides are privileged pharmacophores and important building blocks for the synthesis of natural products drugs, agrochemicals, advanced materials and polymers. Migrastatin lamprolobine, julocrotine, cladoniamide A palasimide and salfredin C-1 are some of the important natural products that have the imide motif [1-4]. Imides have been extensively used in the synthesis of several bioactive natural products [5-11]. Phthalimide possess a Structural features –CO-N(R)-CO- and an imide ring that help them to be biologically active and pharmaceutically useful [12]. Phthalimide have received attention due to their antibacterial, antifungal analgesic [13], antitumor [14,15], anxiolytic [16]. Phthalimide and some of its derivatives have proved to have important biological effects that are similar or even higher than known pharmacological molecules so their biological activity has been subject of biomedical research [17-18]. Gabriel synthesis: this method the sodium or potassium salt of phthalimide is *N*-alkylated with a primary alkyl halide to give the corresponding *N*-alkylphthalimide. The reaction fails with most secondary alkyl halides, Many high strength or electrically conductive polymers contain imide subunits, *i.e.*, the polyimides. One example is Kapton where the repeated unit consists of two imide groups derived from aromatic tetracarboxylic acids [19]. Another example of polyimides is the polyglutarimide typically made from polymethylmethacrylate (PMMA) and ammonia or a primary amine by aminolysis and cyclization of the PMMA at high temperature and pressure typically in an extruder, the toughness of these materials reflects the rigidity of the imide functional group.

Experimental

Material

All chemicals used were of analytical grade and they were obtained from Aldrich and Fluka companies.

Instrument

Melting points were determined by Gallen Kamp capillary melting point apparatus and they were uncorrected. FT-IR spectra were recorded using KBr disk by Shimadzu FT-IR 8400. Softening points were determined by Thermal Microscope Reichert Thermover 160.

¹H-NMR was measured using DMSO as a solvent by Bruker-500 MHz spectrophotometer using TMS as an internal standard.



Synthesis Methods for the Prepared Compounds:

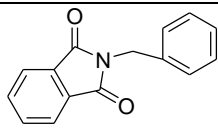
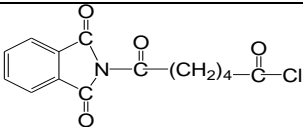
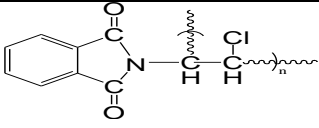
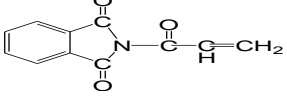
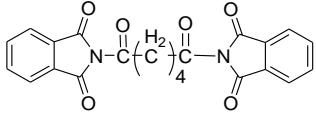
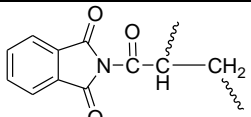
Preparation of Alkylphthalimide

A mixture of (0.01 mole) of phthalimide salt (potassium phthalimide which are prepared by dissolving potassium hydroxide in alcohol and then adding the solution to the calculated amount of phthalimide, the precipitate filtered then dried), 1,4-dioxane or DMF then was added to (0.01 mole) benzyl chloride and adipoyl chloride, acryloyl chloride and PVC escalation for an hour, After that the solvent was evaporated of the solvent, cooled. Then it was dissolved it in suitable solvent over heated and filtered, recrystallized from suitable solvent. Yield (64-91 %). Physical properties of the prepared compound are listed in table 1.

Polymerization of *N*-acryloylchloridephthalimide

0.3 g of prepared monomer was dissolved in 5 ml of DMF, and 0.05 % weight of dibenzoyl peroxide was added, under nitrogen atmosphere, the polymerization tube was covered, heated by water bath at 90 °C about 1 hr. The pale yellow polymer was obtained with 76 % yield with s.p about 210-222 °C (table 1).

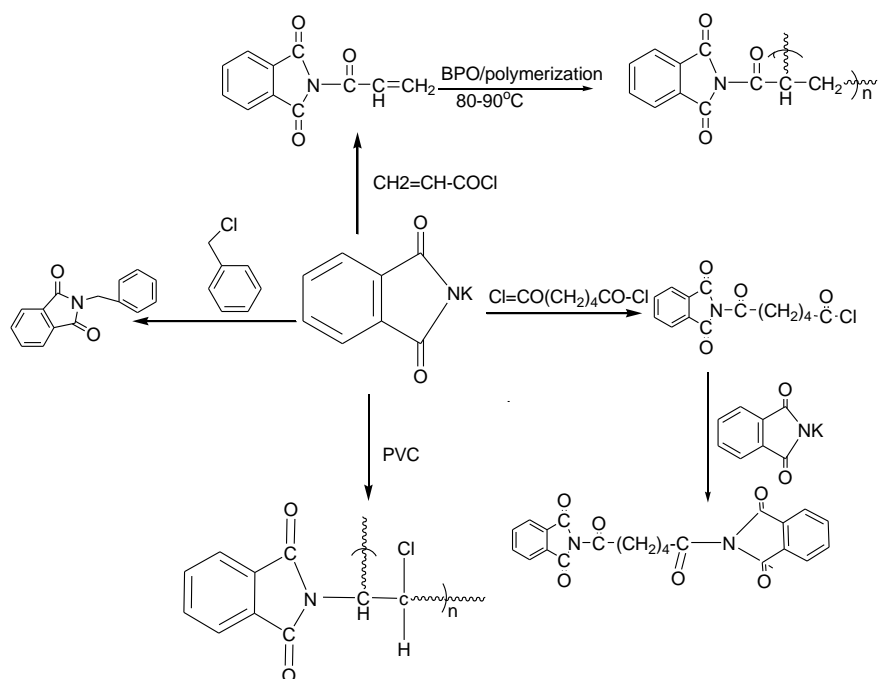
Table 1: Physical properties of the prepared compounds

No.	Structure	Reaction solvent	M.p or s.p (°C)	Yield %
1		1,4_dioxane	Dec. 210	73
2		1,4_dioxane	140	64
3		DMSO	S.P 160-169	91
4		1,4_dioxane	Oil	78
5		Dioxane	190	69
6		DMF	S.P 210-222	76

Results and Disscution

The synthesis sequences for preparation compounds of new series N-subst. Phthalimide derivatives (scheme -1-), a good yield were obtained ranging from (65-91)% and identification by FT-IR spectra with ¹H-NMR for some of them, compounds (1-6) Fig(1-5) shown absorption bands of FT-IR Spectra appearance for (C=O) imide at (1742, 1748, 1750, 1750, 1756) cm⁻¹, while of (C=C) aromatic appearance at (1541, 1620, 1601, 1604) and (C-H) aromatic appearance at (3045, 3030, 3014, 3064) and absorption bands of (C=N) and (C=C) vinyl at (1600-1620) cm⁻¹ and (C=O) acid chloride at(1776) cm⁻¹ and (C-H) aliphatic, while compound (6) shown disappearance of C=C olefinic band at 1620 cm⁻¹, other details are show in table 2.





Scheme 1

Table 2: FT-IR data of prepared compounds

pmoC.	ν (cm^{-1})							Fig No.
	(C = C) Arom.	(C - H) Arom.	(C=O) Imide	(C = C) olef.	(C - H) Alip.	(C=O) Acid chloride	(C-N)	
1	1450, 1495, 1590	3100	1742	-	2890	-	-	1
2	1450, 1485, 1604	3055	1772	-	2952	1890	1367	2
3	1480, 1590, 1601	3087	1750	-	2918, 2987	C-Cl 735	1320	3
4	1400, 1559	3030	1750	1620	2970	-	1360	4
6	1541, 1590	3014	1745	-	2970	-	1305	5

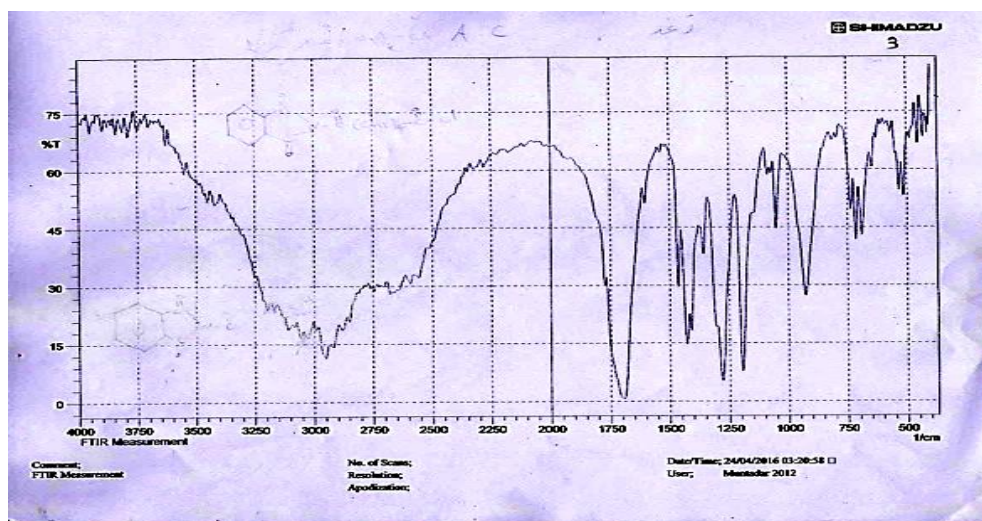


Figure 1: FT-IR spectrum of compound 1



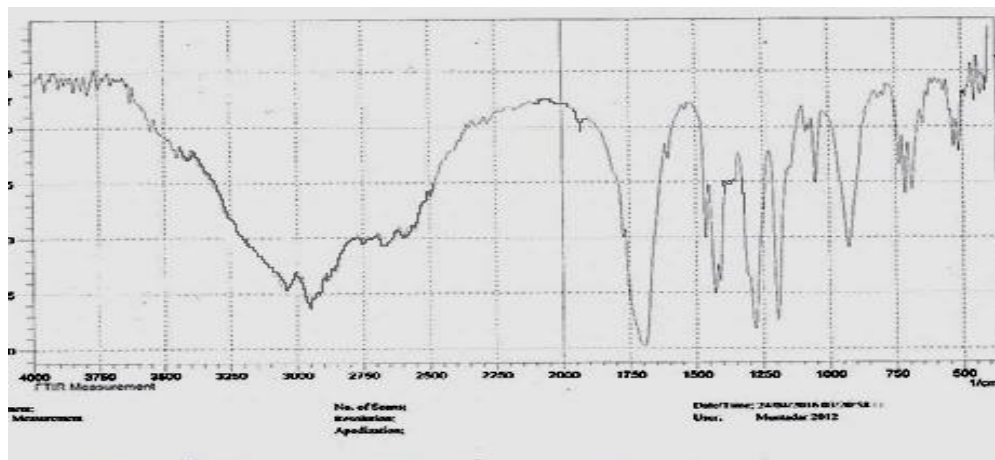


Figure 2: FT-IR spectrum of compound 2

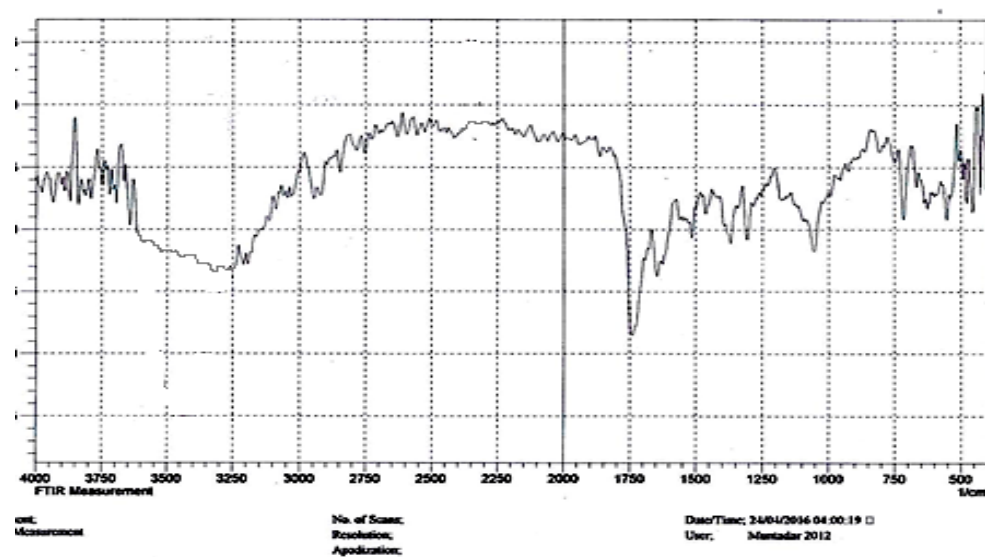


Figure 3: FT-IR spectrum of compound 3

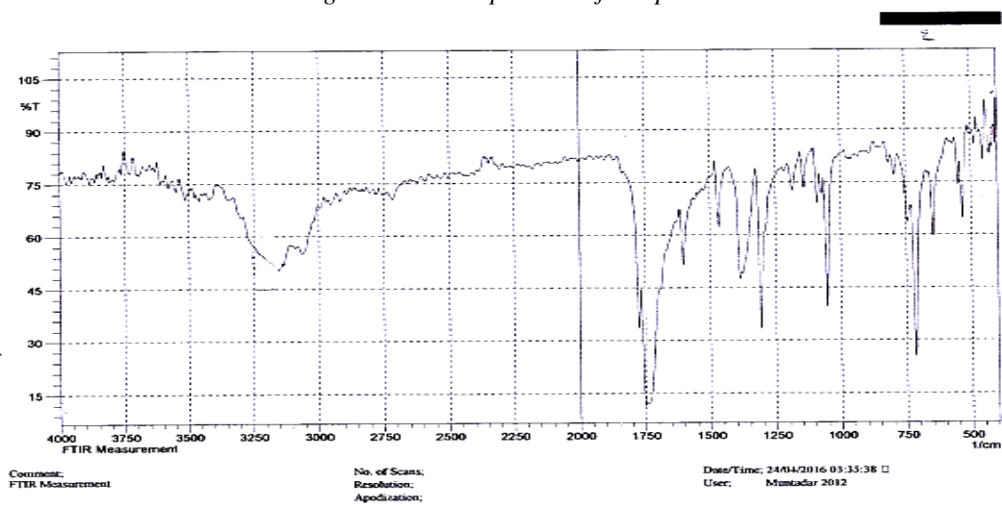


Figure 4: FT-IR spectrum of compound 4



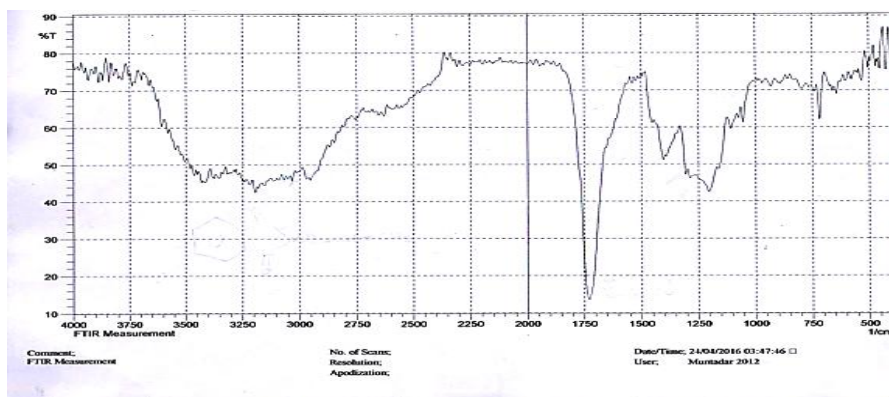
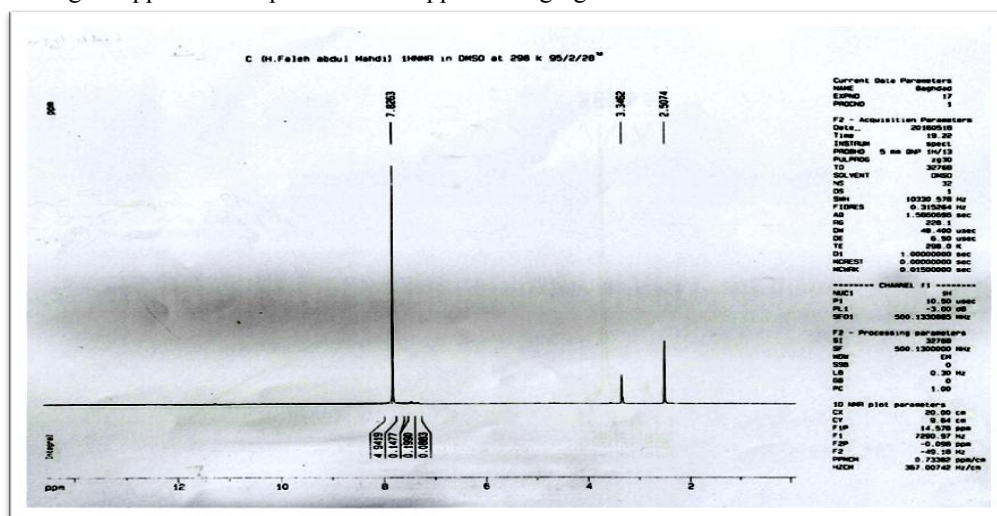
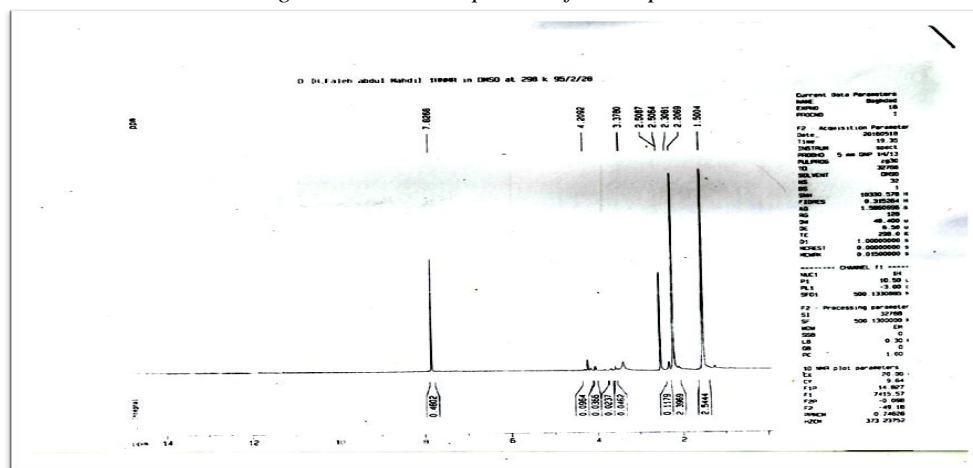


Figure 5: FT-IR spectrum of compound 6

$^1\text{H-NMR}$ spectra of compound (1) shows signal at δ 3.34 ppm, (2H, s, CH_2) high field as expected, other signal at δ 7.82 ppm (9H, s, Ar) Fig (6), while compound (2) shows signal at δ (1.5 -4.2) ppm (8H, (CH_2)₄) and signal at δ 7.82 ppm (4H, s, Ar) Fig (7), compound (3) shows, signals at δ (1.1-4.1) ppm (8H, m, (CH_2)₄) and signal at δ (7.3-8.0) (8H, m, 2ph) Fig(8) .

Note: there are signal appear in all spectra at δ 2.5 ppm belonging to DMSO- d_6 solvent as shown below:

Figure 6: $^1\text{H-NMR}$ spectrum for compound 1Figure 7: $^1\text{H-NMR}$ spectrum for compound (2)

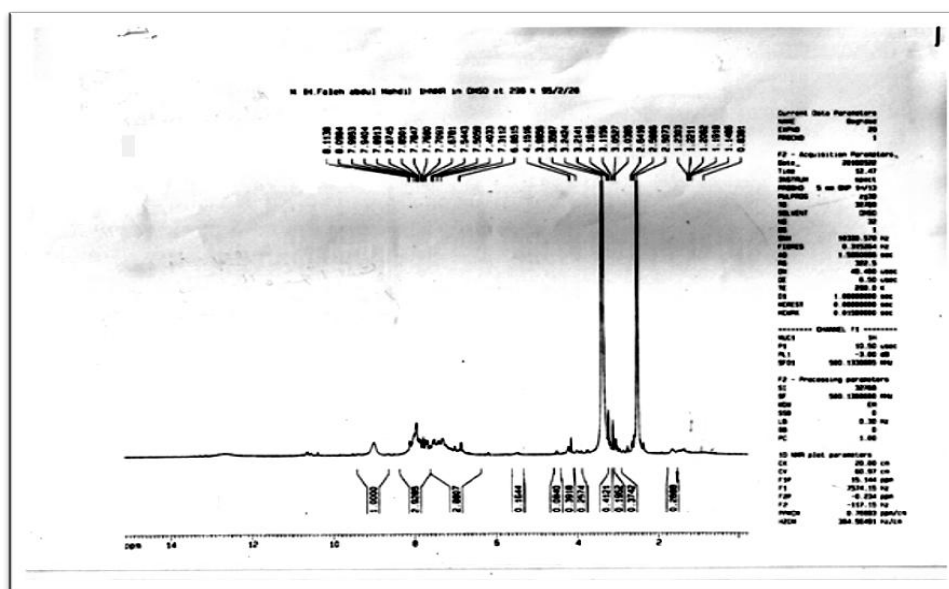


Figure 8: $^1\text{H-NMR}$ spectrum for compound 5

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