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COMPARATIVES STUDIES OF MICROWAVE ASSISTED SYNTHESIS WITH CONVENTIONAL SYNTHESIS OF SOME NOVEL GLUCOSYL THIOBIURETS

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Abstract: *The use of microwave irradiation in organic synthesis has become increasingly popular within the pharmaceutical and academic arenas, because it is a new enabling technology for drug discovery and development. By taking advantage of this efficient source of energy, Presently, thermally driven organic transformations take place by either of two ways: conventional heating or microwave-accelerated heating. The chemistry of thiourea of carbohydrate is extensively elaborated and well documented. These compounds arouse interest as potential biologically active substances and versatile intermediates for preparing various derivatives. This reaction is frequently used as a tool for structure determination. They have been found useful in the treatment of hypertension, as appetite suppersant and as a potential anti oxidant cardio protective agent.Chemistry of sugar isothiocyanate with special reference to their utility as intermediate in the synthesis of nitrogen and sulphur containing open chain and cyclic compound. Several glucosyl thiobiurets deravaives has been prepare by condensation of Tetra-O-acetyl-B-D-glucosyl isocyanate with various aryl thiocarbamides by both convensational and microwave method. The identites of newly synthesis co,mpounds have been established on the basis of usual chemical transformation and IR, NMR, Mass spectral studies.*

Keywords: *Tetra-O-acetyl-B-D-glucosyl isocyanate, Aryl thiocarbamides , glucosyl thiobiurtes. s*

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INTRODUCTION

The use of microwave irradiation in organic synthesis has become increasingly popular within the pharmaceutical and academic arenas, because it is a new enabling technology for drug discovery and development. By taking advantage of this efficient source of energy, compound libraries for lead generation and optimization can be assembled in a fraction of the time required by classical thermal methods. Presently, thermally driven organic transformations take place by either of two ways: conventional heating or microwave- accelerated heating. In the first way, reactants are slowly activated by a conventional external heat source. Heat is driven into the substance, passing first through the walls of the vessel in order to reach the solvent and reactants. This is a slow and inefficient method for transferring energy into the reacting system. In the second way, microwaves couple directly with the molecules of the entire reaction mixture, leading to a rapid rise in temperature. Since the process is not limited by the thermal conductivity of the vessel, the result is an instantaneous localized superheating of any substance that will respond to either dipole rotation or ionic conduction—the two fundamental mechanisms for transferring energy from microwaves to the substance(s) being heated.

Microwave assisted organic synthesis has become an important tool to medicinal chemists for rapid organic synthesis. A huge number of research papers have appeared over the last decades on the application of microwave technology in organic synthesis.[1] Some of the major advantages include spectacular decrease in reaction time, improved conversions, clean product formation and wide scope for the development ofnew reaction conditions.

The use of polymer-supported reagents and scavengers is a powerful technique for expedited synthesis and purification.[2] Rapid transformations using microwave technology has shifted the bottleneck from synthesis to the work-up and purification step. Therefore, chemists are increasingly looking for an expedited synthesis and purification strategy that would combine the use of microwave heating with polymer-assisted solution- phase organic synthesis. This overview [3] covers the recent literature on the significant new applications of polymer-supported reagents and scavengers using microwave heating.

Carbohydrates derivatives have been extensively investigated including synthesis, characterization and biological activity. Partly due to the facts that many natural occurring saccharides and synthesized analogues exhibit various and potent biological activities and they have been widely employed as agrochemicals and pharmaceuticals[4-7].

Because of tremendous biological importance, carbohydrates have aroused much interest to synthetic and medicinal chemistry[8-9].

The N-glucopyranosylated compounds have been known for their great biological importance. They have been found several applications in paper[10], textile[11,12] and food industries[13]. Besides these applications they have been found use as divertic agents, analgesics, antidiabetic compounds, bacteriosatic agents and in many other ways[14]. Some of them have been found to be valuable oxidation dyes[15] for printing and padding the animals and vegetable fibers by standard oxidation dyeing methods. Quite few of them have antitumor and tuberculostatic activity[16].

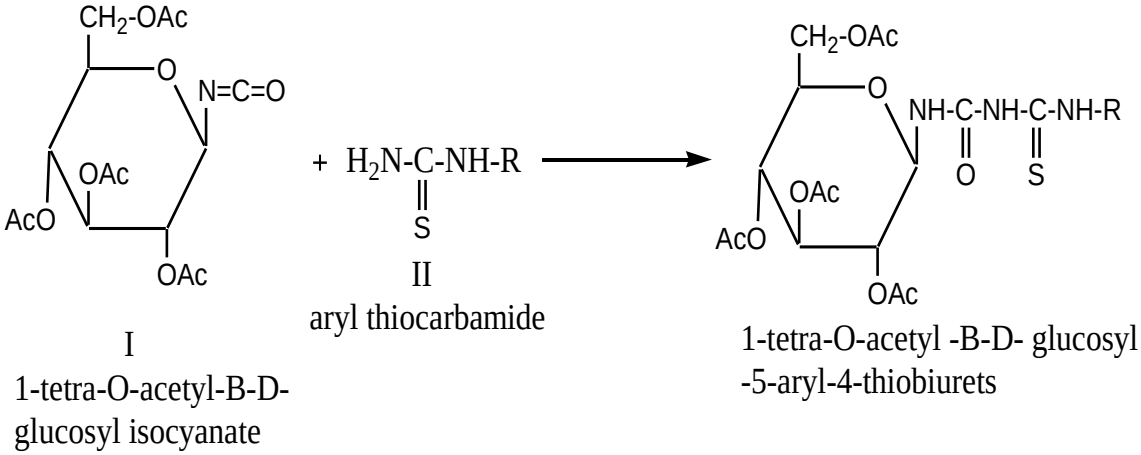
RESULTS AND DISSCUSSION:

The chemistry of thiourea of carbohydrate is extensively elaborated and well documented[1]. These compounds arouse interest as potential biologically active substances and versatile intermediates for preparing various derivatives

Several glucosyl thiobiurets deravaives has been prepare by condensation of Tetra-O-acetyl-B-D-glucosyl isocyanate with various aryl thiocarbamides by both convensational and microwave method. Toluene solution of Tetra-O-acetyl- β -D-glucosyl isocyanate (0.005 M, 1 g) was added to 4amino -1-phenyl thiocarbamide (0.21 gm in 20 ml) and the reaction mixture was under microwave irradiation It was then allowed to cool and pour it in petroleum ether with vigorous stirring; a white granular solid was separated out

This reaction was also carried out by conventational method toluene solution of Tetra-O-acetyl- β -D-glucosyl isocyanate (0.005 M, 1 g) was added to 4amino -1-phenyl thiocarbamide (0.21 gm in 20 ml) and the reaction mixture was reflux for 3hr. It was then allowed to cool and pour it in petroleum ether with vigorous stirring; a white granular solid was separated out. The characterization of products was established by IR, ^{1}HNMR, and MS spectral studies.

Reaction



I
1-tetra-O-acetyl-B-D-glucosyl isocyanate

II
aryl thiocarbamide

1-tetra-O-acetyl -B-D- glucosyl -5-aryl-4-thiobiurets

R= a)1amino-4- phenyl thiocarbamide b)1-amino-3-phenyl thiocarbamides c)1-amino 2-phenyl thiocarbamide

EXPERIMENTAL

Melting points were recorded on electro thermal melting point apparatus are uncorrected. Specificrotations were measured on Equip-Tronic digital polarimeter model no. EQ 800 at 30^0C in $CHCl_3$. IRspectra were recorded on a Perkin Elmer spectrometer. ^{1}H NMR were obtained on a Bruker DRX-300(300 MHz FT NMR) NMR spectrometer in $CDCl_3$ solution with TMS as an internal reference. The massspectra were recorded on a DART mass spectrometerwere recorded. Purity of the compounds was checkedby thin layer chromatography using Merck silica gel coated aluminum plates and petroleum ether: ethylacetate as eluent.

Synthesis of 1-tetra-O-acetyl-β-D-glucosyl-5-p-amino phenyl-4- thiobiurtes

Toluene solution of Tetra-O-acetyl- β -D-glucosyl isocyanate (0.005 M, 1 g) was added to 4amino -1-phenyl thiocarbamide (0.21 gm in 20 ml) and the reaction mixture was under microwave irradiation It was then allowed to cool and pour it in petroleum ether with vigorous stirring; a white granular solid was separated out, crystallized from aqueous ethnaol, m.p. 95°C.

It was found soluble in alcohols acetone, chloroform and benzene while insoluble in water and petroleum ether. It charred when warmed with conc. sulphuric acid. The specific rotation was found to be $[\alpha]_D^{35}$ = - 136° (c, 0.74 in chloroform). The purity was checked by TLC, and recorded Rf value 0.62(Hexene: EtOAc 3:2.1)

ANALYTICAL AND SPECTRAL DATA OF COMPOUNDS:

1) Synthesis of 1-tetra-O-acetyl-β-D-glucosyl-5-p-amino phenyl-4- thiobiurtes

Yield 72 (%); Mp.95^{0}C;[□]$_D^{32}$136 (0.1, in $CHCl_3$);R*f* (Hexane:EtOAC)(1:1)0.62;**IR (KBr)cm- 1**:ν 3023-3292 (Ar-H)str ,1750 (C=O)str, 1543(C=N) str, , 1411 (C-N)str, 927(char. of glucopyranosyl ring), 758 (C=S) str. **^{1}HNMR (CDCl3)ppm:** 7.46-6.32 (m,8H, Ar-H), 5.57-3.87 (m, 7H, glucosyl-H), 2.31-2.01 (m, 12H,OAc),. **MS**(m/z) : 651 (M+),521, 408, 331, 263, 261, 169, 108.(Anal.Calcd. For $C_{29}H_{30}O_{10}N_3S$: C 53.78, H 4.63, O 24.72, N 6.24, S 4.98 Found C 53.76, H 4.60, O 24.71, N 6.22, S 5.0 %).

2) Synthesis of 1-tetra-O-acetyl-β-D-glucosyl-5-m-amino phenyl -4-thiobiuret

Yield 80 (%); Mp.145-150^{0}C;[□]$_D^{32}$155 (0.1, in $CHCl_3$);R*f* (Hexane:EtOAC)(1:1)0.62;**IR (KBr)cm-1**:ν 3000-3292 (Ar-H)str ,1755 (C=O)str, 1543(C=N) str, , 1425 (C-N)str, 927(char. of glucopyranosyl ring), 758 (C=S) str. **^{1}HNMR (CDCl3)ppm:** 7.46-6.32 (m,8H, Ar-H), 5.57-3.87 (m, 7H, glucosyl-H), 2.31-2.01 (m, 12H,OAc),. **MS**(m/z) : 651 (M+),521, 408, 331, 263, 261, 169, 108.(Anal.Calcd. For $C_{29}H_{30}O_{10}N_4S$: C 53.78, H 4.63, O 24.72, N 5.02, S 4.98 Found C 53.76, H 4.60, O 24.71, N 5.47, S 5.0 %).

3) Synthesis of 1-tetra-O-acetyl-β-D-glucosyl-5-o-amino phenyl -4-thiobiuret

Yield 71 (%); Mp.122^{0}C;[□]$_D^{32}$142 (0.1, in $CHCl_3$);R*f* (Hexane:EtOAC)(1:1)0.59;**IR (KBr)cm- 1**:ν 3010-3289 (Ar-H)str ,1718 (C=O)str, 1543(C=N) str, , 1435 (C-N)str, 927(char. of glucopyranosyl ring), 758 (C=S) str. **^{1}HNMR (CDCl3)ppm:** 7.46-6.32 (m,8H, Ar-H), 5.57-3.87 (m, 7H, glucosyl-H), 2.31-2.01 (m, 12H,OAc),. **MS**(m/z) : 651 (M+),521, 408, 331, 263, 261, 169, 108.(Anal.Calcd. For $C_{29}H_{30}O_{10}N_4S$: C 53.78, H 4.63, O 24.72, N 5.02, S 4.98 Found C 53.76, H 4.60, O 24.71, N 5.47, S 5.0 %).

Table No.4:- Comparative study of conventional and microwave assisted synthesis of 1-tetra-O-acetyl-β-D-glucosyl-5-aryl-4-thiobiurets

Sr.No.	-tetra-O-acetyl-β-D-glucosyl-5-aryl -4- thiobiurtes	M.P. °C	Conventional method		Microwave Method	
			Time (hrs)	% Yield	Time (min.)	% Yield
1	-tetra-O-acetyl-β-D-glucosyl-5-p-amino phenyl-4-thiobiurtes	95	4	65.12	15	**72.80**
2	1-tetra-O-acetyl-β-D-glucosyl-5-m-amino phenyl -4-thiobiuret	145 – 150	4	70,07	15	**80.50**
3	1-tetra-O-acetyl-β-D-glucosyl-5-o-amino phenyl -4-thiobiuret	**122**	**4**	**60.31**	**15**	**71.60**

From above comparative study it is conclude that microwave assisted synthesis is faster and gives cleaner product with higher yield than conventional.

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