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Synthesis of Some Novel Organic N-Lactosylated Thio Carbamides Nanoparticles

Poonam T. Agrawal
P.G. Department of Chemistry, Shri R.L.T. College of Science, Akola-444001 (M.S.)
Email address: poonamagrawal2575@rediffmail.com

Abstract:-

Nanoparticles are of great scientific interest as they are effectively a bridge between bulk materials and atomic or molecular structures. Nanoparticles research is currently an area of intense scientific research, due to a wide variety of potential application in biomedical, optical and electronic fields. In view of this it appeared quite interesting to prepared Nanoparticles of organic compounds containing carbohydrates

Key words: Lactosyl thiocarbamides, Nanoparticles and Antimicrobial activity.

Introduction:

Nanoparticles are at the leading edge of the rapidly developing field of nanotechnology .Their unique size depended properties make these materials superior and indispensable in many areas of human activity. In recent years, nanoparticles are gaining importance due to their unique properties and their antimicrobial activities that are significantly different from those of bulk materials [1].

Nanostructure materials are attracting a great deal of attention because of their potential for achieving specific processes and selectivity, especially in biological and pharmaceutical applications [2]. Recent studies have demonstrated that especially formulated nanoparticles have good antibacterial activity [3,4].

In view of applications of lactosyl thiocarbamides and the nanoparticles in medicinal chemistry and in many other ways[5], we herein report the synthesis of 1-hepta-o-benzoyl β-D-lactosyl-3-aryl- thiocarbamides nanoparticles by the use of ultrasonicator.

Experimental:

Specific rotations were measured on Equip-Tronics Digital Polarimeter at 28 ^{0} C in $CHCl_3$. IRspectra were recorded on Perkin-Elmer spectrum RXI FTIR spectrophotometer (4000-450 cm^{-1}). ^1H NMR was recorded in $CDCl_3$ on Bruker DRX-300 spectrometer operating at 300 MHz. The mass spectra were recorded on Jeol-SX-102(FAB) instrument.

a) Preparation of lactose octabenzoate:

In a 1 litre bottle having a tight cork, 55 ml dry pyridine and 55 ml of dry chloroform was taken. The bottle was cooled in an ice-salt bath. Now to this solution previously prepared cooled solution of 55 ml of benzoyl chloride in 55 ml dry chloroform was added with constant stirring. To this solution 20 gm of dry powder of lactose was added in several installments with constant stirring and maintaining the temperature of the reaction mixture below 5^{0} C. This solution was allowed to stand for 24 hr, it was then transferred to a 500 ml conical flask. The solution was washed several times with dilute aqueous sulphuric acid, water. The solution layer was separated by separating funnel. Afterwards the chloroform was removed , a white precipitate was isolated with petroleum ether and purified with chloroform ether with m.p.114^{o} C.

b) Preparation of brominating reagent:

Glacial acetic acid (30 ml) was taken in a conical flask and to it was added red phosphorous (3.0 gm). To this mixture molecular bromine (7 ml) was added gradually with constant shaking and cooling. The resultant mixture was allowed to stand at ice cold temperature for about a 30 min.

c) Synthesis of hepta-O-benzoyl-α-D-lactosyl bromide:

The finally powdered lactose octabenzoate(0.03M, 21.0g) was added gradually to the brominating agent. After the addition the flask was kept for 2hr at room temperature. Then the reaction mixture with chloroform (130ml) then the mixture was shaken vigorously for about 15 min. The resultant mixture was poured into ice cold water. The chloroform layer was then separated. It was washed several with aqueous sodium bicarbonate to remove excess of acetic acid followed by aqueous sodium metabisulphite to remove excess of bromine and finally 2-3 times with water. To the chloroform addition of petroleum ether afforded a solid (16.5 gm). This solid was expected hepta-O-benzoyl-α-D-lactosyl bromide (yield 77%). It was purified by dissolving it in minimum quantity of chloroform and reprecipitating it with petroleum ether m.p.168^{o}C.

(2)Preparation of lead thiocyanate :

Lead thiocyanate was prepared by mixing aqueous solution of lead nitrate and ammonium thiocyanate. The white granular lead thiocyanate was filtered washed with distilled water and dried at 50^{o} C.

(3)Preparation of hepta-O-benzoyl-β-D-lactosyl isothiocyanate :

To a suspension of hepta-O-benzoyl-α-D-lactosyl bromide (21 gm,0.03M) in sodium dried xylene (80ml) was added lead thiocyanate (6gm,0.03M). The reaction mixture was then treated for microwave synthesis for about 3 min. This solution was then cooled and liberated lead bromide was removed by filteration . The xylene filterate was then treated with petroleum ether

(60-80°C) with stirring , a white solid mass obtained (13gm). This solid was expected hepta-O-benzoyl- β-D-lactosyl isothiocyanate.

It was purified by dissolving it in minimum quantity of chloroform and reprecipitating it with petroleum ether, m.p 118-120°C. [found; C;67.07, H;4.46, N;1.22, S;2.9; $C_{72}H_{49}O_{17}NS$ requires; C;66.96, H;4.41, N;1.26, S;2.88%].

(4)Preparation of Nanoparticles of hepta-O-benzoyl-β-D-lactosyl 3-phenyl thiocarbamide:

Take about 1 gm of hepta-O-benzoyl-α-D-lactosyl 3-aryl thiocarbamide and dissolve it completely in the 50ml of solvent in 250 ml beaker. Now put this beaker in sonicator. The highly penetrating acoustic waves are passed through mixture, which create high pressure bubbles in the beaker due to which breakdown of the bulk material is takes place and desired sized nanoparticles are formed. The size determination of nanoparticles are done by the X-ray diffraction studies.

IR SPECTRUM OF 1-HEPTA-O-BENZOYL-β-D-LACTOSYL -3-PHENYL THIOCARBAMIDES:

Absorption observed (Cm^{-1})	Assignment	Absorption Expected (Cm^{-1})
3066	C-H Ar Stretching	3040-3010
1729	C=O stretching	1750-1735
1176	C-O Stretching	1210-1153
1068,909	Charecterstic of Lactose	1100-1000 and 910-900

NMR SPECTRAL STUDIES: The NMR Spectrum [7,8] of compound distinctly displayed signals due, Aromatic Protons at δ 7.47-7.15 ppm, lactosyl protons at δ 5.77-3.76 ppm.

Characterisation of Nanoparticles:

1. **Charterisation using UV - Visible Spectrophotometer:** Characteirisation of nanoparticles was done using visible Spectrophotomter by using model Single Beam UV-Visible Spectrophotometer with software[BI/CI/SP/SB-S-03]of Bio Era make. The UV-Visible Spectrocopy reveals the formation of nanoparticles by showing different absorption those from bulk materials

2. **Size determination of Lactose Octabenzoate Nanoparticle by X-Ray Diffraction Studies:** From the X-Ray diffraction it comes to know that size of nanoparticles are as follows.

Compound	Size of Nanoparticles in nm
1-hepta-O-benzoyl-β-D-lactosyl -3 phenyl thiocarbamide	204
1-hepta-O-benzoyl-β-D-lactosyl -3 -chloro-phenyl thiocarbamide	230
1-hepta-O-benzoyl-β-D-lactosyl -3 tolyl thiocarbamide	300

ANTIMICROBIAL ACTIVITY COMPARISON :

The bulk Lactosyl thiocarbamides and the Nanopartices of Lactosyl thiocarbamides have been screened for antibacterial activity using cup plate agar diffusion methodby measuring the inhibition zone in mm. The compounds were taken at a concentration of 1 mg/ ml using dimethyl sulphoxide as solvent. Amikacin (100ug/ml) was used as a standard for antibacterial activity. The compounds were screened for antibacterial activity against *Escherichia coli*, *Staphylococcus aureus*, in nutrient agar medium.

Antimicrobials	Zone of inhibition in mm					
	Bulk (5a)	Nanoparticles (6a)	Bulk (5b)	Nanoparticles (6b)	Bulk (5c)	Nanoparticles (6c)
E. coli	10	14	12	16	11	15
S. aureus	10	16	11	18	12	18
S. typhi	11	15	12	15	10	16
P. vulgaris	11	15	12	14	10	14
Amikacin	10	20	10	18	12	18
Clandamycine	12	14	11	15	10	15
DMSO	35	28	15	20	20	25

*including the well diameter of 8mm. ** zone of inhibition in mm (15or less) resistance, (16-20mm) moderate and (more than 20mm) sensitive

Conclusion: Lactosyl thiocarbamides Nanoparticles show good antimicrobial activity as compare to the bulk solution of lactosyl thiocarbamides due to their large surface area to volume ratio, which is coming up current interest in the researchers.

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