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A Systematic Investigation of Nanoparticle Synthesis of 1-Tetra-O-Acetyl –B-D-Glucosyl-3-aryl Thiocarbamide

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Abstract:-

In recent years, nanotechnology is an escalating field of modern research involving synthesis design, characterization, production, and application of structures, devices, and systems by controlling shape and size at the nanometer scale. Nanotechnology also involves the synthesis of nanoparticles. These compounds arouse interest as potential biologically active substances and versatile intermediates for preparing various derivatives. To achieve the principle of green chemistry process, it leads to the search for green synthesis of nanoparticles. Here we have synthesized 1-Tetra-O-acetyl-B-D-glucosyl -3-aryl thiocarbamide by reaction of Tetra-O-Acetyl-B-D-glucosyl isothiocyanate with various aryl amines The identities of newly synthesis compounds have been established based on usual chemical transformation and U.V, IR, NMR, Mass and Particle Size analysis Analytical studies.

Keywords: TAG Isothiocyanate , Aryl Amines and Tetra-O-acetyl-B-D-glucosyl -3-aryl thiocarbamides nanoparticles.

Introduction:-

Described as the manipulation of atomic matter, nanotechnology was described theoretically in the 1960s by Richard Feynman, and the practice emerged a decade later. After Taniguchi's, Drexler's, and other scientist's valuable contributions, nanomedicine has developed [1,2] and recently, the three main applications of nanomedicine are in tissue engineering, nanoprobes, and nanoparticles for drug delivery. The field of nanotechnology is one of the most active research areas in modern materials science. Nanoparticles exhibit new or improved properties based on specific characteristics such as size, distribution, and morphology. There have been impressive developments in the field of nanotechnology in the recent past years, with numerous methodologies developed to synthesize nanoparticles of particular shape and size depending on specific requirements. New applications of nanoparticles and nanomaterials are increasing rapidly.

Nanotechnology, as defined by size, is naturally very broad, including the field of science as diverse as surface science, organic chemistry, molecular biology, semiconductor physics, energy storage, microfabrication, molecular engineering, etc. The associated research and applications are equally diverse, ranging from extensions of conventional device physics to completely new approaches based upon molecular self-assembly, from developing new materials with dimensions on the nanoscale to direct control of matter on the atomic scale. Nanotechnology may create many new materials and devices with various applications, such as in Nanomedicines, Nanoelectronics, and biomaterial energy production and consumer products.

Lipid-based Nanoencapsulation systems are useful in the properties of antioxidants. It enhances the performance of antioxidants just by improving their solubility. Antioxidants

protect our body against age-related, and chronic diseases. When antioxidants are given in their free form, they cannot pass cell membranes, so can easily be cleared from the general circulations reason behind the usefulness of nanocapsulation.[3]

Carbohydrates are important, abundant, and a fundamental class of biomolecules containing Carbon, Hydrogen, and oxygen. The old view on carbohydrate as a natural energy source (starch and glycogen) and structural material (e.g., cellulose, collagen, proteoglycans, and DNA backbone) have expanded, and it is a fact that the role of carbohydrate is much more sophisticated and complex. Today carbohydrates are known to have a variety of functions in mammals [4-5]. Carbohydrates play an essential role in a vast array of biological processes, and mainly there are many advantages; for example, carbohydrate-based drugs show low toxicity and immunogenicity[6]. Thus because of biological importance, carbohydrates have aroused much interest in synthetic and medicinal chemistry[7, 8].

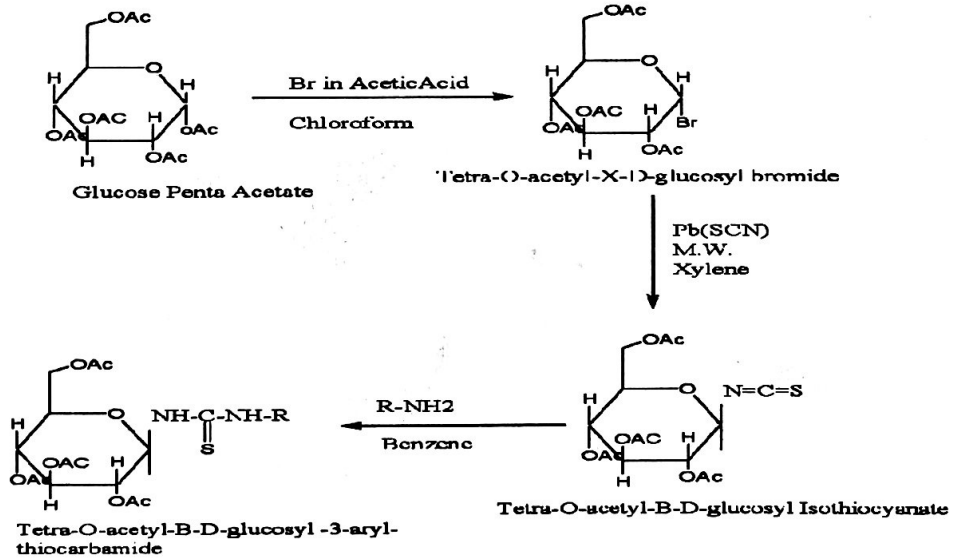
Carbohydrates derivatives have been extensively investigated, including synthesis, characterization, and biological activity. Partly due to the fact that many naturally occurring saccharides and synthesized analogs exhibit various and potent biological activities, and they have been widely employed as agrochemicals and pharmaceuticals[9-10].

Results And Discussion:

Nanoparticles:

A nanoparticle isa sub-classification of the ultrafine particle with lengths in two or three dimensions greater than 0.001 micrometer (1 nanometer) and smaller than about 0.1 micrometer (100 nanometers) and which may or may not exhibit a size-related intensive property. This term is a subject of controversy regarding the size range and the presence of a size-related property. Current usage emphasizes size and not properties in the definition. The length scale may be a hydrodynamic diameter or a geometric length appropriate to the intended use of the nanoparticle. The chemistry of thiourea of carbohydrates is extensively elaborated and well documented. These compounds arouse interest as potential biologically active substances and versatile intermediates for preparing various derivatives

Reaction Scheme:-



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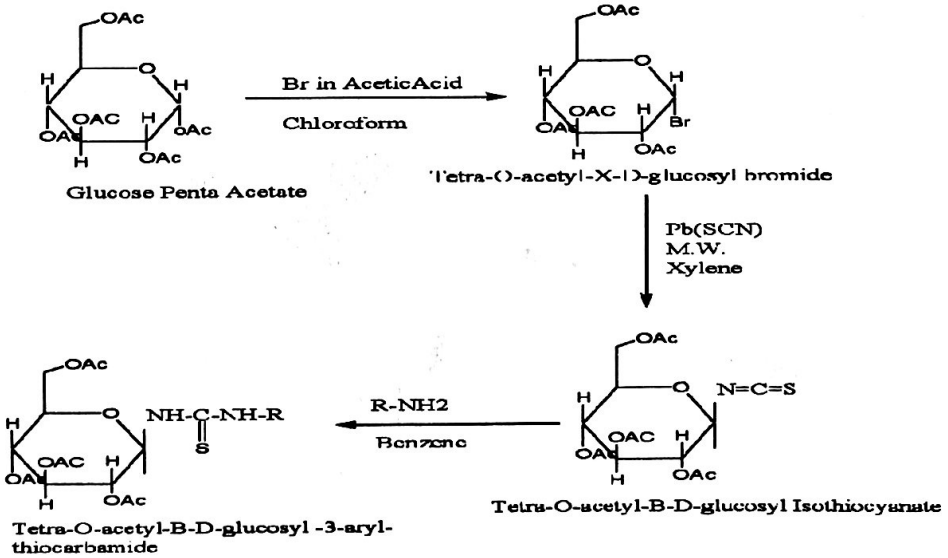

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aryl amines in benzene medium after that reaction mixture was titurate several times with petroleum ether . The product is confirmed based on the melting point and other studies.

Experimental:

Melting points recorded on electrothermal melting point apparatus are uncorrected. Specific Rotations 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 mass spectra were recorded on a DART mass spectrometer. Particle Size was analyzed by Malvern particle size analyzer.

I) Preparation of Tetra-O-acetyl-β-D-glucopyranosyl isothiocyanate :

This has been prepared by the interaction of tetra-O-acetyl-□-D-glucopyranosyl bromide and lead thiocyanate, the former was prepared according to the procedure described earlier. Details of typical experimental are as follows :

a) Microwave assisted preparation of glucose penta acetate :-

Peracetylation of glucose to give the acetyl derivative with small excess of acetic anhydride under the catalyst of either Potassium or Sodium acetate (anhydrous) was found practically quantitative in less than 15 min with microwave heating.

Herein, we reported first time peracetylation of glucose in molecular proportion of acetic anhydride (30 ml) using catalyst sodium acetate 0.8 gm. Under Microwave heating the reaction was complete in less than 10 min. Product was isolated by pouring in ice cold water with constant stirring and cooling.

The glucose penta acetate is separated out; purification of the product was done under a water ethanol system. Melting point of Glucose penta acetate was found to be 110°C.

b) **Synthesis of Tetra-O-acetyl-□-D-glucopyranosyl bromide** :

The finely powdered glucose pentaacetate (21.0g) was added gradually to the brominating reagent. After the addition the flask was kept for 2 hr. at room temperature. The reaction mixture was mixed with chloroform (50 ml) then the mixture was shaken vigorously for about 15 min. The resultant mixture was poured in ice cold water.

The chloroform layer was then separated. It was washed several time with aqueous sodium bicarbonate to removed excess of acetic acid followed by the aqueous sodium meta-bisulphate to remove the excess of bromine and finely 2-3 times with water. To the chloroform layer addition of petroleum ether afforded a solid (15 g). This solid was expected to be tetra-O-acetyl-ά-D-glucopyranosyl bromide, it was crystallized from ethanol, m.p. 88-90°C.

d) **Preparation of lead thiocyanate :**

Lead thiocyanate was prepared by mixing aqueous solution of lead nitrate and ammonium thiocyanate. The white granular lead cyanate was filtered, washed with distilled water and dried at 50°C.

Preparation of Tetra-O-acetyl-β-D-glucopyranosyl isothiocyanate :

To a suspension of tetra-O-acetyl-□-D-glucopyranosyl bromide (21 g) in sodium dried xylene (80 ml) was added lead cyanate (15g). The reaction mixture was refluxed gently for 3 hr. with frequent shaking. This solution was then cooled and liberated lead bromide was removed by filtration. The xylene filtrate was then treated with petroleum ether (60-80°) with stirring, a pale

yellow solid obtained (12 g). This solid was expected to be tetra-O-acetyl-β-D-glucopyranosyl isocyanate. It was purified by dissolving it in a minimum quantity of chloroform and reprecipitating it with petroleum ether. m.p 115-120°C

Table No-2-: Study of synthesis of 1-tetra-*O*-acetyl-β-D-Glucosyl isothiocyanate under microwave irradiation[11]

Sr. No.	Amount of G-Bromide	Amount of Xylene	Time	Power in watt	Temp. °C	% Yield
1	10.0 gm	80 ml	35 min	P-70	120-130	90%
2	20.0 gm	120 ml	40 min	P-80	130-140	80%
3	30.0 gm	150 ml	40 min	P-80	135-145	65%

- Lead thiocyanate was taken in equimolar proportion of G-Bromide.
- G-Bromide :- 1-tetra-*O*-acetyl-□-D-Glucosyl-Bromide

Experiment No. 1:- synthesis of 1- tetra-*O*-acetyl-β-D-glucosyl-3-p-amino phenyl thiocarbamides

Benzene solution of 1-tetra-O-acetyl-□-D-glucopyranosyl isothiocyanate (0.005 M, 1.0 g in 20 ml) was added to benzene solution of 1,4 phenyl diamine (0.005 M, 0.35 g in 10 ml) and reaction mixture was kept under microwave irradiation . Afterwards, solvent benzene was removed by distillation and resultant syrupy mass was triturated several times with petroleum ether, a granular solid was obtained, crystallized from ethanol-water, m.p. 162-167°C.

The product was found soluble in ethanol, acetone, chloroform and benzene while insoluble in water and petroleum ether. It charred on heating with conc. sulphuric acid. It was found non-desulphurised when boiled with alkaline plumbite solution. The product was optically active and its specific rotation was found to be $[\square]_D^{28}$ = 125.20° (c, 0.96 in chloroform). The purity of the product was checked by TLC, Rf value 0.93 (CCl_4 : EtOAc, 3:2).

Analytical and Spectral Data of Compounds:

1) Synthesis of 1-tetra-O-acetyl-β-D-glucosyl-3-*p*-amino phenyl thiocarbamide

Yield 71 (%); Mp.162-167^{0}C;$[\alpha]_D^{32}$125.42°(0.1,in $CHCl_3$);R*f* (Hexane:EtOAC)(1:1)0.93; **IR (KBr)cm- 1**:ν 3373 (N-H)str 3055 (Ar-H)str ,1745 (C=O)str, 1624(C=N) str, 758 (C=S) str , 1242 (C-N)str, 939(char. of glucopyranosyl ring), 900 (mono Substituted Benzene) str.. **^{1}HNMR (CDCl3)ppm:** 7.49-7.19 (m,8H, Ar-H), 5.17-5.99 (m, 7H, glucosyl-H), 2.31-2.01 (m, 12H,OAc),. **MS**(m/z) : 498 (M+)Not located ,387, 331, 169, 108.

Preparation of Nanoparticles of 1-tetra-O-acetyl-β-D-glucosyl-3-*p*-amino phenyl thiocarbamide:

Take about 1 gm of 1-tetra-O-acetyl-β-D-glucosyl-3-*p*-amino phenyl thiocarbamide and dissolve it completely in the 20ml of solvent in a 250 ml beaker and add polyvinyl alcohol as a stabilizer 1.5ml . Now put this beaker in a sonicator. The highly penetrating acoustic waves are passed through the mixture, which creates high-pressure bubbles in the beaker due to which breakdown of the bulk material took place and desired sized nanoparticles are formed. Then stirred mixture about 6hr. in a magnetic stirrer at room temperature. The size determination of nanoparticles is done by the particle size analyzer studies

Characterization of Nanoparticles:

1. Characterization using UV-Spectrophotometer: Single Beam UV-Spectrophotometer with software BI/CI/SP/SB-S-03 of Bio Era make. The UV-Visible Spectroscopy reveals the