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Synthesis And Evaluation of Nanoparticle OF 3-Oxo-2-Tetra-O-Acetyl-B-D-Glucosyl-4-Aryl-5-Galactosylimino-1,2,4-Thiadizolidines (Hydrochlorides)

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ABSTACT:- Nanoparticles-based chemical reaction are developing as alternative to bulk material based chemical reaction Due to their numerous benefits such as partials are transformed into nano dimension boost chemical activity and photochemical stability and simplicity of introduction of multifunctionality. In this work we propose Nano synthesis of 3-oxo-2-tetra-O-acetyl-β-D-glucosyl-4-aryl-5-galactosylimino 1,2,4 thiadiazolidine (hydrochlorides) have been prepared by the interaction of 1-tetra-O-acetyl- β -D-glucosyl-3-aryl carbamides and N-tetra-O-acetyl- β -D-galactosyl-S-chloro isothiocarbamoyl chloride. All the synthesized compounds were characterized on the basis of elemental analysis and XRD studies. The polarimetric study of the title compounds has been carried out.

KEYWORDS: TAG Isothiocyanate , 1-tetra-O-acetyl- β -D-glucosyl-3-aryl carbamides 1,2,4-Thiadiazole,

I. INTRODUCTION

The N-aryl/alkyl-S-chloro isothiocarbamoyl chlorides were prepared by Ottman and Hooks[1] by the controlled chlorination of related isothiocyanates. Many chemists have thoroughly studied the chemistry of N-phenyl-S-chloro isothiocarbamoyl chloride, which is particularly useful in the synthesis of nitrogen and sulfur containing five and six membered heterocyclic compounds[2-6]. In the modern period, heterocyclic chemicals and medications are related. In the textile industry, it has been demonstrated that 1, 3, 5 thiadiazines and their derivatives have brightening and fiber finishing qualities[7]. Significant pharmacological effects, including spasmolytic, anesthetic, cardiovascular, and hypometabolic effects, have been demonstrated by thiadiazines. They are also employed as therapeutic substances and as fungicidal and insecticidal agents. It has been discovered that heterocyclic compounds have anti-inflammatory, anti-parasitic, anti-tubercular, and antidiabetic properties[8-10]. The wide range of applications of 1,2,4-thiadiazoles as phar macophores in medicinal chemistry has attracted great interest in their synthesis. Since the last assessment of the synthesis and therapeutic uses of these heterocyclic compounds, 1,2,4-thiadiazoles have seen impressive growth in many areas of chemistry. This quick development has expanded our understanding of the nature and behavior of these compounds as well as the 1,2,4-thiadiazole ring system.

II. RELATED WORK

Furthermore, 1,2,4-thiadiazole compounds have lately discovered significant new applications and gained industrial and economic significance. A complement to the current reviews on the chemistry of 1,2,4-thiadiazoles is justified by the importance of this new information[11].

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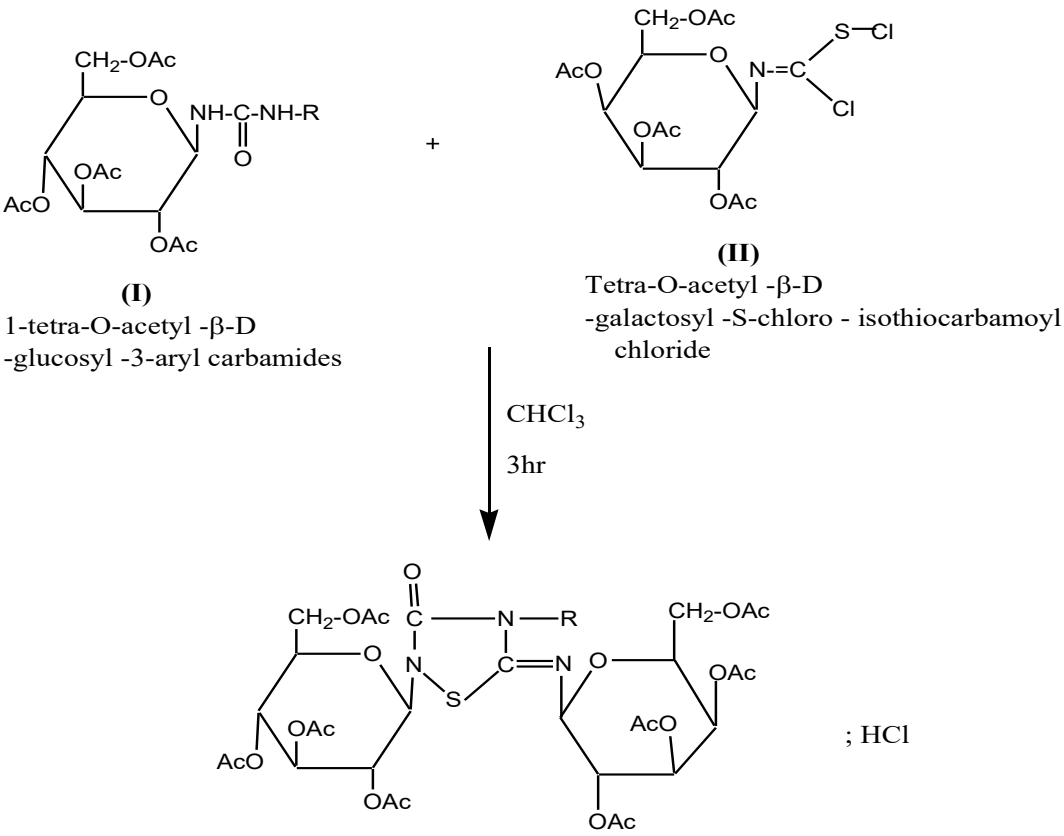
III. METHODOLOGY



(I)
1-tetra-O-acetyl -β-D
-glucosyl -3-aryl carbamides

(II)
Tetra-O-acetyl -β-D
-galactosyl -S-chloro - isothiocarbamoyl chloride

(III)
3-oxo-2-tetra-O-acetyl-β-D-glucosyl-4-aryl-5-tetra-O-acetyl-β-D-galactosylimino -1,2,4- thiadiazolidine(Hydrochloride)

Thiocarbamide was synthesized by methods[12];Tetra-O-acetyl-β-D-glucosyl isothioyanate was react with 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. Then the nanoparticles are prepared with the help of ultra sound sonicator. Particle size and morphological study is done with the help of SEM, TEM at SAIF facility in IIT Bombay.

IV.EXPERIMENTAL

1. Synthesis of 1- tetra-*O*-acetyl-β-D-glucosyl-3-aryl carbamides.

Benzene solution of 1-tetra-O-acetyl-β-D-glucosyl isocyanate (0.005 M, 1.0 g in 20 ml) was added to benzene solution of aniline (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. 95°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-desulphurisable when boiled with alkaline plumbite solution. The product was optically active and its specific rotation was found to be $[\alpha]_D^{28} = 145.20°$ (c, 0.96 in chloroform). The purity of the product was checked by TLC, Rf value 0.93 (CCl_4 : EtOAc, 3:2).

2. Preparation of N-Tetra-O-acetyl- β -D-galactosyl-S-chloro isothiocarbamoyl chloride :

The N-tetra-O-acetyl-β-D-galactosyl-S-chloro isothiocarbamoyl chloride was prepared by the interaction of tetra-O-acetyl- β -D-galactosyl isothiocyanate and calculated quantity of Cl_2 gas. The details of typical experiment are as follows :

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Though the chloroformic solution of tetra-O-acetyl- β -D-galactosyl isothiocyanate (0.1M, 4.0g in 20 ml) pure dry chlorine gas (C 1.9 g) was passed maintaining the temperature at 10°C. The resultant yellow solution was filtered to remove suspended impurities and the clear solution was mixed with petroleum ether (60-80°). The solvent was then removed by distillation under vacuum. The resultant oil was again diluted with petroleum ether and distilled under vacuum. N-tetra-O-acetyl- β -D-galactosyl-S-chloro isothiocarbamoyl chloride was obtained as pale yellow oil.

3. Synthesis of 3-oxo-2-tetra-O-acetyl-β-D-glucosyl-4-aryl-5-galactosylimino 1,2,4 thiadiazolidine

When the interaction of 1-tetra-O-acetyl- β -D-glucosyl-3-phenyl carbamide and N-tetra-O-acetyl- β -D-galactosyl-S-chloro isothiocarbamoyl chloride has been carried out in boiling chloroform medium for 3 hr. Evolution of hydrogen chloride was noticed. After heating solvent was removed by distillation, the syrupy mass was left. The residual mass triturated several times with petroleum ether (60-80°) gave a solid, crystallized from analysis of this product indicated its molecular formula as $C_{36}H_{43}O_{19}N_3S_1$, 2HCl.

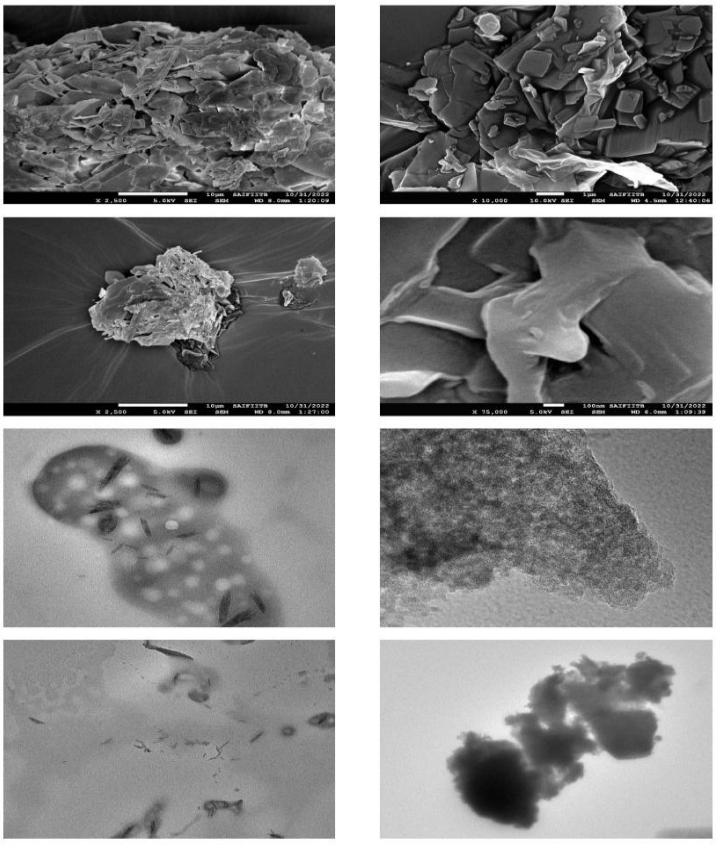
4. Preparation of nanoparticles of 3-oxo-2-tetra-O-acetyl-β-D-glucosyl-4-aryl-5-galactosylimino 1,2,4 thiadiazolidine

Take about 1 gm of 3-oxo-2-tetra-O-acetyl-β-D-glucosyl-4-aryl-5-galactosylimino 1,2,4 thiadiazolidine and dissolve it completely in the 20ml of solvent in a 250 ml beaker and add poly vinyl 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 magnetic stirrer at room tempeture.

V. RESULT AND DISCUSSION

Characterisation of Nanoparticles.

The analysis of particle size and morphology involves utilizing SEM and TEM analyses.(Ref. Fig. 1). To examine nanoparticles depicted in SEM/TEM images, ImageJ open-source software is employed. This software facilitates the calculation of key parameters, including particle size, shape, and distribution. Histograms are plotted for these parameter (Ref. Fig. 2) Results of various parameters are tabulated below in Table No. 1



SEM AND TEM IMAGES OF THE COMPOUND

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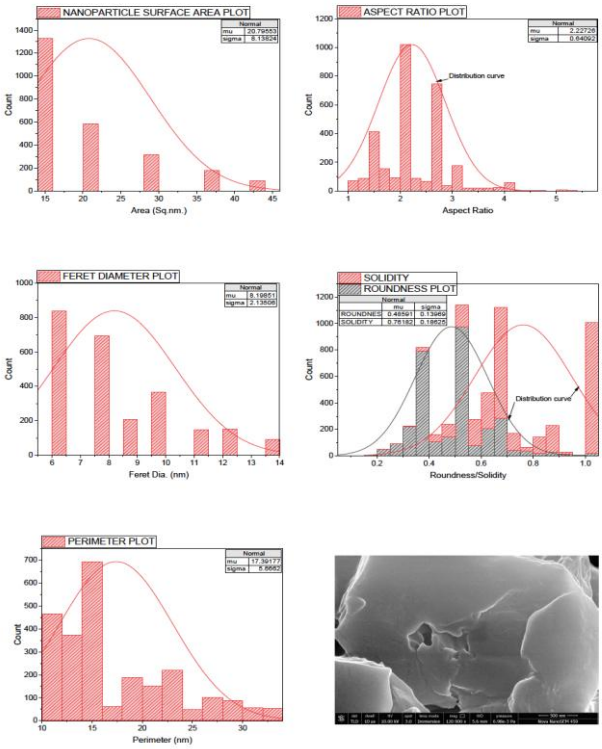

Fig. No. 2 Histogram Of Sample Tem Image Of 3-Oxo-2-Tetra-O-Acetyl-B-D-Glucosyl-4-Aryl-5-Galactosylimino-1,2,4 Thiadiazolidine

Sr.No.	Parameters	Median	Mean Value	Std. Dev.
1	Surface Area	21.91 sq.nm	20.79 sq.nm	8.14
2	Feret's Diameter	8.55 nm	8.20 nm	2.14
3	Feret Angle	116.56 deg	107.13 deg	45.97
4	Perimeter	16.87 nm	17.39 nm	5.67
5	Aspect Ratio	2.00	2.22	0.64
6	Roundness	0.50	0.49	0.14
7	Solidity	0.67	0.76	0.19

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VI.CONCLUSION

From the above table, it can be seen that

The measurements tabulated collectively provide information about the size (area, Feret diameter), orientation (Feret angle), and shape (perimeter) of the nanoparticles.. A higher standard deviation suggests a greater range of parameter values, indicating potential heterogeneity among the nanoparticles. Aspect ratio of 2.22 suggests that the nanoparticles are, on average, somewhat elongated or have a more extended shape. Roundness value of 0.49 suggests that the nanoparticles, on average, have a shape that deviates from a perfect circle. Solidity value of 0.76 suggests that, on average, the nanoparticles have a shape that is somewhat irregular with more convex hulls compared to concavity.The median of the parameters, represents the middle value in the dataset when arranged in ascending order. It can be seen that it is more or less equal to mean value

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