International Journal of Education, Modern Management, Applied Science & Social Science (IJEMMASSS)
ISSN : 2581-9925, Impact Factor: 6.882, Volume 06, No. 01(II), January - March, 2024, pp. 79-84

AN OPERATIVE NANO SYNTHESIS OF 1,2,4 THIADIAZOLIDINE AS A DERIVATIVE OF GLUCOSE ISOTHIOCYANATE

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ABSTRACT

Nanoparticle-based probes are emerging as alternatives to molecular probes due to their various advantages, such as bright and tunable optical property, enhanced chemical and photochemical stability, and ease of introduction of multifunctionality. In this article synthesis of nanoparticle of 1 2 4 -dithiadiazolidine derivative by using Tetra-O-acetyl-B-D-glucosyl thiocarbamide fusion with N-tetra-O-acetyl-β-D-galactosyl-S-chloroisothiocarbamoyl chloride These compounds arouse interest as potential biologically active substances and versatile intermediates for preparing various derivatives The identities of newly synthesis compounds have been established on the basis of usual chemical transformation and U.V, SEM, TEM Analytical studies.

Keywords: *1 2 4 -Dithiadiazolidine, N-tetra-O-acetyl-β-D-galactosyl-S-chloroisothiocarbamoyl Chloride, Glucosyl Thiocarbamides.*

Introduction

Carbohydrates constitute a crucial chemical class, serving as the foundational chemistry for numerous drugs and drug intermediates. Notably, various drugs, including amino glycoside antibiotics, incorporate carbohydrate structures. This chemical class is characterized by its extensive diversity, with organic compounds present in all known plants, animals, and microbial life. Carbohydrates play a pivotal role in providing energy and structural support in plants, while in mammalian tissues, they serve a spectrum of specialized functions[1-5]. These functions encompass activities such as cell and organ differentiation, as well as immune protection for newborns.Of particular interest within the realm of carbohydrates is the synthesis of nitrogen-linked glucosyl compounds. This focus stems from the manifold applications of such compounds in medicinal chemistry and other diverse fields. Sugar isocyanates emerge as versatile synthetic intermediates in carbohydrate chemistry, attracting considerable attention in both synthetic and medicinal chemistry spheres. Nanoparticles, with a size ranging from 1 to 100 nanometers, exhibit distinct characteristics that set them apart from their bulk counterparts. This diminutive scale imparts unique properties to nanoparticles, sparking considerable scientific interest. Their versatility extends across various domains, encompassing electronics, medicine, environmental science, and energy[6-10].

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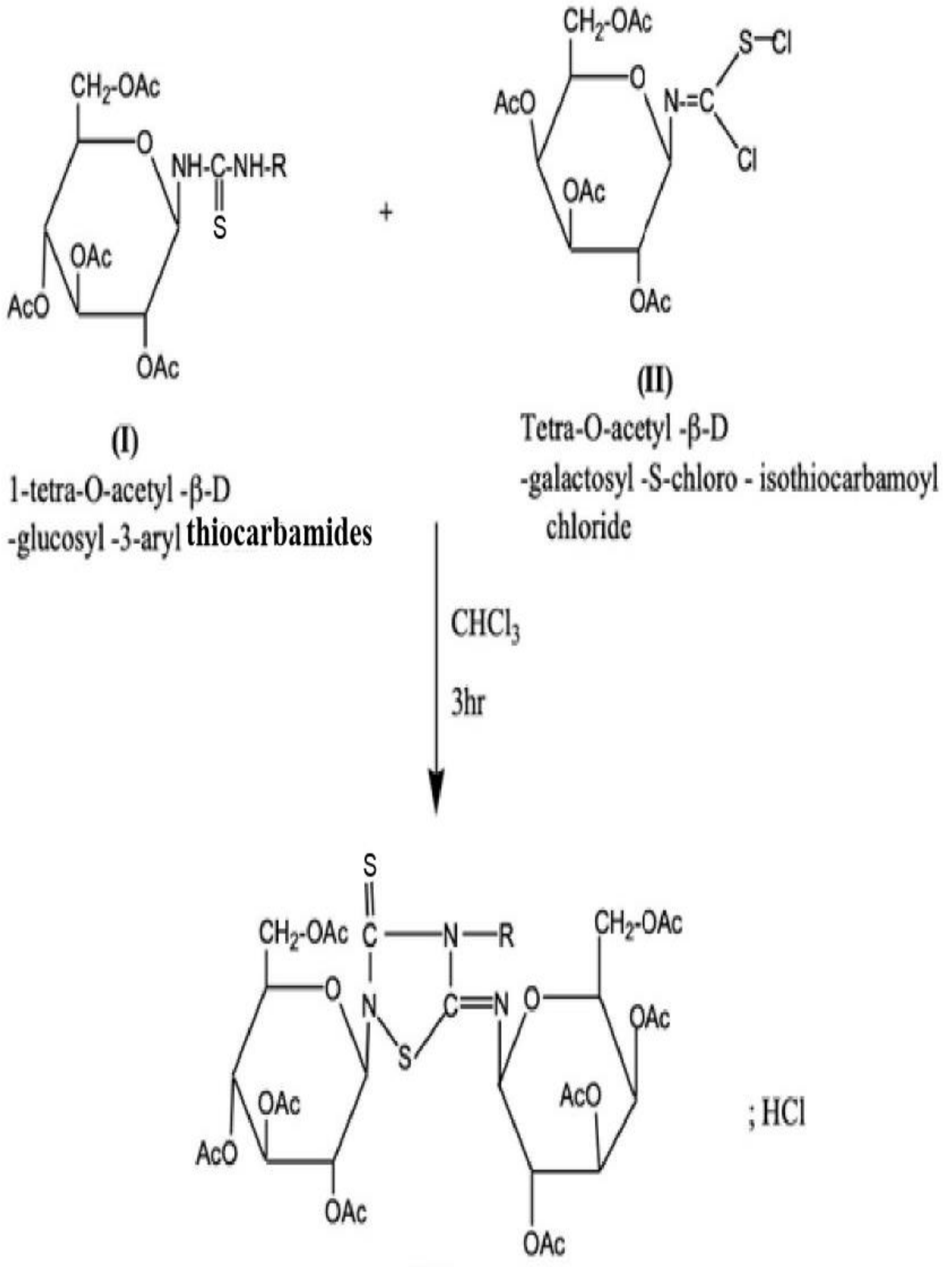
The key to their broad applicability lies in their exceptional properties, notably a high surface area-to-volume ratio. This quality makes nanoparticles valuable tools in diverse fields. In particular, in the realm of medical science, nanoparticles have emerged as versatile and potent assets. Their potential to enhance disease diagnosis and treatment underscores their significance in improving patient outcomes.



(III)

3-oxo-2-tetra-O-acetyl-β-D-glucosyl-4-aryl-5-tetra-O-acetyl-β-D-galactosylimino -1,2,4- dithiadiazolidine

Reaction Schemes

Thiocarbamide[11] was synthesized by both methods; conventional and Microwave method. 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 ultrasound sonicator. Particle size and morphological study is done with the help of SEM, TEM at SAIF facility in IIT Mumbai.

Experimental

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

Benzene solution of 1-tetra-O-acetyl-β-D-glucosyl 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-desulphurisable when boiled with alkaline plumbite solution. The product was optically active, and its specific rotation was found to be $[\alpha]_D^{28}$ = 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).

- **Preparation of N-Tetra-O-acetyl- β -D-galactosyl-S-chloroisothiocarbamoyl Chloride**

The N-tetra-O-acetyl-β-D-galactosyl-S-chloroisothiocarbamoyl 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:

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-chloroisothiocarbamoyl chloride was obtained as pale yellow oil.

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

When the interaction of 1-tetra-O-acetyl- β -D-glucosyl-3-phenyl thiocarbamide and N-tetra-O-acetyl- β -D-galactosyl-S-chloroisothiocarbamoyl 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 $C_{36}H_{43}O_{18}N_3S_2$, 2HCl.

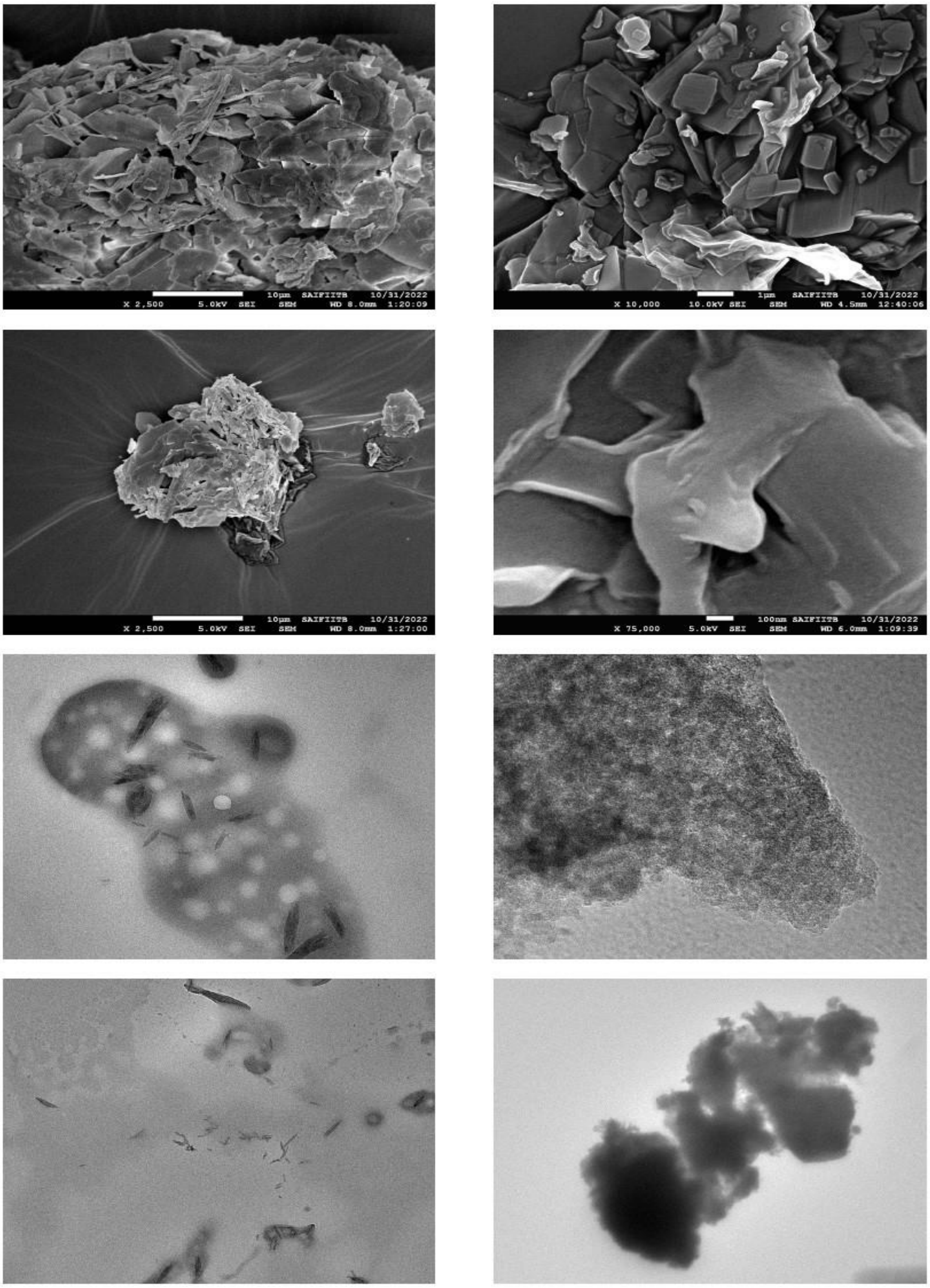
- **Preparation of nanoparticles of 3-oxo-2-tetra-O-acetyl-β-D-glucosyl-4-aryl-5-galactosylimino 1,2,4 dithiadiazolidine**

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 alchole as a stibilizer 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.

Analytical Study of Compound

- **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

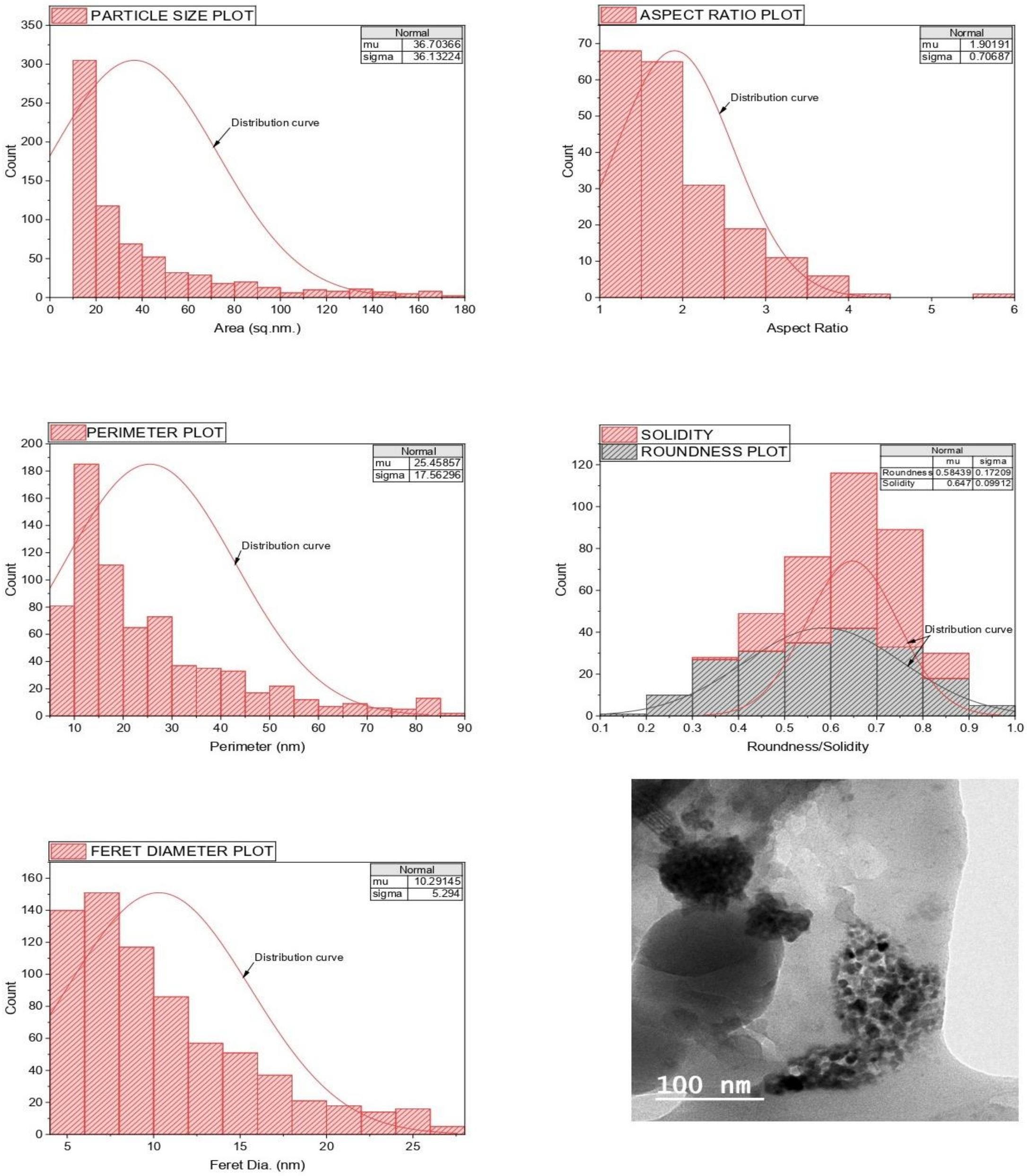


FIG. 2: Histogram of sample tem image of 3-oxo-2-tetra-o-acetyl-β-d-glucosyl-4-aryl-5-galactosylimino-1,2,4 dithiadiazolidine

Table 1: Nanoparticle Charecteristics

Sr. No.	Parameters	Mean Value	Std. Dev.	Median
1	Particle size	36.70 sq.nm	36.13	20.29 sq.nm
2	Feret's Diameter(Long.Cal.)	10.29 nm	5.30	8.12 nm
3	Feret Angle	110.35 deg	45.84	123.69 nm
4	Perimeter	25.46 nm	17.56	19.11 nm
5	Aspect Ratio	1.90	0.71	2.00
6	Roundness	0.58	0.17	0.50
7	Solidity	0.60	0.10	0.70

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. These parameters are valuable for characterizing the morphology and dimensions of nanoparticles in various scientific and research applications.
- Aspect ratio of 1.9 suggests that the nanoparticles are, on average, somewhat elongated or have a more extended shape. The standard deviation of 0.71 implies a greater range of aspect ratios, indicating potential variability in the shapes of the nanoparticles.
- Roundness value of 0.58 suggests that the nanoparticles, on average, have a shape that deviates from a perfect circle. The lower the roundness value, the more irregular or non-circular the shape of the particles tends to be.The standard deviation of 0.17 provides information about the variability in roundness across the nanoparticle population.
- Solidity value of 0.60 suggests that, on average, the nanoparticles have a shape that is somewhat irregular or concave. The lower the solidity value, the more irregular or indented the shape of the particles tends to be.

Acknowledgements

Authors are thankful to IIT Mumbai for providing the SEM TEM spectral data. Authors also thanks to Dr. V. D. Nanoty, Principal for encouragement and providing necessary facilities.

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