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Nanotechnology-Enabled synthesis of 1-tetra-O-acetyl-β -D-glucosyl -5-aryl-2,4- dithiobiurets

Kalpana M. Kalange, Ashish G. Sarap and P.T. Agrawal
Department of Chemistry,
Shri R. L. T. College of Science, Akola-444001(Maharashtra) India
Email: vrkhodkumbhe@gmail.com

Abstract: - Nanoparticle-based probes are increasingly being favored over molecular probes due to their numerous advantages, including their bright and adjustable optical properties, improved chemical and photochemical stability, and the ease with which multifunctionality can be introduced. This article focuses on the synthesis of nanoparticles derived from 1-tetra-O-acetyl-β-D-glucosyl-5- aryl-2,4-dithiobiurets, achieved through the fusion of Tetra-O-acetyl-β-D-glucosyl isothiocyanate with aryl thiocarbamides. These compounds are of interest due to their potential as biologically active substances and their versatility as intermediates for producing various derivatives. The characterization of the newly synthesized compounds was conducted using standard chemical transformations, as well as analytical techniques such as UV spectroscopy, scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

Key Words: - 1-tetra-O-acetyl-β -D-glucosyl -5-aryl-2,4-dithiobiurets, Tetra-O-acetyl-B-D-glucosyl isothiocyanate, Aryl thiocarbamides

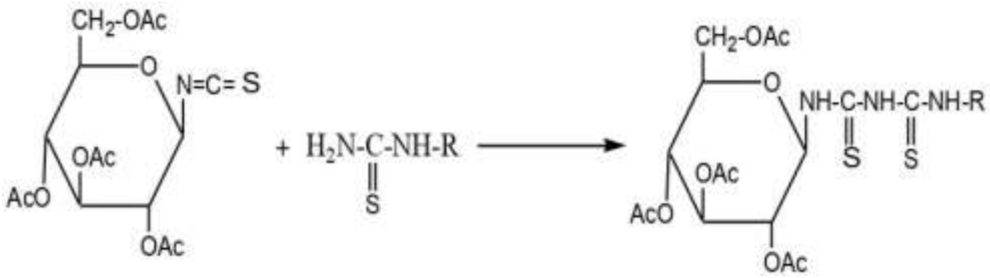
Introduction: Carbohydrates represent a critical chemical category, serving as the fundamental chemistry for numerous pharmaceuticals and drug precursors. Notably, various drugs, such asamino glycoside antibiotics, incorporate carbohydrate motifs. This chemical group is notable for its vast diversity, as organic compounds from this class are found in all known plants, animals, and microbial organisms. Carbohydrates play essential roles in supplying energy and structural support in plants, while in mammalian tissues, they fulfill a spectrum of specialized functions[1-5]**.** These functions include tasks like cell and organ differentiation, as well as providing immune protection for newborns.

One particularly intriguing aspect of carbohydrate chemistry revolves around the synthesis of nitrogen-linked glucosyl compounds. This focus arises from the myriad applications of such compounds in medicinal chemistry and other diverse fields. Sugar isocyanates emerge as adaptable synthetic intermediates in carbohydrate chemistry, garnering significant attention in both synthetic and medicinal chemistry realms.

Nanoparticles, typically ranging from 1 to 100 nanometers in size, possess distinctive characteristics that distinguish them from their bulk counterparts. This minute scale imbues nanoparticles with unique properties, sparking considerable scientific interest. Their versatility extends across various domains, including electronics, medicine, environmental science, and energy[6-10]**.**

Their extensive usability stems from their outstanding characteristics, particularly their high ratio of surface area to volume. This attribute renders nanoparticles invaluable in a variety of fields. Especially within the realm of medical science, nanoparticles have become versatile and powerful resources. Their ability to improve disease diagnosis and treatment highlights their importance in enhancing patient outcomes.

Reaction schemes:



(I)	(II)	(III)
Tetra-O-acetyl-β-D-isothiocyanate	Aryl thiocarbamides	1-tetra-O-acetyl-β-D-Glucosyl glucosyl -5-aryl-2,4-dithiobiurets

Thiocarbamide[11] was synthesized using both conventional and Microwave methods. The reaction involved the reaction of Tetra-O-acetyl-β-D-glucosyl isothiocyanate with aryl amines in a benzene medium, followed by titration of the reaction mixture with petroleum ether. The confirmation of the product was based on its melting point and other relevant studies. Subsequently, nanoparticles were prepared using an ultrasound sonicator. The particle size and morphology were analyzed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM) at the SAIF facility in IIT Mumbai.

EXPERIMENTAL:

Preparation of 1-aryl thiocarbamides[11] :

These have been prepared by the interaction of ammonium thiocyanate and appropriate aryl amine hydrochlorides. Details of typical preparation (where, aryl=phenyl) are as follows :

Aniline (52 ml) and concentrated hydrochloric acid (52 ml) were heated in a 500 ml round-bottom flask until its hydrochloride was formed. The hydrochloride was dissolved in water, and to this solution, a solution of ammonium thiocyanate (40 g in 150 ml water) was added. After thorough mixing, the mixture was boiled over a gentle Bunsen flame until the boiling solution became turbid due to the separation of phenyl thiocarbamide. This hot solution was poured with stirring into 250 ml of cold water. The result was the formation of 1-phenyl thiocarbamide (40 g) as a white solid. The compound was further crystallized from boiling water, and its melting point was determined to be 154°C.

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

In a suspension of tetra-O-acetyl-α-D-glucosyl bromide (21 g) in sodium-dried xylene (80 ml), lead thiocyanate (15 g) was added. The reaction mixture was gently refluxed for 3 hours with frequent shaking. After cooling, liberated lead bromide was removed by filtration. The xylene filtrate was then treated with petroleum ether (60-80°C) under stirring, yielding a pale yellow solid (12 g). This solid was anticipated to be tetra-O-acetyl-β-D-glucosyl isothiocyanate. To purify the compound, it was dissolved in a minimal quantity of chloroform and reprecipitated with petroleum ether indicated its molecular formula as $C_{15}H_{19}O_9NS$

Synthesis of 1-Tetra-O-acetyl- β -D-glucosyl-5-phenyl-2,4-dithiobiuret

In a toluene solution, the interaction between tetra-O-acetyl-β-D-glucosyl isothiocyanate (0.005 M, 1.9 g in 20 ml) and phenyl thiocarbamide (0.005 M, 0.76 g in 10 ml) was initiated. The reaction mixture was refluxed over a boiling water bath for 3 hours. Subsequently, the solvent was distilled off, and the resulting sticky mass, isolated as a residue, was subjected to multiple triturations with petroleum ether, yielding a white solid. The final product was crystallized from ethanol-water, exhibiting a melting point of 94°C indicated its molecular formula as $C_{22}H_{26}O_9N_3S_2$.

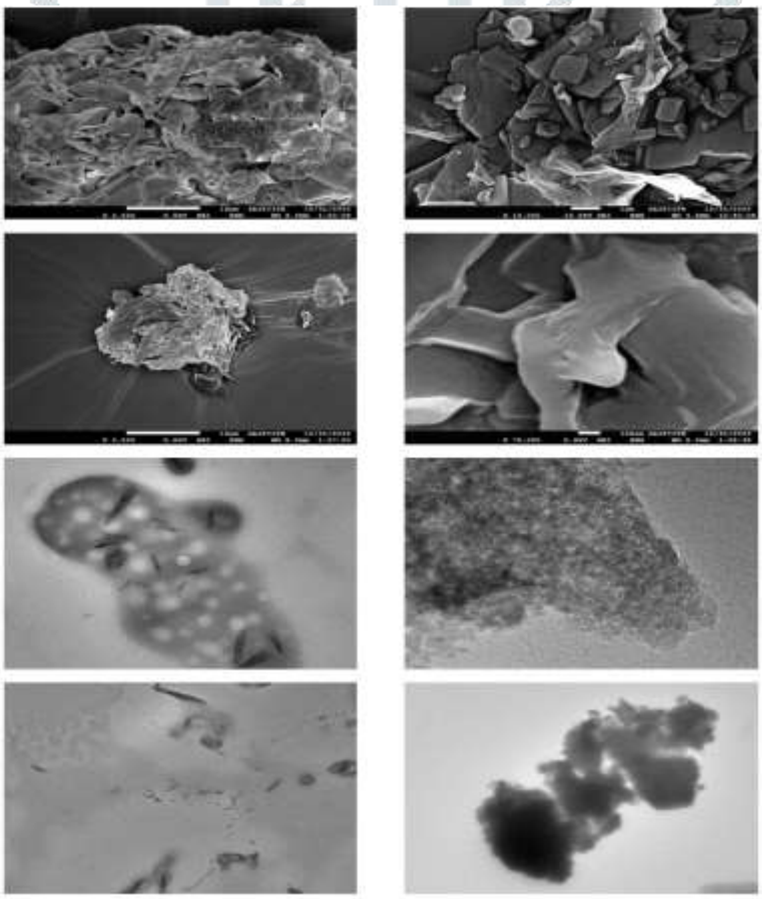
Preparation of nanoparticles of 1-Tetra-O-acetyl- β -D-glucosyl-5-phenyl-2,4-dithiobiuret

Take about 1 gm of 1-Tetra-O-acetyl- β -D-glucosyl-5-phenyl-2,4-dithiobiuret 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

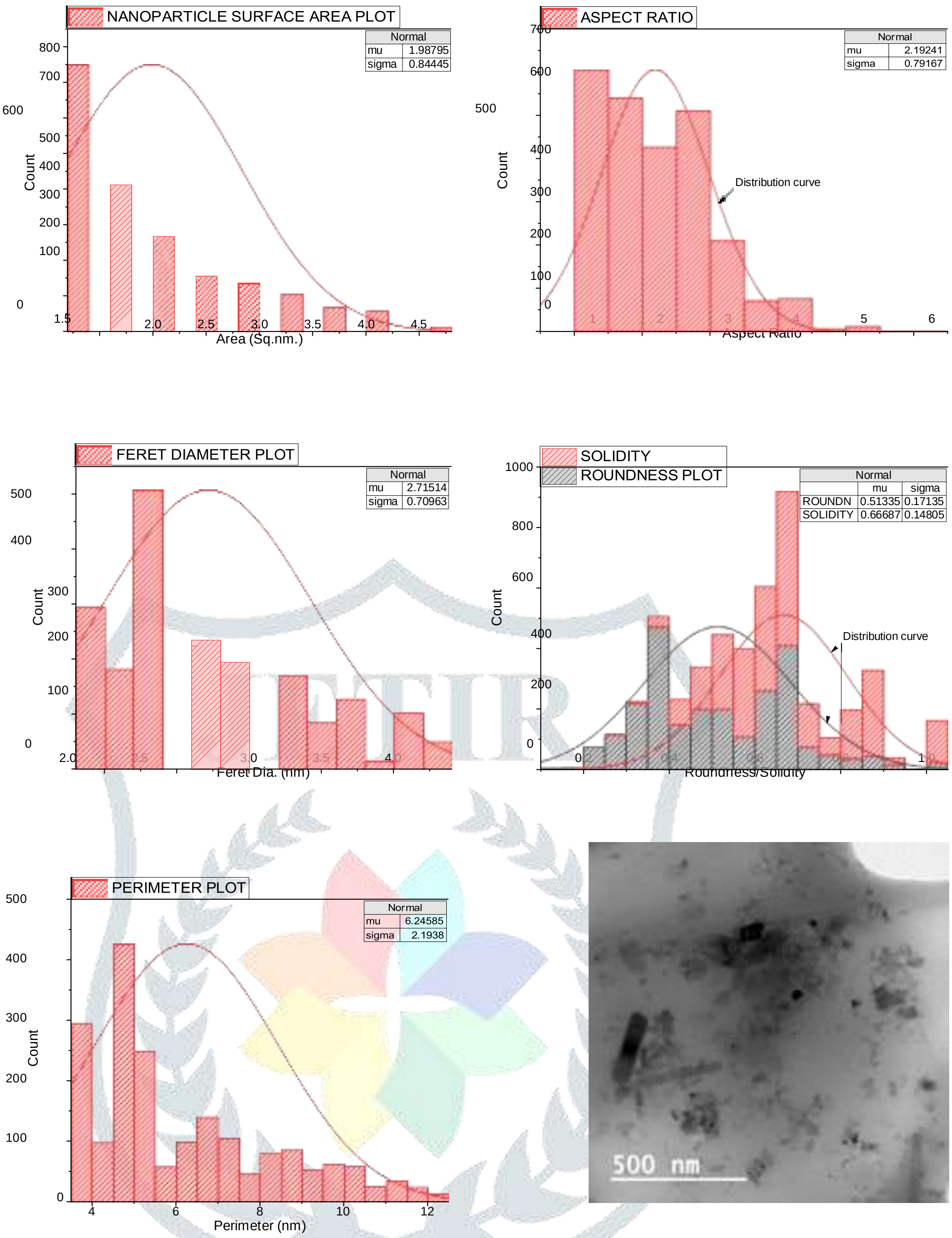


FIG. NO. 2 HISTOGRAM OF SAMPLE TEM IMAGE OF

1-Tetra-O-acetyl- β -D-glucosyl-5-phenyl-2,4-dithiobiuret

Table 1: Nanoparticle Charecteristics.

The medians, mean values and standard deviations of various nanoparticle parameters determined by ImageJ software are listed below (Ref. Fig. No. 2)

Sr.No.	Parameters	Median	Mean Value	Std. Dev.
1	Surface Area	2.10 sq.nm	1.99 sq.nm	0.84
2	Feret's Diameter(Long.Cal.)	2.90 nm	2.72 nm	0.71
3	Feret Angle	108.44 deg	96.75 deg	46.58
4	Perimeter	6.80 nm	6.25 nm	2.2
5	Aspect Ratio	2.02	2.19	0.78
6	Roundness	0.50	0.51	0.17
7	Solidity	0.67	0.67	0.15

CONCLUSIONS:

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 2.19 suggests that the nanoparticles are, on average, somewhat elongated or have a more extended shape. Roundness value of 0.51 suggests that the nanoparticles, on average, have a shape that deviates from a perfect circle. Solidity value of 0.67 suggests that, on average, the nanoparticles have a shape that is somewhat irregular or concave.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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