

TRIAZINE Dendrimer: Synthesis and Characterization

**Hindurao B.Kale^{1a*}, Sampat D. Nawale², Manoj Shirbhate³,
Mrunal K.Shirsat⁴, Santosh R.Tarke⁵**

*1a. College of Pharmacy, Paniv-413113, Solapur
Research Scholar, Sunrise University, Alwar, Rajasthan 301028*

2. Principal, Delight Institute of Pharmacy, Pune: - 412216

3. Principal, Amrutvahini Institute of Pharmacy, Sangamner: - 422608

4. Principal, SBSPM B-Pharmacy College Ambajogai

5. Professor, SBSPM B-Pharmacy College Ambajogai

Abstract

The word “dendrimer” begun from two Greek words; i) Dendron: meaning tree, and ii) Meros: meaning portion or fragment. A dendrimer could be a Nanoparticle. It may be a branched macromolecule having a tall degree of atomic consistency, limit atomic weight, dispersion, particular estimate and shape characteristics, and a highly-functionalized, terminal surface. A arrangement of triazine based dendrimer vary in spacer length of center was synthesized by disparate strategy utilizing triazine trichloride and diethanolamine as branching units. Synthesized dendrimer were characterized by ghostly strategies such as FT-IR, ¹H-NMR, ¹³C-NMR, ESI-Mass spectrometry and Basic examination. A number of dendrimer were synthesized utilizing diverse center in spacer length by dissimilar strategy. Characterization of these Dendrimer was palatably carried out by ghostly strategy. It was watched that chlorine terminated dendrimer were somewhat more thermally steady than hydroxyl ended dendrimer through TGA. On the total, warm solidness was expanded with increment in generation of dendrimer. Warm solidness was autonomous of center moiety or spacer length of dendrimer of same era. With increment within the dendrimer era Glass move temperature was too expanded and was too comparable to TRIAZINE dendrimer.

Key words: - Triazine, Dendrimer, Nanoparticle, Fragment

Correspondence Author:- Hindurao B.Kale

Research Scholar, Sunrise University, Alwar, Rajasthan 301028

INTRODUCTION**Triazine:**

Triazines are six membered fragrant heterocycles comprised of three carbon and three nitrogen atoms. A triazine is one of three natural chemicals, isomeric with each other, whose atomic equation is C₃H₃N₃ and whose observational equation is CHN.

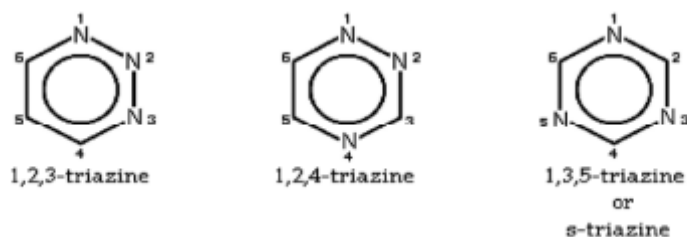
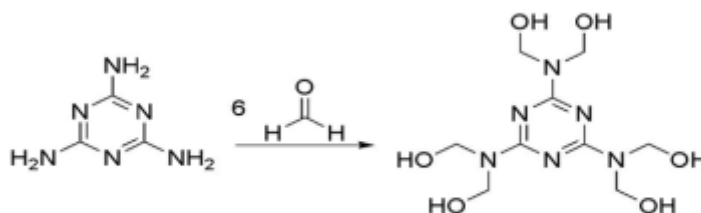


Figure 2.1 : The three isomers of triazine, with ring numbering.

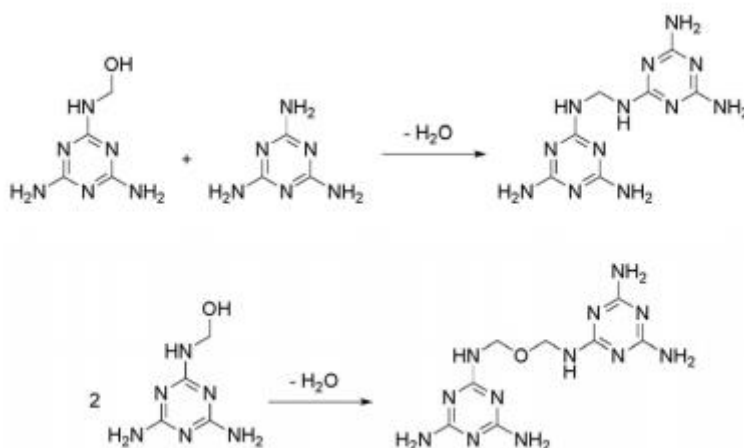
Structure: The triazine structure could be a heterocyclic ring, closely resembling to the six-membered benzene ring but with three carbons supplanted by nitrogens. The three isomers of triazine are

recognized from each other by the positions of their nitrogen molecules, and are alluded to as 1,2,3-triazine, 1,2,4-triazine, and 1,3,5-triazine. Other fragrant nitrogen heterocycles are pyridines with 1 ring nitrogen atom, diazines with 2 nitrogen particles within the ring and tetrazines with 4 ring nitrogen molecules. Triazines are weaker bases than pyridine.

Scheme 1. Mechanical union of melamine Melamine is most frequently responded with formaldehyde for the generation of tars. This hone started in 1935 in Germany.¹²² The polymers shaped from elamineformaldehyde tars have amazing chemical and physical properties. The gums have various applications counting covers, pastes and cements, molding compounds, coatings, and paper and textiles.¹²² The response of melamine with formaldehyde is appeared underneath in Conspire 2. Anyplace from one to six of the hydrogen particles on the amine bunches of melamine may be supplanted by methylol bunches. Methylolmelamines are unsteady due to the plausibility of advance condensation or resinification, as appeared in Plot 3 with a monomethylolmelamine, but comparable responses happen with the other di or tri-methylolmelamines. The most seasoned application of melamine gums is cements for wood such as plywood or molecule board. Making a frothed tar produces difficult, however adaptable, lightweight materials which are utilized for sound cover, fire security, and cleaning items. Other applications incorporate impregnating tars to treat papers for embellishing purposes, paper assistants to improve damp pliable quality, calfskin tanning operators, strengtheners for building materials, concrete added substances, ion-exchange tars, and wood additives.



Scheme 1. Initial reaction of formaldehyde and melamine

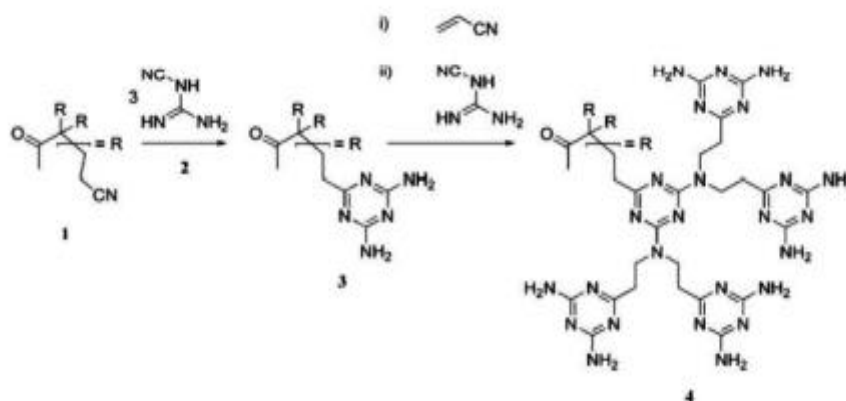


Scheme 2. Condensation and resinification of methylmelamine

Engineered Courses to Triazine Dendrimers Much of the early work within the range centered on the optimization of courses for the blend of these species utilizing either cycloaddition responses or triazine substitution reactions.

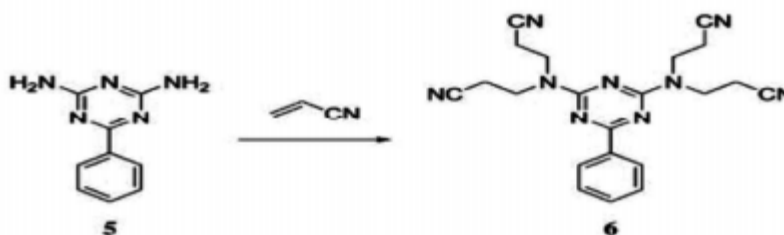
Cycloaddition Method:

To the most excellent of our knowledge, the primary case of the blend of a triazine-based dendrimer employing a dissimilar approach was point by point in two licenses that were submitted in 1994,¹³² one of which was checked on in a isolated account in 1995 (Conspire: 6).¹³³ In both cases, an iterative blend was created in which the triazine units were arranged by the cycloaddition of terminal nitrile-functional bunches, 1, with nitrile substituted guanidine, 2, to manage an amino-terminated dendrimer, 3. Cycle produces higher eras such as 4. The union laid out within the obvious by Meijer et al.⁹⁷ is of specific intrigued since it subtle elements a method that can be utilized to plan commercially pertinent amounts of dendrimer item. More recent reports have illustrated the utility of the cycloaddition strategy to manage triazine-based dendritic and hyperbranched materials,¹³⁴ a few of which have found application within the development of permeable, hydrogen-bonded systems. In expansion, cyclotrimerization courses have been utilized to create materials important for coordinates optics.



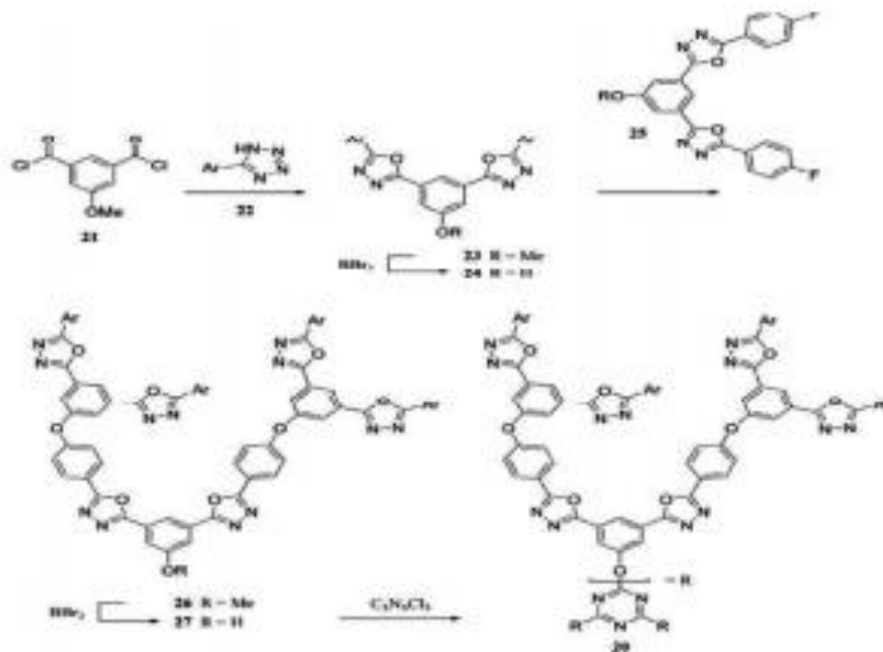
Scheme 3. Initial efforts to prepare triazine based employed cycloaddition methods

In all the cycloaddition strategies, the chemistry of the iterative handle is the same: cyanoethylation of a pendant amine shapes a dendron with twice the number of nitrile bunches. Ensuing elaboration of the dendron by cycloaddition between the fringe nitriles and nitrile-substituted guanidine subsidiaries increments the dendrimer generation. A strategy coming about in compound 6, with highlights strikingly comparative to that of Vögtle's, was depicted in a obvious by Niederhauser¹³⁸ in 1951,²⁷ a long time some time recently Vögtle's work. Niederhauser knew of the capacity to diminish nitrile bunches to amines: he portrayed such a strategy in a obvious recorded in 1945.¹³⁹ Either through the decrease of the nitriles to amines or advance cycloaddition responses with the nitriles, Niederhauser might have laid claim to the primary dendrimer. Here was a close miss to the starting of dendrimer chemistry. In spite of the fact that a more thorough (and entirely illogical) look of the writing may give more close misses, our intrigued in triazines requires its incorporation. The elaboration of Niederhauser tetracyanoethylbenzoguanamine to manage a fundamentally flawed generation-four dendrimer, in any case, took over 40 a long time to achieve.

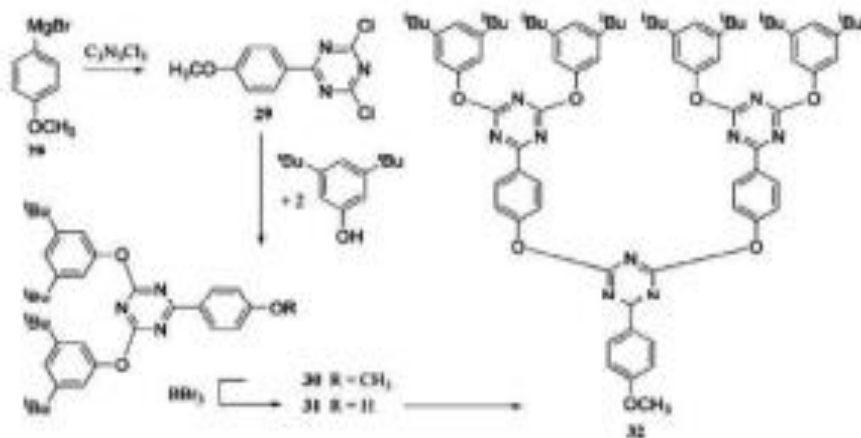


Scheme 4. Niederhauser's route tetracyanoethyl benzoguanamine

Reaction Scheme



Scheme 5. Triazine as the core of a dendrimer



Scheme 6. Different nucleophiles are used to synthesize a dendrimer

EXPERIMENTAL

Materials

Triazine trichloride, 1,3-propanediamine, 1,4-butanediamine, 1,6-hexanediamine, dichloromethane (DCM), acetone and methanol were acquired from Sigma- Aldrich Ltd. Sodium hydroxide was obtained from Merck Ltd. All other reagents and solvents required for the amalgamation and examination were utilized as gotten.

Analytical Techniques

Thermo gravimetric examination was performed between temperature ranges between 0- 600°C on Mettler Toledo TGA (Switzerland) instrument in Nitrogen environment. Differential Checking Calorimetry was too performed on Mettler Toledo (Switzerland) instrument between -50°C to 600°C in Nitrogen climate. Thickness estimations on Triazine-based dendrimers detailed in this proposition were performed utilizing an Ubbelohde device of estimate 1 drenched in a water shower thermo-stated at 30°C. FTIR was carried out within the run of 250–4000 cm^{-1} utilizing Perkin Elmer-Spectrum RX-FTIR spectrometer instrument within the course of KBr pellet strategy for strong tests or nujol reflect strategy for fluid tests. $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectra were recorded in Bruker Avance II 400 (Germany) utilizing TMS as inner standard utilizing either D_2O or DMSO-d_6 as solvents. Mass spectra were recorded on Waters Micromass Q-TOF Small scale (USA) instrument prepared with electrospray ionization. Essential examination was recorded on Perkin Elmer 2400 CHN essential analyzer.

Methods

General procedure for synthesis of G0.5 dendrimer (AG0.5, BG0.5, CG0.5)

Cyanuric Chloride (0.02mmol) was broken up in dichloromethane and kept in an ice shower. A arrangement of a diamine (0.01mmol) containing sodium hydroxide (0.02 mmol) in water was included drop astute within the arrangement of cyanuric chloride at 0-5 °C with mixing. The arrangement was blended at 0-5 °C for 2 hrs. At that point the arrangement was sifted, washed with methanol and acetone and dried beneath vacuum: A white coloured strong was shaped.

Characterization Data of AG0.5 dendrimer

Yield: 79%

$^1\text{H-NMR}$ (400MHz, DMSO-d_6) δ ppm: 1.8301-1.8649(m, 2H, N-CH₂-CH₂-CH₂-N), 3.4304-3.4660(m, 4H, N-CH₂-CH₂-CH₂-N) [Fig. 2.25. $^1\text{H-NMR}$ spectrum of AG0.5 dendrimer].

$^{13}\text{C-NMR}$ (75MHz, DMSO-d_6) δ ppm: 28.70 (N-CH₂-CH₂-CH₂-N), 38.18 (N-CH₂-CH₂-CH₂-N), 166.01 (triazine C-N), 171.66 (triazine C-Cl) [Fig. 2.26. $^{13}\text{C-NMR}$ spectrum of AG0.5 dendrimer].

ESI-Mass: Calculated: 371; obtained 372 (M+1) [Fig. 2.27. ESI-Mass Spectrum of AG0.5 dendrimer]

FT-IR (KBr, cm^{-1}) : 3291 (secondary N-H stretching), 2872, 2780 (Aliphatic C-H stretching), 1722, 1625 (aromatic C=N), 841, 792 (C-Cl stretching) [Fig. 2.24. FT-IR spectrum of AG0.5 dendrimer].

Characterization data for BG0.5 Dendrimer

Yield: 81 %

$^1\text{H-NMR}$ (400MHz, DMSO-d_6) δ ppm: 1.5483-1.6603 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 3.2929-

3.3010 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-N) [Fig. 2.29. ¹H-NMR spectrum of BG0.5 Dendrimer].

¹³C-NMR (75MHz, DMSO-*d*₆) δ ppm: 25.27, 25.51(N-CH₂-CH₂-CH₂-CH₂-N), 40.22, 40.88 (N-CH₂-CH₂-CH₂-CH₂-N), 165.10(triazine C-N), 169.46 (Triazine C-Cl) [Fig.2.30. ¹³C-NMR spectrum of BG0.5 Dendrimer].

ESI-Mass: Calculated: 383; obtained 384 (M+1) [Fig.2.31. ESI-Mass spectrum of BG0.5 Dendrimer]

Characterization Data of CG0.5 dendrimer

Yield, 85%; FT-IR (KBr, cm⁻¹) : 3281(N-H), 2884, 2779(aliphatic C-H), 1722,

¹³C-NMR (75MHz, DMSO-*d*₆) δ ppm: 25.20, 25.29 (N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N), 30.52 (N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N), 40.52, 41.57(N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N), 168.34(triazine C-N), 169.40(Triazine C-Cl)[Fig.2.34. ¹³C-NMR spectrum of CG0.5 Dendrimer].

1624(C=N of triazine), 844, 796(C-Cl); [Fig.2.32. FT-IR spectrum of CG0.5 Dendrimer].

¹H-NMR (400MHz, DMSO-*d*₆) δ ppm: 1.2898- 1.3558 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N), 1.4941-1.5383 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N) 3.2445- 3.2881(m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N); [Fig. 2.33. ¹H-NMR spectrum of CG0.5 Dendrimer].

ESI-Mass: Calculated: 411; obtained 412 (M+1) [Fig.2.35. ESI-Mass spectrum of CG0.5 Dendrimer]

General procedure for synthesis of G1 Dendrimer (AG1, BG1, CG1)

G0.5 dendrimer (AG0.5, BG0.5, CG0.5) (0.01mmol) was dissolved in an excess of diethanolamine (0.04mmol) which was used as both solvent and reactant. The resulting mixture was refluxed for 2 hrs. After cooling, it was dispersed and washed by acetone repeatedly to give generation 1 dendrimer which was light brown coloured with honey like consistency.

Characterization data of AG1 dendrimer

Yield: 75%.

¹³C-NMR (75MHz, D₂O) δ ppm: 28.72 (N-CH₂-CH₂-CH₂-N), 38.22 (N-CH₂-CH₂-CH₂-N), 50.66 (N-CH₂-CH₂-OH), 60.18 (N-CH₂-CH₂-OH), 166.61(triazine C-N), 171.16 (triazine C-O).

FTIR (KBr, cm⁻¹) : 3373(O-H stretching), 2949, 2881(aliphatic C-H stretching), 1664(Aromatic C=N stretching), 1056 (C-O stretching).

¹H-NMR (400MHz, D₂O) δ ppm: 1.9091-1.9403(m, 2H, N-CH₂-CH₂-CH₂-N), 3.3211-3.3460 (m, 4H, N-CH₂-CH₂-CH₂-N), 3.5980-3.6485 (m, 16H, N-CH₂-CH₂-OH), 3.9480-3.9856 (m, 16H, N-CH₂-CH₂-OH).

ESI-Mass: Calculated Molecular weight 644; Obtained: 645 (M+1), 646 (M+2)

Characterization data of BG1 dendrimer

Yield: 78%.

FT-IR (Nujol, cm⁻¹) : 3363(O-H stretching), 2967(aliphatic C-H), 1667(aromatic C=N), 1064(C-O stretching)

¹H-NMR (400 MHz, D₂O) δ ppm: 1.5433-1.5770(m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 3.2929-3.3010(m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 3.5600-3.6293(m, 16H, N-CH₂-CH₂-OH),

3.8873-3.9418(m, 16H, N-CH₂-CH₂-OH)

¹³C-NMR (75MHz, D₂O) δ ppm: 25.22, 25.55 (N-CH₂-CH₂-CH₂-CH₂-N), 40.25, 40.80 (N-CH₂-CH₂-CH₂-CH₂-N), 57.04 (N-CH₂-CH₂-OH), 61.50 (N-CH₂-CH₂-OH), 165.11 (triazine C-N), 169.45 (triazine C-O).

ESI-Mass: Calculated Molecular Weight 658; Obtained: 657(M+1), 658(M+2)

Characterization data of CG1 dendrimer

Yield: 75%;

¹H-NMR (400MHz, D₂O) δ ppm: 1.3138-1.3358 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N), 1.4945- 1.5145 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N), 3.4191- 3.4381 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N) 3.5785-3.6151 (m, 16H, N-CH₂-CH₂-OH), 3.7801- 3.8145(m, 16H, N-CH₂-CH₂-OH); [Fig. 2.37. ¹H-NMR Spectrum for CG1 dendrimer]

FTIR (KBr, cm⁻¹) : 3364 (O-H), 2941 (aliphatic C-H), 1668 (C=N of triazine), 1063 (C-O); [Fig. 2.36. FT-IR Spectrum for CG1 dendrimer].

¹³C-NMR(75MHz,D₂O)δppm:25.20,25.29(N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 30.52 (N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N),40.52,41.57(N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N), 58.28 (N-CH₂-CH₂-OH), 61.04 (N-CH₂-CH₂-OH), 168.04 (triazine C-N), 169.90 (triazine C-O)[Fig. 2.38. ¹³C-NMR Spectrum for CG1 dendrimer]

ESI-Mass: Calculated Molecular Weight 686; Obtained: 687(M+1) [Fig. 2.39. ESI-Mass spectrum for CG1 dendrimer]

Characterization of AG1.5 dendrimer

Yield: 84%

¹³C-NMR (75MHz, DMSO-*d*₆) δ ppm: 28.70 (N-CH₂-CH₂-CH₂-N), 38.10 (N-CH₂-CH₂-CH₂-N), 51.01 (N-CH₂-CH₂-O-tria), 60.58 (N-CH₂-CH₂-O-tri), 163.01 (triazine C-N), 168.66 (Triazine C-O), 170.18 (Triazine C-Cl).

FT-IR (KBr, cm⁻¹) : 3217 (Secondary N-H stretching), 2833, 2780 (Aliphatic C-H stretching), 1777, 1752, 1721 (aromatic C=N of triazine), 1053 (C-O stretching), 775 (C-Cl stretching).

¹H-NMR (400MHz, DMSO-*d*₆) δ ppm: 1.9201-1.9549 (m, 2H, N-CH₂-CH₂-CH₂-N), 3.3425-3.3759 (m, 4H, N-CH₂-CH₂-CH₂-N), 4.0175-4.0759 (m, 16H, N-CH₂-CH₂-O-Tri), 4.1641-4.2015 (m, 16H, N-CH₂-CH₂-O-Tri).

ESI-Mass: Calculated Molecular Weight: 1826; Obtained: 1827 (M+1).

Characterization data for BG1.5 dendrimer

Yield: 77%.

¹³C-NMR (75MHz, DMSO-*d*₆) δ ppm:25.29 (N-CH₂-CH₂-CH₂-CH₂-N), 40.80 (N-CH₂-CH₂-CH₂-CH₂-N), 57.10 (N-CH₂-CH₂-O-tri), 61.59 (N-CH₂-CH₂-O-tri), 163.37.

FT-IR (KBr, cm⁻¹) : 3445 (Secondary N-H stretching), 2781 (Aliphatic C-H stretching), 1777, 1753, 1722 (Aromatic C=N stretching), 1053 (C-O stretching), 772 (C-Cl stretching)

¹H-NMR (400MHz, DMSO-*d*₆) δ ppm:1.5433-1.5770(m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 3.2929-3.3010(m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 4.0529-4.1151(m, 16H, N-CH₂-CH₂-O-tri), 4.1925-4.2311(m, 16H, N-CH₂-CH₂-O-tri) [Fig. 2.53. ¹H-NMR Spectrum of CG1.5 dendrimer] (Triazine C-N), 167.41 (Triazine C-N-(CH₂-CH₂-O)₂), 170.43 (Triazine C-O), 172.46

(Triazine C-Cl).

ESI-Mass: Calculated Molecular Weight: 1840; Obtained: 1841 (M+1).

Characterization data for CG1.5 dendrimer

Yield 88%.

^{13}C -NMR(75MHz,D₂O) δ ppm:25.20,25.29(N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 30.52 (N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 40.52, 41.57(N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N), 58.20 (N-CH₂-CH₂-O-tri), 61.04 (N-CH₂-CH₂-O-tri), 163.01(Triazine C-N), 166.66(Triazine C-N-(CH₂-CH₂-O-)₂), 170.18(Triazine C-O), 172.28 (Triazine C-Cl)

FT-IR (KBr, cm⁻¹) : 3214 (N-H), 2834, 2832, 2780 (aliphatic C-H), 1779, 1753, 1717(C=N of triazine), 1061(C-O), 786 (C-Cl) [Fig. 2.40. FTIR Spectrum of CG1.5 dendrimer]

^1H -NMR (400MHz, D₂O) δ ppm: 1.3138-1.3358 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 1.4945-1.5145(m,4H,N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 3.4191-3.4381

(m, 4H, CH₂-NH); 3.8585-3.9350 (m, 16H, N-CH₂-CH₂-O-Tri), 4.0301-4.0832(m, 16H, N-CH₂-CH₂-O-Tri)[Fig. 2.41. FTIR Spectrum of CG1.5 dendrimer]

[Fig. 2.42. ^{13}C -NMR Spectrum of CG1.5dendrimer]

ESI-Mass: Calculated Molecular Weight: 1870; Obtained: 1871 (M+1). [Fig. 2.43. ESI-Mass Spectrum of CG1.5dendrimer]

General procedure for synthesis of G2 Dendrimer (AG2, BG2, CG2)

Generation 1.5 dendrimer (AG1.5, BG1.5, CG1.5) (0.01mmol) was dissolved in an excess of diethanolamine (0.16 mmol) which was used as both solvent and reactant. The resulting mixture was refluxed for 2 hrs. After cooling, it was dispersed and washed by acetone repeatedly to give generation 2 dendrimer which was light brown coloured with honey like consistency.

Characterization data for AG2 dendrimer

Yield: 77%.

^{13}C -NMR (75MHz, D₂O) δ ppm: 28.50 (N-CH₂-CH₂-CH₂-N), 38.08 (N-CH₂-CH₂-CH₂-N), 60.81(N-CH₂-CH₂-O-tri), 61.28 (N-CH₂-CH₂-OH),63.88(N-CH₂-CH₂-Otri),

FT-IR (Nujol, cm⁻¹) : 3356(O-H stretching), 2942, 2881, 2840 (aliphatic C-H stretching), 1676, 1619(Aromatic C=N stretching), 1065(C-O stretching)

^1H -NMR (400MHz, D₂O) δ ppm: 1.9331-1.9694 (m, 2H, N-CH₂-CH₂-CH₂-N), 3.3225-3.3519 (m,4H,N-CH₂-CH₂-CH₂-N), 3.6813-3.7303 (m, 64H,N-CH₂-CH₂-OH), 3.8631-3.9182 (m, 64H, N-CH₂-CH₂-OH), 4.0175-4.0759 (m, 16H, N-CH₂-CH₂-O-tri), 4.1641-4.2015 (m, 16H, N-CH₂-CH₂-O-tri).65.70 (N-CH₂-CH₂-OH), 163.01(Triazine C-N), 168.66(Triazine C-N-(CH₂-CH₂-O-)₂), 170.78(Triazine C-O), 172.28 (Triazine C-N(CH₂-CH₂-OH)₂).

ESI-Mass: Calculated Molecular weight: 2926; Obtained: 2927 (M+1)

Characterization data for BG2 dendrimer

Yield: 78%.

FT-IR (Nujol, cm⁻¹): 3356(O-H stretching), 2942, 2881, 2831 (aliphatic C-H stretching), 1681, 1619(Aromatic C=N stretching), 1071(C-O stretching)

^1H -NMR(400MHz,D₂O) δ ppm:1.5729-1.5931(m,4H,N-CH₂-CH₂-CH₂-N),3.1957-3.2220(m,

4H, N-CH₂-CH₂-CH₂-CH₂-N), 3.5817-3.6720 (m, 64H, N-CH₂-CH₂-OH), 3.7276-3.8027(m, 64H, N-CH₂-CH₂-OH), 3.9002-3.9890(m, 16H, N-CH₂-CH₂-O-tri), 4.0522-4.1272(m, 16H, N-CH₂-CH₂-O-tri)

¹³C-NMR (75 MHz, D₂O,) δ ppm: 25.12 (N-CH₂-CH₂-CH₂-CH₂-N), 40.08 (N-CH₂-CH₂-CH₂-N), 55.54 (N-CH₂-CH₂-O-tri), 57.60 (N-CH₂-CH₂-OH), 59.87 (N-CH₂-CH₂-O-tri), 61.51 (N-CH₂-CH₂-OH), 163.88 (Triazine C-N), 164.54(Triazine C-N-(CH₂-CH₂-O-)2), 170.99 (Triazine C-O), 172.41(Triazine C-N(CH₂-CH₂-OH)2)

ESI-Mass: Calculated Molecular weight: 2941; obtained: 2942 (M+1)

Characterization data for CG2 dendrimer

Yield 75%

FT-IR (Nujol, cm⁻¹) : 3389(O-H stretching), 2939, 2950(aliphatic C-H stretching), 1671, 1619(Aromatic C=N stretching), 1068 (C-O stretching) [Fig. 2.44. FT-IR spectrum of CG2 dendrimer]

¹H-NMR (400MHz, D₂O) δ ppm: 1.3158-1.3355 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 1.4984-1.5138 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 3.4191-3.4381(m, 4H, CH₂-NH), 3.6743-3.7837(m, 64H, N-CH₂-CH₂-OH), 3.8126-3.8970(m, 64H, N-CH₂-CH₂-OH), 3.9803-4.0433 (m, 16H, N-CH₂-CH₂-O-tri), 4.0917-4.1672 (m, 16H, N-CH₂-CH₂-O-tri) [Fig. 2.45. ¹H-NMR spectrum of CG2 dendrimer].

¹³C-NMR (75MHz, D₂O) δ ppm: 25.70 (N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 30.31 (N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 40.81 (N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 60.81 (N-CH₂-CH₂-O-tri), 61.28 (N-CH₂-CH₂-OH), 63.78 (N-CH₂-CH₂-O-tri), 65.80 (N-CH₂-CH₂-OH), 163.66(Triazine C-N), 168.66(Triazine C-N-(CH₂-CH₂-O-)2), 170.18(Triazine C-O), 172.21(Triazine C-N(CH₂-CH₂-OH)2) [Fig. 2.46. ¹³C-NMR spectrum of CG2 dendrimer]

ESI-Mass: Calculated Molecular weight: 2970; obtained: 2971 (M+1) [Fig. 2.47. ESI-Mass spectrum of CG2 dendrimer]

General procedure for synthesis of G2.5 Dendrimer (AG2.5, BG2.5, CG2.5)

Cyanuric Chloride (0.32mmol) was dissolved in dichloromethane and kept in an ice bath. A solution of G2 dendrimer (AG2, BG2, CG2) (0.01mmol) containing sodium hydroxide (0.32 mmol) in water was added dropwise in the solution of cyanuric chloride at 0-5 °C with stirring. The solution was stirred at 0-5 °C for 2 hrs and refluxed for 6 hrs. Then the solution was filtered, washed with methanol and acetone and dried under vacuum: A white coloured solid was formed.

Characterization data for AG2.5 dendrimer

Yield: 82%

FT-IR (KBr, cm⁻¹) : 3215(Secondary N-H stretching), 2881, 2833(aliphatic C-H stretching), 1778, 1752, 1722(Aromatic C=N stretching), 1054 (C-O stretching), 776 (C-Cl stretching).

¹H-NMR (400MHz, DMSO-*d*₆) δ ppm: 3.3382-3.3728(t, 4H, N-CH₂-CH₂-CH₂N), 3.9662-4.0226(m, 80H, N-CH₂-CH₂-O-Tri), 4.0777-4.1440(m, 80H, N-CH₂-CH₂-O-Tri).

¹³C-NMR (75MHz, DMSO-*d*₆) δ ppm: 28.8 (N-CH₂-CH₂-CH₂-N), 38.2(N-CH₂-CH₂-CH₂-N), 60.0(outer N-CH₂-CH₂-O-tri) , 61.2(inner N-CH₂-CH₂-O-tri), 63.3(outer N-CH₂-CH₂-O-tri), 65.5(inner N-CH₂-CH₂-O-Tri), 164.1(triazine C-N), 166.4(inner C-N-(CH₂-CH₂-O-)2), 171.7(outer C-N-(CH₂-CH₂-O-)2), 172.5(outer C-Cl), 174.0(inner triazine C-O).

ESI-Mass: Calculated Molecular Weight: 7660: Obtained: 7661 (M+1)

Characterization data for BG2.5 dendrimer

Yield: 71%.

FT-IR (KBr, cm^{-1}) : 3215 (Secondary N-H stretching), 2884, 2833(aliphatic C-H stretching), 1779, 1752, 1717(Aromatic C=N stretching), 1051 (C-O stretching), 762 (C-Cl stretching)

$^1\text{H-NMR}$ (400 MHz, DMSO-*d*₆) δ ppm: 1.5515-1.5913 (m, 4H, N-CH₂-CH₂-CH₂-N), 3.2573-3.3052(m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 4.0153-4.1380(m, 80H, N- CH₂-CH₂-O-Tri), 4.2168-4.2929 (m, 80H, N-CH₂-CH₂-O-Tri)

$^{13}\text{C-NMR}$ (75 MHz, DMSO-*d*₆) δ ppm: 25.11(N-CH₂-CH₂-CH₂-CH₂-N), 40.80 (N- CH₂-CH₂-CH₂-CH₂-N), 55.51 (outer N-CH₂-CH₂-O-tri), 57.61 (innerN-CH₂-CH₂-Otri), 59.88(outer N-CH₂-CH₂-Otri), 61.57 (inner N-CH₂-CH₂-O-), 162.54(Triazine C-N), 165.24(inner C-N-(CH₂-CH₂-O)-₂), 168.08(outer C-N-(CH₂-CH₂-O)-₂),170.00(outer C-Cl), 171.12 (inner triazine C-O)

ESI-Mass: Calculated Molecular Weight: 7673; Obtained: 7674 [M+1].

Characterization data for CG2.5 dendrimer

Yield: 70%;

$^{13}\text{C-NMR}$ (75MHz, DMSO-*d*₆) δ ppm: 25.20(N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 30.05 (N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N), 40.45 (N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N),60.06(N-CH₂-CH₂-O-tria),61.23(N-CH₂-CH₂-OH),63.30(N-CH₂-CH₂-O-tria),65.53 (N-CH₂-CH₂-OH), 164.11 (Triazine C-N), 166.41 (innerC-N-(CH₂-CH₂-O)-₂),168.81 (outer Triazine C-O), 171.74 (outer C-N-(CH₂-CH₂-O)-₂), 172.57(outer C-Cl),174.04 (inner triazine C-O)[Fig. 2. 50. $^{13}\text{C-NMR}$ spectrum of CG2.5 dendrimer]

ESI-Mass: Calculated Molecular Weight: 7703; Obtained: 7704 [M+1]. [Fig.2.51. ESI-Mass spectrum of CG2.5 dendrimer]

FT-IR (KBr, cm^{-1}) : 3280 (N-H), 2852, 2819 (aliphatic C-H), 1750, (C=N of triazine), 1045(C-O), 787 (C-Cl); [Fig. 2.48. FT-IR spectrum of CG2.5 dendrimer]

$^1\text{H-NMR}$ (400MHz, DMSO-*d*₆) δ ppm: 1.3118-1.3351 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N), 1.4984-1.5150 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-CH₂-N),3.4151-3.4389 (m, 4H, CH₂-NH), 4.0153- 4.1380 (m, 80H, N-CH₂-CH₂-O-tri), 4.2168-4.2929(m, 80H,N-CH₂-CH₂-O-tri)[Fig.2.49. $^1\text{H-NMR}$ spectrumofCG2.5dendrimer]

Thin Layer Chromatography (TLC):- Hexane: Methanol; 9:1.

General procedure for synthesis of Generation 3 dendrimer (AG3, BG3, CG3)

Generation 2.5 dendrimer (AG2.5, BG2.5, CG2.5) (0.01mmol) was dissolved in an excess of diethanolamine (0.64 mmol) which was used as both solvent and reactant. The resulting mixture was refluxed for 2 hrs. After cooling, it was dispersed and washed by acetone repeatedly to give generation 3 dendrimer which was light brown colored with honey like consistency.

Characterization data for AG3 dendrimer [55]

Yield: 71 %

FT-IR (Nujol, cm^{-1}) : 3371(O-H Stretching), 2948, 2839(Aliphatic C-H stretching), 1733(Aromatic C=N stretching), 1063(C-O stretching) [Fig.2.52. FT-IR Spectrum of AG3 dendrimer]

$^1\text{H-NMR}$ (400MHz,D₂O) δ ppm:3.3211-3.3440(t,4H,N-CH₂-CH₂-CH₂-N),3.6438-3.7243 (m, 264H, N-CH₂-CH₂-OH), 3.7929-3.8586(m, 264H, N-CH₂-CH₂-OH),3.9107-3.9520 (m, 80H, N-CH₂-CH₂-O-Tri),4.0042-4.0315(m,80H,N-CH₂-CH₂-O-Tri)[Fig. 2.53. $^1\text{H-NMR}$ Spectrum of AG3 dendrimer]

^{13}C -NMR (75MHz, D₂O) δ ppm: 28.00(N-CH₂-CH₂-CH₂-N), 38.14(N-CH₂-CH₂-CH₂-N), 59.90(N-CH₂-CH₂-OH), 60.62 (N-CH₂-CH₂-O-Tri),63.31(N-CH₂-CH₂-OH),66.69(N-CH₂-CH₂-O-tri), 168.16(Triazine C-N), 169.91(inner C-N-(CH₂-CH₂-O)-₂),171.51(Outer triazine C-O), 175.87(outer C-N-(CH₂-CH₂-O)-₂), 177.20(outer C-N-(CH₂-CH₂-O)-₂), 179.11(Outer Triazine C-O) [Fig.2.54. ^{13}C -NMR Spectrum of AG3 dendrimer].

ESI-Mass: Calculated Molecular Weight: 12044; Obtained 12045 (M+1). [Fig.2.55. ESI-Mass Spectrum of AG3 dendrimer]

Characterization data for BG3 dendrimer [56]

Yield: 72 %

^{13}C -NMR (75 MHz, D₂O) δ ppm: 24.94(N-CH₂-CH₂-CH₂-CH₂-N), 40.08 (N-CH₂-CH₂-CH₂-CH₂-N), 57.00 (N-CH₂-CH₂-O-tri), 58.50 (N-CH₂-CH₂-OH), 59.00 (N-CH₂-CH₂-O-tri), 60.60 (N-CH₂-CH₂-OH), 163.55(Triazine C-N), 165.00(inner C-N-(CH₂-CH₂-O)-₂), 166.51(Outer triazine C-O), 168.82(outer C-N-(CH₂-CH₂-O)-₂), 170.05, (C-N-(CH₂-CH₂-O)-₂) 171.11 (Triazine C-O)[Fig. 2.58.

^{13}C -NMR Spectrum of BG3dendrimer]

FT-IR (Nujol, cm⁻¹) : 3364 (O-H Stretching), 2941 (Aliphatic C-H stretching), 1668, 1621 (aromatic C=N stretching), 1063 (C-O stretching) [Fig.2.56. FT-IR Spectrum of BG3 dendrimer].

^1H -NMR (400 MHz, D₂O) δ ppm: 1.5155-1.5959 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-N),3.2577-3.3050(m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 3.5623-3.6760 (m, 264H, N-CH₂-CH₂-OH), 3.8117-3.9276 (m, 264H, N-CH₂-CH₂-OH), 4.0330-4.1107 (m, 16H, N-CH₂-CH₂-O-),4.1918-4.2476(m,16H,N-CH₂-CH₂-O-)[Fig.2.57. ^1H -NMR Spectrum of BG3 dendrimer]

ESI-Mass: Calculated molecular weight: 12070; Obtained: 12070(M+) [Fig.2.59. ESI-Mass Spectrum of BG3 dendrimer]

Characterization data for CG3 dendrimer

Yield: 71 %.

FT-IR (Nujol, cm⁻¹) : 3368 (O-H), 2947, 2872 (aliphatic C-H), 1639 (C=N of triazine), 1033 (C-O); [Fig.2.60. FT-IR Spectrum of CG3 dendrimer].

^1H -NMR (400MHz, D₂O) δ ppm: 1.3138-1.3353 (m, 4H, m, 4H, N-CH₂-CH₂-CH₂-CH₂-CH₂-N),1.4954-1.5181(m,4H,N-CH₂-CH₂-CH₂-CH₂-CH₂-N),3.4144-3.4333 (m, 4H, CH₂-NH), 3.6833-3.7574 (m, 264H, N-CH₂-CH₂-OH), 3.8829-3.9243 (m, 264H, N-CH₂-CH₂-OH) 4.0953-4.1880(m, 80H, N-CH₂-CH₂-O-tri),4.2432-4.3168 (m, 80H, N-CH₂-CH₂-O-tri)[Fig.2.61. ^1H -NMR Spectrum of CG3dendrimer]

^{13}C -NMR(75MHz,D₂O):25.50(N-CH₂-CH₂-CH₂-CH₂-CH₂-N),30.14(N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 40.44 (N-CH₂-CH₂-CH₂-CH₂-CH₂-N), 59.92 (N-CH₂-CH₂-O-tria), 60.60(N-CH₂-CH₂-OH),63.39 (N-CH₂-CH₂-O-tria),66.69(N-CH₂-CH₂-OH), 168.11(Triazine C-N), 169.99(inner C-N-(CH₂-CH₂-O)-₂),171.55(outerTriazineC-O),175.81(outerC-N-(CH₂-CH₂-O)-₂),177.28(outerC-N-(CH₂-CH₂-O)-₂), 179.11(Triazine C-O)[Fig. 2.62. ^{13}C -NMR Spectrum of CG3dendrimer]

ESI-Mass: Molecular Wt. 12086 found: 12087(M+1) [Fig.2.63.ESI-Mass Spectrum of CG3 dendrimer].

Thin Layer Chromatography (TLC): -Methanol: Chloroform; 7: 3.

RESULT AND DISCUSSION

Table 1. Physical Description of Dendrimer generations (AG0.5-AG3)

Compound	Molecular formula	Appearance	Solubility in water	Surface groups
AG0.5	C ₉ H ₈ Cl ₄ N ₈	White solid	Insoluble	Cl (4)
AG1	C ₂₅ H ₄₈ N ₁₂ O ₈	Brown liquid	Soluble	OH (8)
AG1.5	C ₄₉ H ₄₀ Cl ₁₆ N ₃₆ O ₈	White solid	Insoluble	Cl(16)
AG2	C ₁₁₃ H ₂₀₀ N ₅₂ O ₄₀	Brown liquid	Soluble	OH(32)
AG2.5	C ₂₀₉ H ₁₆₈ Cl ₆₄ N ₁₄₈ O ₄₀	White solid	Insoluble	Cl(64)
AG3	C ₄₆₅ H ₈₀₈ N ₂₁₂ O ₁₆₈	Brown liquid	Soluble	OH (128)

Table 2. Physical Description of Dendrimer generations (BG0.5-BG3)

Compound	Molecular formula	Appearance	Solubility in water	Surface groups (number)
BG0.5	C ₁₀ H ₁₀ Cl ₄ N ₈	White solid	Insoluble	Cl (4)
BG1	C ₂₆ H ₅₀ N ₁₂ O ₈	Brown liquid	Soluble	OH (8)
BG1.5	C ₅₀ H ₄₂ Cl ₁₆ N ₃₆ O ₈	White solid	Insoluble	Cl(16)
BG2	C ₁₁₄ H ₂₀₂ N ₅₂ O ₄₀	Brown liquid	Soluble	OH(32)
BG2.5	C ₂₁₀ H ₁₇₀ N ₁₄₈ O ₄₀ Cl ₆₄	White solid	Insoluble	Cl(64)
BG3	C ₄₆₆ H ₈₁₀ N ₂₁₂ O ₁₆₈	Brown liquid	Soluble	OH(128)

Table 3. Physical Description of Dendrimer generations (CG0.5-CG3)

Compound	Molecular formula	Appearance	Solubility in water	Surface groups (number)
CG0.5	C ₁₂ H ₁₄ Cl ₄ N ₈	White solid	Insoluble	Cl (4)
CG1	C ₂₈ H ₅₄ N ₁₂ O ₈	Brown liquid	Soluble	OH (8)
CG1.5	C ₅₂ H ₄₆ Cl ₁₆ N ₃₆ O ₈	White solid	Insoluble	Cl(16)
CG2	C ₁₁₆ H ₂₀₆ N ₅₂ O ₄₀	Brown liquid	Soluble	OH(32)
CG2.5	C ₂₁₂ H ₁₇₄ N ₁₄₈ O ₄₀ Cl ₆₄	White solid	Insoluble	Cl(64)
CG3	C ₄₆₈ H ₈₁₄ N ₂₁₂ O ₁₆₈	Brown liquid	Soluble	OH(128)

In infra-red range of chlorine ended (G0.5, G1.5 and G2.5) dendrimers, characteristic retention groups showed up in run of 1680-1780 cm⁻¹ for C=N extending, 1055-1065 cm⁻¹ of C-O extending and 750-780 cm⁻¹ for C-Cl extending, while O-H extending of hydroxyl bunches was truant. In infrared range of hydroxyl ended compounds appeared all characteristics flag as over but C-C extending and an extra top for -OH recurrence was gotten at 3350- 3400 cm⁻¹. All synthesized dendrimer were characterized by 1H-NMR spectroscopy. 1H-NMR range of AG0.5 dendrimer appeared crests at δ 1.8031-1.8649 ppm and δ 3.4304- 3.4660 ppm which compare to 1,3-propane diamine moiety. 1H-NMR range of BG0.5 dendrimer appeared crests at δ 1.5583- 1.6603 ppm and δ 3.2929-3.3010 ppm which compare to 1,4-butane diamine moiety. 1H-NMR range of CG0.5 dendrimer appeared multiplets at δ 1.2898-1.3558, 1.4941-1.5383 and 3.2445- 3.2881 ppm for 1,6-hexanediamine moiety. 13C-NMR range of AG0.5 dendrimer appeared crests at δ 28.70, 38.18 ppm for propyl carbons and crests at δ 166.61, 171.66 ppm for triazine carbons. 13C-NMR range of AG1 dendrimer appeared crests at δ 28.72, 38.22 ppm for propyl carbons, at δ 50.66, 60.18 ppm for diethanolamine carbons and at δ 166.62, 171.16 ppm for triazine carbons. 13C-NMR range of AG1.5 dendrimer appeared crests at δ 28.70, 38.10 ppm for propyl moiety, δ 51.01, 60.58 ppm for diethanolamine moiety and crests at δ 163.01, 168.66, 170.18 ppm for triazine carbons. 13C-NMR Range of AG2 dendrimer appeared crests at δ 28.50, 38.08 ppm for propyl carbons, δ 61.28, 65.70 ppm for external diethanolamine carbons 60.81, 63.88 ppm for inward diethanolamine carbons and δ 163.01, 168.66, 170.78, 172.28 ppm for triazine carbons. 13C-NMR range of AG2.5 dendrimer appeared crests at δ 28.80, 38.20 ppm for propyl carbons, δ 61.20, 65.50 ppm for external diethanolamine carbons, δ 60.00, 63.30 ppm for inward diethanolamine carbons, δ 164.10, 166.40, 171.70, 172.50, 174.00 ppm for triazine carbons. 13C-NMR range of AG3 dendrimer appeared crests at δ 28.00, 38.14 ppm for propyl carbon, δ 59.50, 63.31 ppm for external diethanolamine carbon, δ 60.62, 66.69 ppm for internal diethanolamine carbon and δ 168.16, 169.91, 171.51, 175.87, 177.20, 179.11 ppm for triazine carbons. 13C-NMR range appeared δ 25.20, 30.05, 40.45 for center portion, 60.06, 63.30 for inward diethanolamine portion δ 61.23, 65.53 for external diethanolamine portion and δ 164.11, 166.41, 168.81, 171.74, 172.57, 174.94 triazine portion of structure. 13C-NMR range of CG3 dendrimer, 25.50, 30.14, 40.44 for center, 59.92, 60.60 for external diethanolamine portion, 63.39, 66.69 for internal diethanolamine portion and 168.11, 169.99, 171.55, 175.81, 177.28, 179.11 triazine portion of structure. 13C-NMR range of center compound CG0.5 appeared crests at δ 25.20, 25.29, 30.52, 40.52, 41.47 ppm for aliphatic carbons of hexane gather at center and crests δ 168.34, 169.40 for triazine portion of structure. 13C-NMR range of CG1 dendrimer showed additional crests at δ 58.04 and δ 61.04 for diethanolamine portion of the structure. 13C-NMR range of CG1.5 dendrimer appeared crests at δ 25.20, 25.29, 30.52 for center carbons, δ 41.57, 61.04

for diethanolamine portion and δ 163.01, 166.66, 170.18, 172.28 triazine portion of structure. ^{13}C -NMR range of CG2 dendrimer appeared δ 25.70, 30.31, 40.81 for center, δ 60.81, 63.78 for inward diethanolamine portion, δ 61.28. All the synthesized dendrimer eras were characterization by Electrospray ionization mass spectrometry. Gotten atomic particle crests compares to calculated atomic weights e.g. gotten atomic particle crests for AG3, BG3, CG3 were 12044, 12070 and 12087 individually though calculated atomic weight were 12043, 12070 and 12086 Daltons separately. Atomic particle crests of AG2, BG2, CG2 were gotten at 2926, 2942 and 2971 esteem while found atomic weights were 2925, 2941 and 2970 Daltons individually.

Table 4. Elemental data of Dendrimer

Compound	Percentage of Elements					
	Theoretical			Practical		
	C	H	N	C	H	N
AG3	44.83	5.80	54.65	38.70	6.98	24.67
BG3	44.77	5.46	55.67	38.85	6.78	25.43
CG3	44.49	5.32	57.32	38.41	6.85	25.78

All dendrimer generations were further characterized by elemental analysis. As shown in Table 2.5.4., all the experimental percentage of C, H and N for all dendrimer generation matched the calculated percentage of C, H, and N. The results of TGA of dendrimer generations are displayed in Fig 2.64-2.73. Thermal decomposition patterns for both chlorine terminated compound and hydroxyl terminated dendrimer were different. It was observed that full generation dendrimers lost 16-20% weight up to 160-170 °C. As full generation dendrimers were terminated by hydroxyl groups bound moisture may have contributed to initial weight loss. Dendrimers AG1, AG2 and AG3 decomposed in the range of 210-420 °C, 250-430 °C and 280-420 °C respectively. It was found that dendrimers BG1, BG2 and BG3 decomposed in the range of 150-450 °C, 200-500 °C and 290-450 °C respectively. It was revealed that CG1, CG2 and CG3 dendrimers were decomposed in the range of 235-345 °C, 260-400 °C and 310-450 °C respectively. Half generation dendrimers AG0.5, BG0.5 and CG0.5 were decomposed in the range of 310-420 °C, 310-430 °C and 290-350 °C respectively. Similarly, AG1.5, BG1.5, CG1.5, AG2.5, BG2.5 and CG2.5 dendrimer were decomposed in the range of 340-420 °C, 320-440 °C, 325-410 °C, 330-430 °C, 350-440 °C and 325-400 °C respectively. Chlorine terminated half generation dendrimers showed overall higher thermal stability compared to full generation dendrimers. Thermal decomposition data are furnished in Table 2.5.5-2.5.7. The integral procedural decomposition temperature (IPDT) and T50% were calculated for all dendrimers by Doyle's method. Overall thermal stability of dendrimers was increased with increase in generation.

Table 5. Glass Transition temperatures of dendrimer (AG0.5-CG3)

Products		Glass Transition Temperature (°C)	
		On set	Mid-Point
Hydroxyl terminated compounds	AG 1	-6.5	-6.9
	AG 2	-7.6	-4.4
	AG 3	0.0	1.7
Chlorine terminated compounds	AG 0.5	-14.0	-15.4
	AG 1.5	-15.4	-13.8
	AG 2.5	-11.8	-11.7
Hydroxyl terminated compounds	BG 1	-6.7	-7.4
	BG 2	-6.1	-4.8
	BG 3	0.5	3.4
Chlorine terminated compounds	BG 0.5	-14.6	-15.3
	BG 1.5	-14.6	-13.7
	BG 2.5	-14.8	-12.0
Hydroxyl terminated compounds	CG 1	-7.6	-6.4
	CG 2	-6.4	-4.8
	CG 3	0.8	2.2
Chlorine terminated compounds	CG 0.5	-12.2	-17.8
	CG	-16.1	-12.9

1.5		
CG	-13.8	-11.4
2.5		

Table 6.Glass Transition temperatures of PAMAM dendrimer

Products	Glass Transition Temperature (°C)	
	On set	Mid-Point
olyamidoamine (PAMAM) dendrimers ^a	-14	-12
	-11	-4
	-7	0

^a Reported for PAMAM dendrimer by Uppuluri et al

Dendrimer having regularly moo values of glass move temperatures. It is decided by DSC strategy [Table 2.5.8-2.5.9]. Glass move temperatures of dendrimer era were of moo esteem for both full and half era dendrimers which were comparable to PAMAM dendrimers. Glass move temperatures of hydroxyl ended triazine dendrimers were small higher than chlorine ended half era dendrimers.. It was moreover watched that with increment in era number or atomic weight, number of branches and terminal bunches of dendritic engineering were expanded which driven to diminished chain versatility and expanded glass move temperature. Hence, in case of both full and half era dendrimers, glass move temperature expanded with increment in atomic weight or era number.

Surface Modification ofDendrimer

The surface modification of 4.0 generation dendrimer was done by conjugation with polyethylene glycol (PEG) and different polyphenols like 2,4-dihydroxy Benzoic Acid(BA), Ferulic acid(FA) and Gallic acid(GA).

Preparation of PEG Conjugated 4.0 TRIAZINE Dendrimer

The conjugation of 4.0 era TRIAZINE dendrimer with polyethylene glycol (PEGylation) was done utilizing epichlorohydrin as a cross connecting operator. Six distinctive polyethylene glycols of distinctive atomic weight (200, 300, 400, 600, 2000, and 4000) were utilized for PEGylation. The status of response was checked by copper sulfate test, UV, IR, NMR and mass spectroscopy.

Surface Modification of 4.0 Generation TRIAZINE Dendrimer byPolyphenols

For surface modification of 4.0 generation TRIAZINE dendrimer, three different Polyphenols were used:

1. 2,4-dihydroxy Benzoic Acid(BA)
2. Ferulic acid(FA)
3. Gallic acid(GA)

Characterization of Prepared Surface Modified Dendrimer

Identification of Dendrimer by Color Reaction

The distinctive PEGylated and surface adjusted dendrimers were firstly distinguished by response of copper sulfate watery arrangement (1% w/v) with surface altered dendrimer arrangement (0.1% w/v) in methanol. The concentrated of violet color of 4.0 G dendrimers was watched. The concentrated of violet color of 4.0 G dendrimers diminishes after PEGylation and other surface alterations, due to connection of diverse bunches on free – NH₂ bunches which is mindful for violet color.

Ultraviolet Spectroscopy

The dendrimer test and surface adjusted dendrimer tests were taken as arrangement (0.01% w/v) in refined water and checked within the run of 200 nm to 500 nm against refined water in UV spectrophotometer.

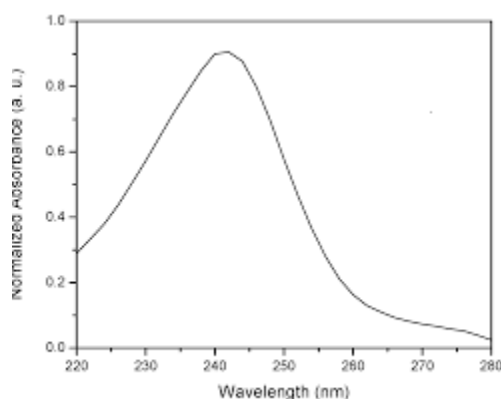


Fig. No. 2: UV spectra of 4.0 generation TRIAZINE dendrimer

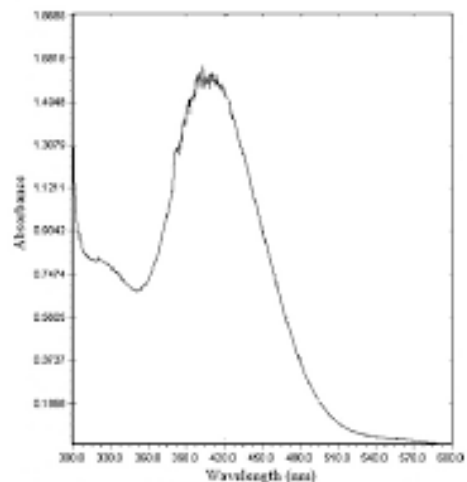


Fig. No. 3: UV spectra of 4.0 generation TRIAZINE dendrimer-BA conjugate

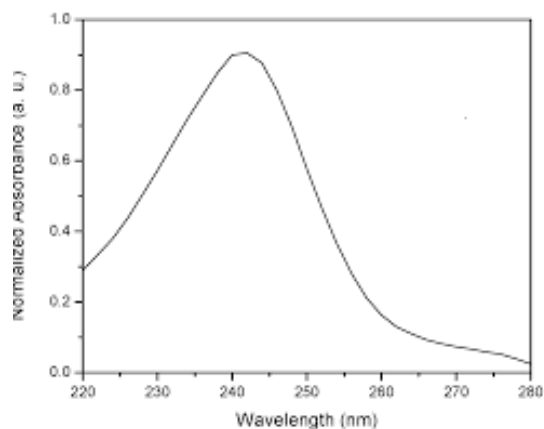


Fig. No. 4: UV spectra of 4.0 generation TRIAZINE dendrimer-FA conjugate

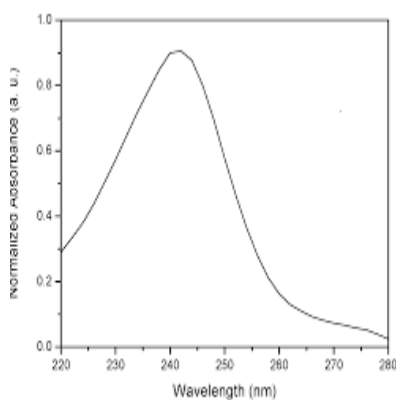


Fig. No. 4: UV spectra of 4.0 generation TRIAZINE dendrimer - GA conjugate

Table No. 6: max value of 4.0 generation TRIAZINE dendrimer and different conjugates

S.No.	Type of Conjugate	λ_{\max} [in nm]
1	4.0 Generation TRIAZINE Dendrimer	248.5
2	4.0 Generation TRIAZINE Dendrimer - PEG conjugate	353.0
3	4.0 Generation TRIAZINE Dendrimer -BA Conjugate	258.0
4	4.0 Generation TRIAZINE Dendrimer -FA Conjugate	241.0
5	4.0 Generation TRIAZINE Dendrimer - GA conjugate	245.0

IR Spectroscopy

IR spectra of the dendrimer sample and surface-modified dendrimer samples were taken using FTIR-2000A (ABB Spectrophotometer). Surface group changes, due to surface modification of dendrimer, were studied through IR spectra. Various peaks were interpreted for different groups.

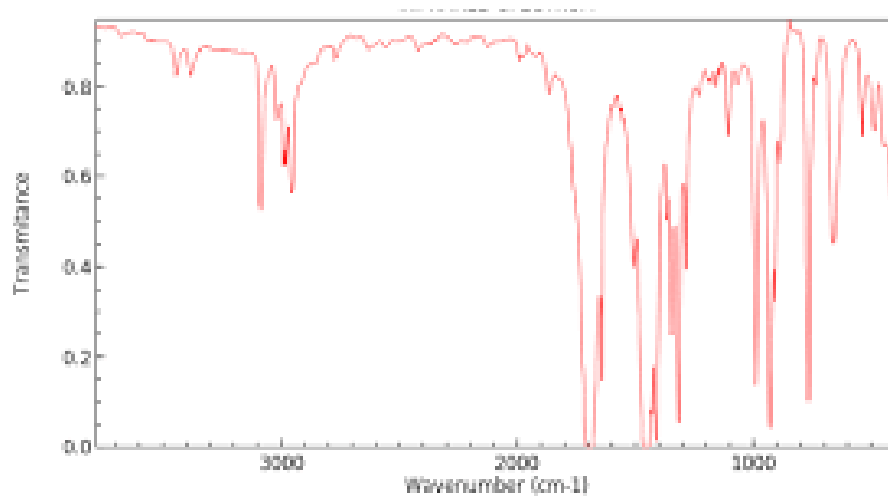


Fig. No. 6: IR spectra of 4.0 generation TRIAZINE dendrimer

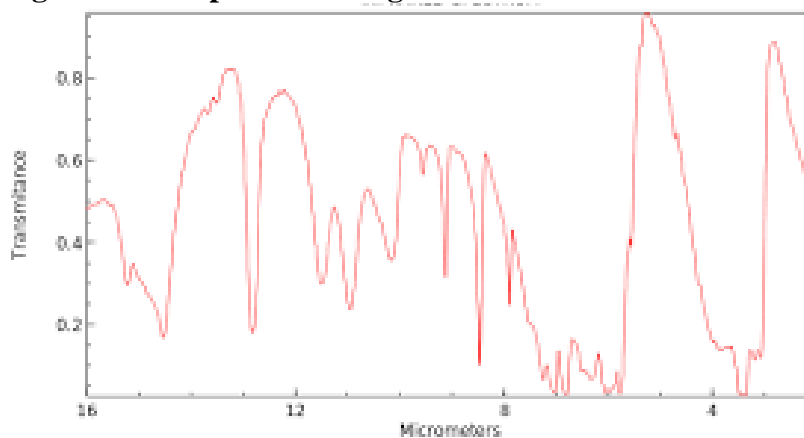


Fig. No. 7: IR spectra of 4.0 TRIAZINE dendrimer-PEG conjugate

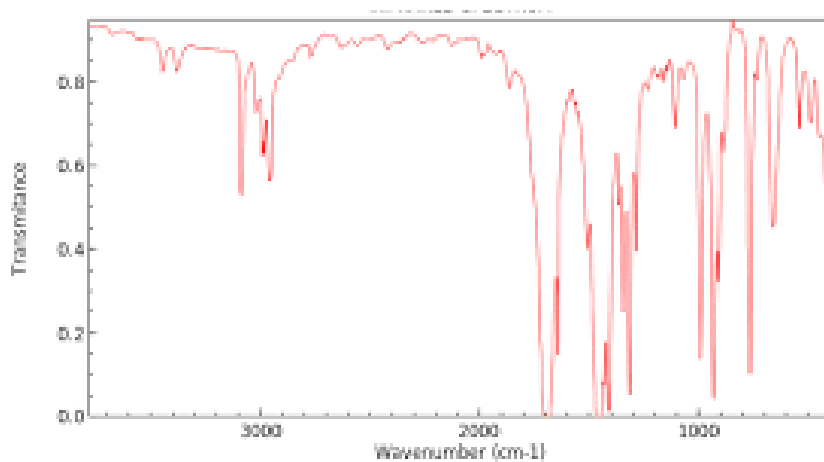


Fig. No. 8: IR spectra of 4.0 generation TRIAZINE dendrimer-BA conjugate

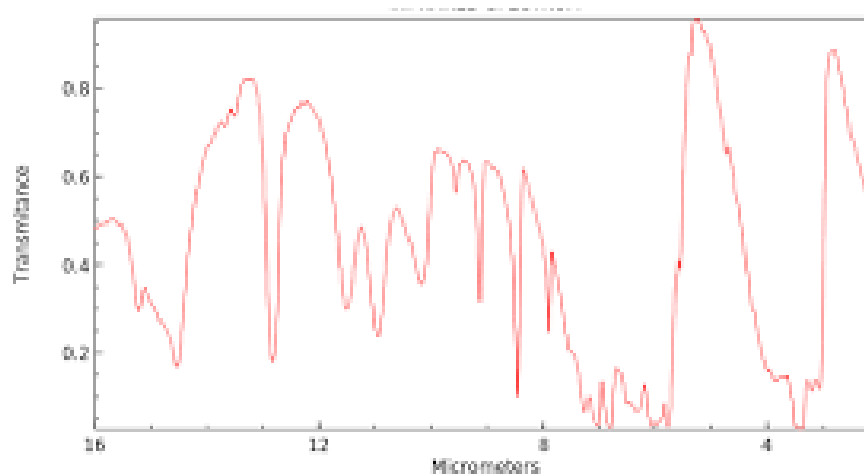
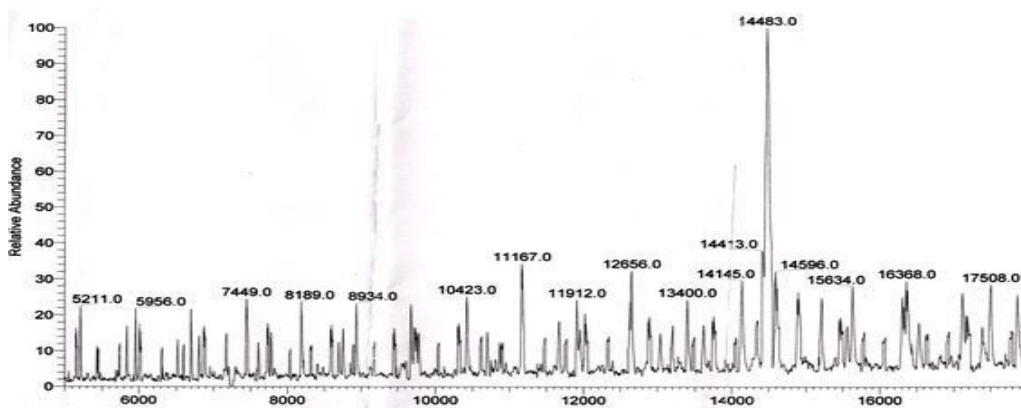


Fig. No. 9: IR spectra of 4.0 generation TRIAZINE dendrimer-FA conjugate

Mass Spectroscopy

To confirm the molecular weight of 4.0 generation TRIAZINE dendrimer and surface modified systems mass spectral analysis was performed. The electrospray mass spectra were recorded on a MICROMASS QUATTRO II triple mass spectrometer. The increase in mass further confirmed the conjugation of different molecules to 4.0 generation TRIAZINE dendrimer. The Mass Spectra of 4.0 generation TRIAZINE dendrimer and



different conjugations are shown in Fig.

Fig. No. 10: Mass spectra of 4.0 generation TRIAZINE dendrimer

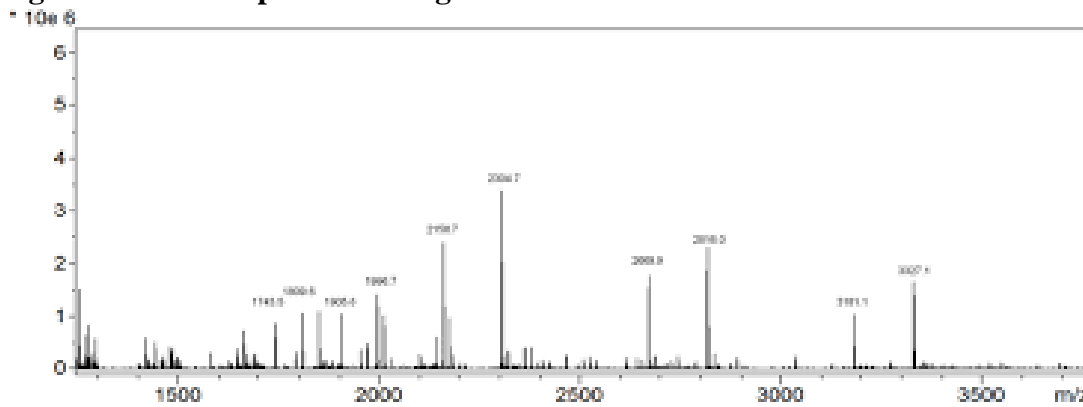


Fig. No. 11: Mass spectra of 4.0 generation TRIAZINE dendrimer –PEG

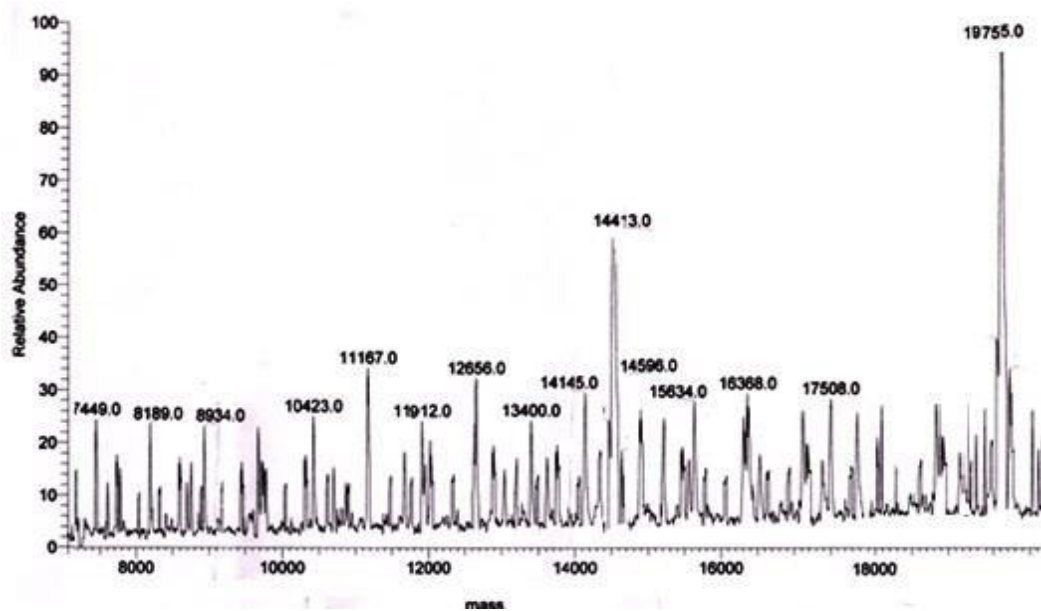


Fig. No. 12: Mass spectra of 4.0 generation TRIAZINE dendrimer –FA Conjugate

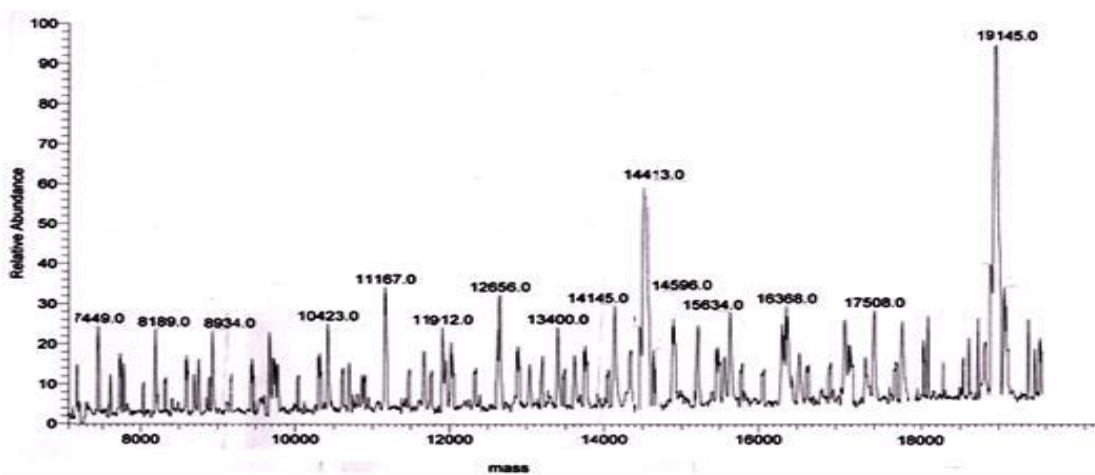


Fig. No. 13: Mass spectra of 4.0 generation TRIAZINE dendrimer -GA conjugate

The 4.0 generation TRIAZINE dendrimer shows m+1 peak at 14483 and PEGylated dendrimer shows m+1 peak at 24067. These values showed that 16 chains of PEG are attached on 4.0 generation TRIAZINE dendrimer. Mass spectral analysis for different conjugations of Polyphenols, indicate the change in the molecular weight of 4.0 generation TRIAZINE dendrimer on conjugation.

Summary and Conclusion

A arrangement of triazine based dendrimers vary in spacer length of center was synthesized by disparate strategy utilizing triazine trichloride and diethanolamine as branching units. Synthesized dendrimers were characterized by ghostly methods such as FT-IR, ¹H-NMR, ¹³C-NMR, ESI-Mass spectrometry and Essential analysis. Thermal solidness of dendrimer was surveyed by Thermo gravimetric investigation. T50% and fundamentally procedural deterioration temperature were unflinching by Doyle's strategy. Warm soundness expanded with the increment in era number. Differential Checking Calorimetry was utilized to decided glass move temperature of dendrimers extending between -17 to 0. To get a unpleasant estimation of estimate of dendrimer, hydrodynamic span was measured utilizing inborn consistency. Hydrodynamic span too expanded with increment in era number. A arrangement of triazine based dendrimers utilizing distinctive diamine N,N'-bis(4,6-dichloro- 1, 3,5-triazin-2-yl) alkane diamine with number of carbons 3, 4 and 6 as center which shift in spacer length were synthesized and detailed. The amalgamation was carried out in orderly way, watching appropriate response conditions and refinement and reflux wherever required for getting superior abdicate. Synthesized dendrimers were assist characterized by FT-IR, ¹H-NMR, ¹³C-NMR, ESI-Mass Spectroscopy, Natural Examination additionally warm properties were assessed.

References

1. Ahuja S, Scypinski S. (2001). *Hand book of Modern Pharmaceutical Analysis*. Academic Press, Vol-3, pp 1-22
2. Kealey K, Haines PJ. (2002). *Analytical Chemistry*. Bios scientific Publisher Limited, (1stedⁿ), pp 1-2
3. Shethi PD. (2001). *Quantitative analysis of pharmaceutical formulation*. CBS Publishers & Distributors, New Delhi, (1stedⁿ), pp 1-7
4. Munson JW. (2001). *Pharmaceutical Analysis- Modern Methods*. (Part-B), Marcel Dekker publisher's, pp 16-18

5. Sharma BK. (2005). *Instrumental methods of chemical analysis*. Goel Publishing house, Meerut,(24thedⁿ), pp C-286-C-311
6. Gopinath R, Rajan S, Meyyanathan SN, Krishnaveni N, Suresh BA.(2007). RP-HPLC method for simultaneous estimation of Solifenacin and aceclofenac in tablets. *Indian Journal of Pharmaceutical Science*, 69(2):137-140
7. Mahaparale PR, Sangshetti JN,Kuchekar BS. (2007). Simultaneous Spectrophotometric estimation of aceclofenac and Solifenacin in tablet dosage form. *Indian Journal of Pharmaceutical Science*, 69(3):289-292
8. Patel PM, Desai HJ, Patel RC, Patel NM. (2007). Spectrophotometric method for estimation of rabeprazole. *Indian Journal of Pharmaceutical Science*, 69(3):318-320
9. Prasad CVN, Parihar C, Sunil P,Parimoo P.(1997). Simultaneous determination of amlodipine HCL, Hydrochlorthiazide and atenolol in combined formulation derivative spectroscopy. *Journal of Pharmaceutical and Biomedical Analysis*, 39:877-884
10. Sockalingam A,Narayanareddy I,pitchaimuthu S, SeshaiiahKS. (2005). Simultaneous quantification of stavudine, lamivudine and nevirapine by UV spectroscopy, reverse phase HPLC and HPTLC. *Journal of Pharmaceutican and Biomedical Analysis*, 39:801-804
11. Shaikh KA, Devkhile AB. (2008). Simultaneous Determination of Aceclofenac, Solifenacin, and Chlorzoxazone by RP-HPLC in Pharmaceutical Dosage form.*Journal of Chromatographic Science*, 46(7): 649-652
12. Bakshi M, Singh S. (2002). Development of validated stability-indicating assay methods-critical review.*Journal of Pharmaceutical and Biomedical Analysis*,28:1011-1040
13. BurkhardA, DanielR, ThomasR, Stephan B, Werner U (2003) BMC. Biomedical A.B. chromatography17(4):268-275
14. Bakshi M, Singh B, Singh A, Singh S. (2001). The ICH guidelines in practice: stress degradation studies on omidazole and development of a validated stability-indicating assay. *Journal of Pharmaceutical and Biomedical Analysis*, 26:891-897
15. Bakshi M, Singh S. (2004). The ICH guidelines in practice: establishment of inherent stability of secnidazole and development of a validated stability- indicating HPLC assay method. *Journal of Pharmaceutical and Biomedical Analysis*, 22:1-7

16. Singh S, Singh B, Bahuguna R, Wadhwa L, Saxena R. (2006). Stress degradation studies on ezetimibe and development of validated stability-indicating HPLC assay. *Journal of Pharmaceutical and Biomedical Analysis*, 41:1037-1040
17. HongDD, Shah M, CarstensenJT, RhodesCT. (2005). *Drug Stability Principle and Practices*. Marcel Dekker, Inc. New York, pp 358-368
18. International Conference on Harmonization (ICH), Q2A *Text on Validation of Analytical Procedures: Definitions and Terminology* (Vol. 60) US FDA Federal Register, 1995
19. International Conference on Harmonization (ICH), Q2B, *Validation of Analytical Procedures: Definitions and Terminology* (Vol. 60) US FDA Federal Register, 1995
20. Martindale. (2002). *The Complete Drug Reference*. Pharmaceutical Press: London, (33rdedⁿ), pp 321.3
21. Anonymous. (1996). Indian pharmacopoeia. Controller of publication, Delhi, vol.I, pp 347-348
22. Lalhariatpulli, TC, KawathekarN. (2005). Derivative spectrophotometric estimation of Pioglitazone and Metformin HCL. *Indian Drugs*, 42(11): 740-743
23. Anonymous. (2002). British Pharmacopoeia. The stationary office, London, vol.I, pp 1123-1124
24. Rentnall, AE, ClarkeGS. (1998). *Analytical Profiles of Drug Substances and Excipients*, edited by Brittain HG, Academic Press, London, vol-25, pp 243-246
25. Subramaniam G, Shetty R, Agarwal S, Pandey S, Udupa N. (2005). Simultaneous reverse phase HPLC estimation of Solifenacin and rofecoxib in tablets. *Indian Journal of Pharmaceutical Science*, 2(67):247-249
26. John M, John FA, Donald J, Birkett A. (2001). A simple HPLC assay for urinary Solifenacin metabolites and its use to characterize the c₃h mouse as a model for Solifenacin metabolism studies. *Clinical and Experimental Pharmacology and Physiology*, 2(11):209 – 217