Data Article

Dataset of ^{1}H-nuclear magnetic resonance and mass spectra of surface modified Poly(amidoamine) dendrimers with LFC131 peptide

Chuda Chittasupho [a], Chaiyawat Aonsri [b], Witcha Imaram [b,c,*]

[a] Department of Pharmaceutical Sciences, Faculty of Pharmacy, Chiang Mai University, Chiang Mai, Thailand
[b] Department of Chemistry and Center of Excellence for Innovation in Chemistry, Faculty of Science, Kasetsart University, Bangkok, Thailand
[c] Special Research Unit for Advanced Magnetic Resonance, Department of Chemistry, Faculty of Science, Kasetsart University, Bangkok, Thailand

ARTICLE INFO

ABSTRACT

In the present article, we describe the spectroscopic data of poly(amido)amine dendrimers generation 5.0 (G5 PAMAM) conjugated with LFC131 peptide at different specified reaction points. The raw data regarding the ^{1}H NMR and mass spectra of G5 PAMAM dendrimers with and without LFC131 peptide conjugation and with or without FITC labelled are presented for comparison.

© 2021 The Authors. Published by Elsevier Inc. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/)

* Corresponding author.
E-mail address: witcha.i@ku.ac.th (W. Imaram).

Specifications Table

| Subject	Chemistry
Specific subject area	Structural characterization
Type of data	Figure
How data were acquired	The data was acquired on Bruker Avance III HD 400 MHz nuclear magnetic resonance spectrometer, Bruker AVANCE III 500 MHz nuclear magnetic resonance spectrometer, and Micro flex mass spectrometer.
Data format	Raw
	Analyzed
Parameters for data collection	NMR samples were prepared with deuterium oxide or methanol (methanol-d4). The sinapic acid was used as a matrix for MALDI-TOF analysis.
Description of data collection	NMR spectral data were recorded at 25 °C on a Bruker Avance III HD 400 MHz NMR spectrometer at Kasetsart University or Bruker AVANCE III 500 MHz NMR spectrometer at the Suranaree University of Technology using standard Bruker pulse programs (Bruker BioSpin GmbH).
Data source location	Institution: 1) Kasetsart University and 2) Suranaree University of Technology
	City/Town/Region: 1) Bangkok and 2) Nakhon Ratchasima
	Country: Thailand
	Latitude and longitude (and GPS coordinates, if possible) for collected samples/data: 1) 13°50′32.96″ N 100°34′2.98″ E and 2) 14°52′13.49″ N 102°01′15.19″ E
Data accessibility	With the article.
Related research article	Chittasupho, C; Aonsri, C; and Imaram, W. Targeted dendrimers for antagonizing the migration and viability of NALM-6 lymphoblastic leukemia cells, *Bioorganic Chemistry*, 107, (2021), 104601. doi:10.1016/j.bioorg.2020.104601

Value of the Data

- The dataset represents NMR data and MS data used for characterization of PAMAM dendrimer-based multifunctional conjugates synthesized through partially acetylation, conjugation with fluorescein isothiocyanate (FITC), and conjugation with the LFC131 peptide on the surface primary amine groups of the generation 5 (G5) PAMAM dendrimer.
- The data can benefit researchers who are interested in engineering new dendrimers with multifunctional capabilities as it can serve as a benchmark for the synthesis and characterization of new targeted drug delivery systems.
- The NMR data can be used as references for chemical shifts of other peptide conjugated PAMAM dendrimers and the MS data can be used to estimate the degree of conjugation.

1. Data Description

The dataset contains ^{1}H NMR data obtained through sequential synthetic modifications on the surface of the fifth generation of poly(amidoamine) dendrimer (G5 PAMAM) (see Fig. 1 for its ^{1}H NMR spectrum). The ^{1}H NMR spectra of the modified surface by acetylation (G5 PAMAM-Ac, Fig. 2) and conjugation with FITC (G5 PAMAM-Ac-FITC, Fig. 3) were recorded on Bruker Avance III HD 400 MHz NMR spectrometer at Kasetsart University. The ^{1}H NMR spectra of the conjugated LFC131 peptide with partially acetylated PAMAM (LFC131-G5 PAMAM-Ac, Fig. 4) and its corresponding unconjugated mixture (Fig. 5) were recorded on Bruker AVANCE III 500 MHz NMR spectrometer at the Suranaree University of Technology. To estimate the number of conjugated peptides on the dendrimer, a calculation could be made from the mass difference obtained from the MALDI-TOF analyses of G5 PAMAM-Ac (Fig. 6A) and LFC131-G5 PAMAM-Ac (Fig. 6B). The MALDI-TOF mass spectrum (Fig. 6C) of the unconjugated mixture of LFC131 and G5 PAMAM-Ac
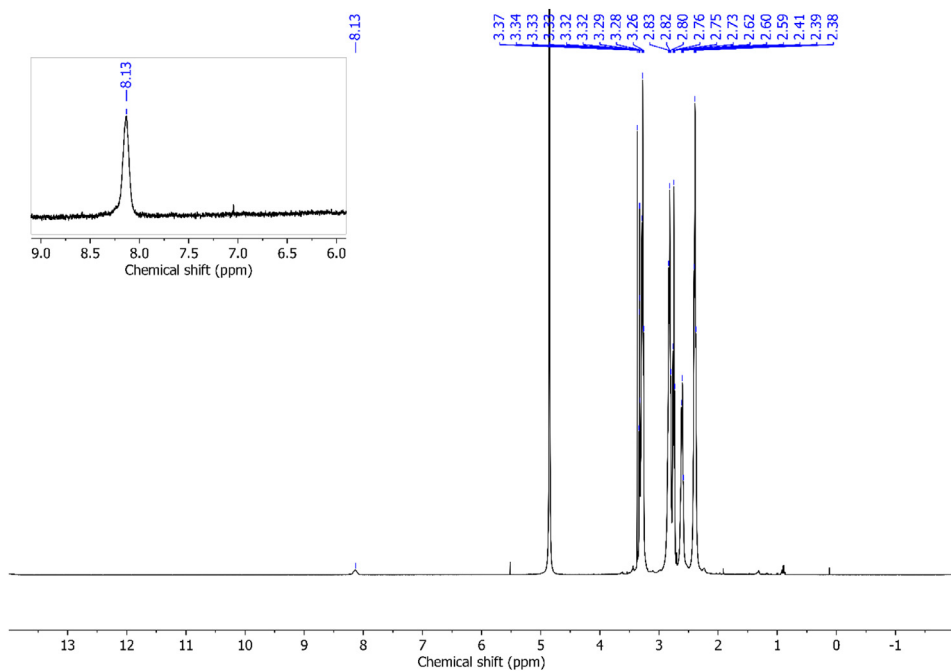
Fig. 1. The 1H NMR spectrum of G5 PAMAM (400 MHz, MeOH-d4).
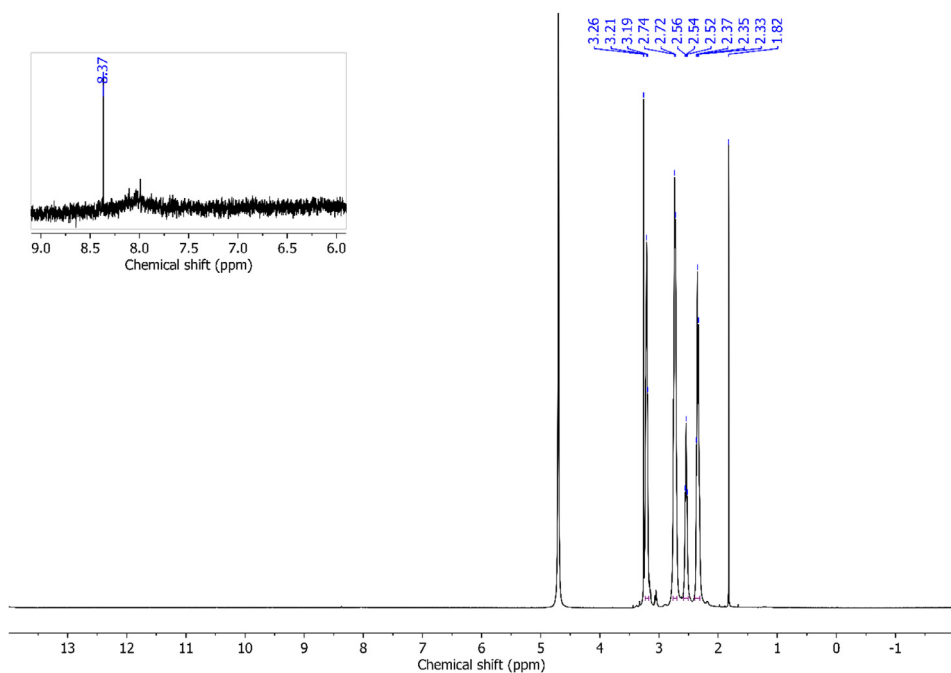
Fig. 2. The ^{1}H NMR spectrum of G5 PAMAM-Ac (400 MHz, Deuterium Oxide).
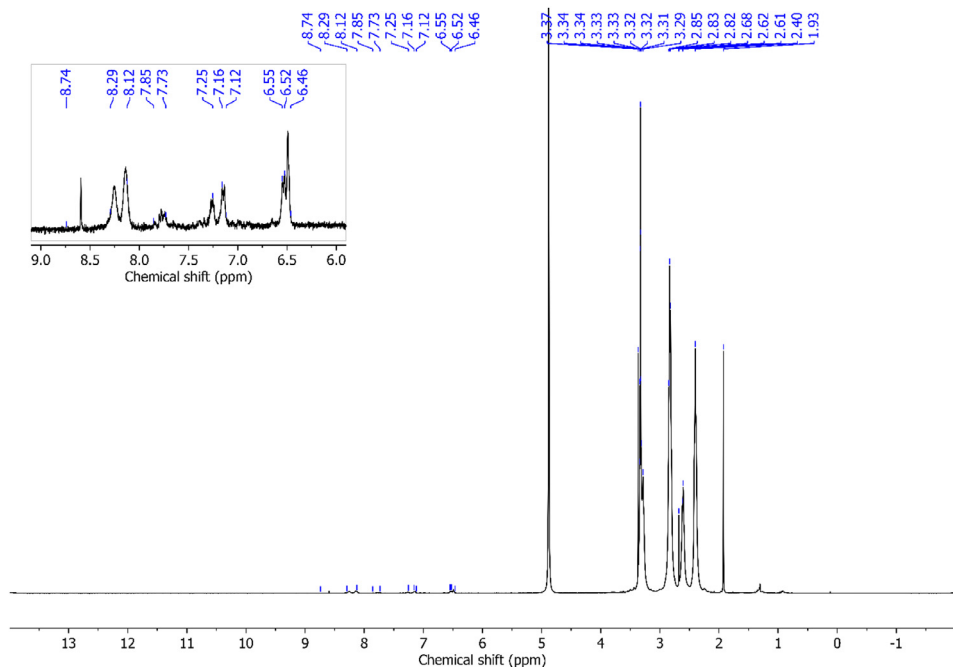
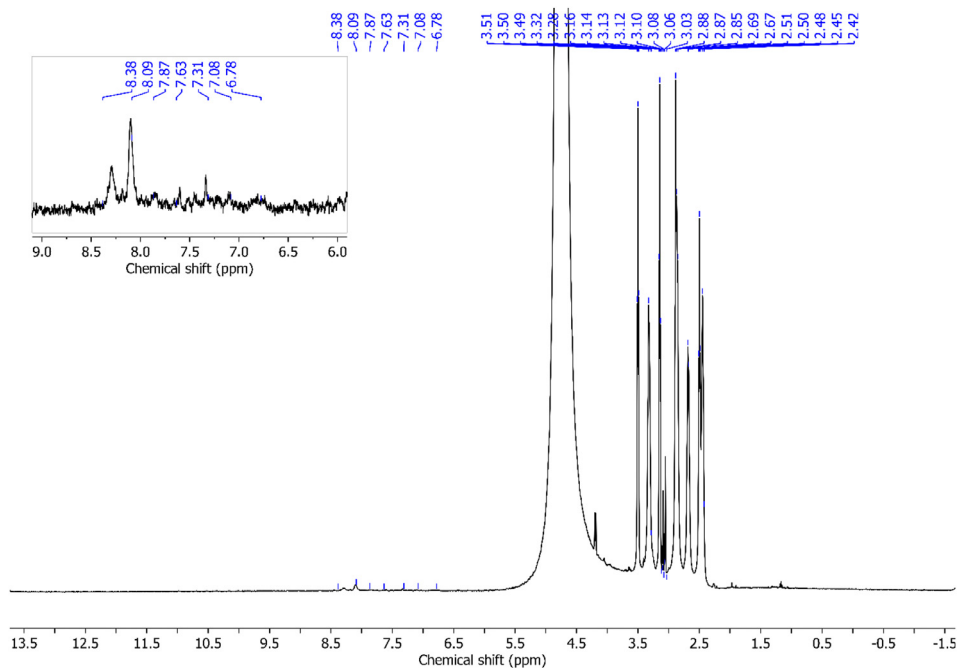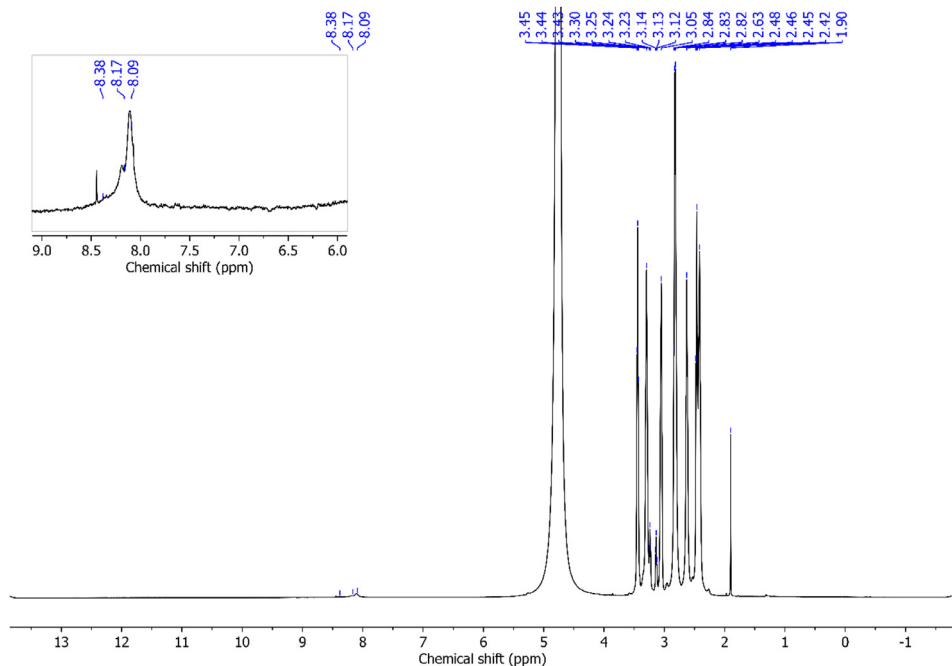**Fig. 3.** The ^{1}H NMR spectrum of G5 PAMAM-Ac-FITC (400 MHz, MeOH-d4).

Fig. 4. The ^{1}H NMR spectrum of LFC131-G5 PAMAM-Ac (500 MHz, Deuterium Oxide).

Fig. 5. The ¹H NMR spectrum of an unconjugated mixture of LFC131 and G5 PAMAM-AC without a coupling agent (500 MHz, Deuterium Oxide).

was provided for additional comparison. The raw NMR and MALDI-TOF MS data are shared as supplemental files in the form of FID files and ascii files, respectively.

1.1. ¹H NMR spectra of G5 PAMAM and partially acetylated G5 PAMAM

Partially acetylation (approximately 19%) of G5 PAMAM dendrimers (G5 PAMAM-Ac) was characterized by ¹H NMR spectroscopy (Fig. 2), δ (ppm) = 3.20 (m, -CH₂- G5 PAMAM), 2.73 (m, -CH₂- G5 PAMAM), 2.54 (m, -CH₂- G5 PAMAM), 2.35 (m, -CH₂- G5 PAMAM), 1.82 (s, -CH₃ Ac). In comparison to the ¹H NMR of G5 PAMAM (Fig. 1), the presence of the acetyl groups on PAMAM dendrimer was confirmed by ¹H NMR signal at 1.82 ppm.

1.2. ¹H NMR spectra of FITC conjugated on partially acetylated G5 PAMAM dendrimers

FITC was conjugated to partially acetylated G5 PAMAM dendrimers, which was confirmed by the presence of thiourea bond in ¹H NMR spectrum (Fig. 3), δ (ppm) = 8.29 (m, -NH-), 8.12 (bs, -NH-), 7.73 (m, Ar-H), 7.25 (m, Ar-H), 7.14 (m, Ar-H), 6.52 (m, Ar-H) 3.23 (m, -CH₂- G5 PAMAM), 2.83 (m, -CH₂- G5 PAMAM), 2.61 (m, -CH₂- G5 PAMAM), 2.40 (m, -CH₂- G5 PAMAM), 1.93 (s, -CH₃ Ac). The thiourea moiety was indicated by the chemical shifts of N–H signals at 8.29 and 8.12 ppm. The chemical shifts of 7.73 to 6.46 ppm are assigned to aromatic protons of FITC.
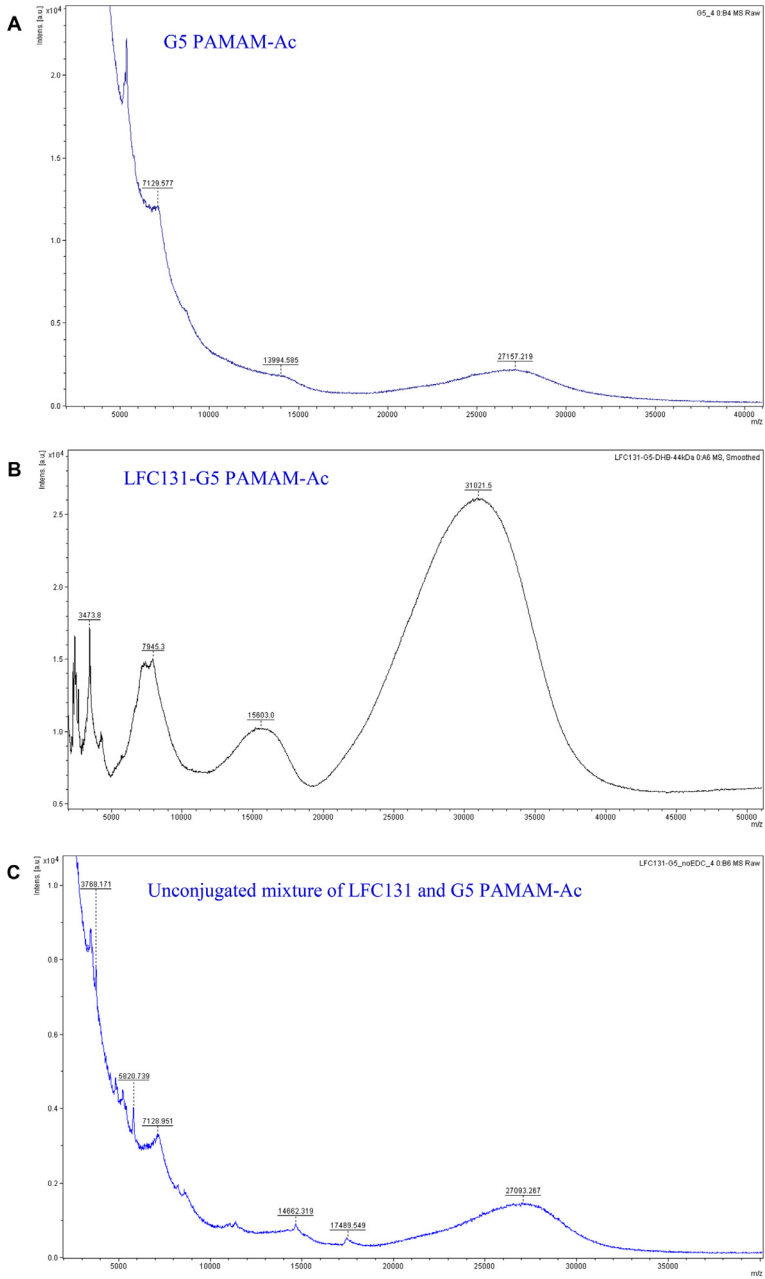
Fig. 6. MALDI-TOF spectra of dendrimers. (A) G5 PAMAM-Ac (B) LFC131-G5 PAMAM-Ac (reaction with EDC). (C) unconjugated mixture of LFC131 and G5 PAMAM-Ac. Dendrimers were dissolved in purified water (10 mg/mL) and sinapic acid was used as a matrix for analysis.

1.3. ^{1}H NMR spectra of LFC131 peptide conjugated G5 PAMAM dendrimers

The partially acetylated G5 PAMAM dendrimers (G5 PAMAM-Ac) were conjugated with LFC131 peptide, the LFC131 peptide conjugated G5 PAMAM dendrimers was characterized by ^{1}H NMR spectroscopy (Fig. 4), δ (ppm) = 8.38 (bs, -NH-), 8.09 (bs, -NH-), 7.87 (CH Nal), 7.63 (CH Nal), 7.31 (Nal), 7.08 (CH Tyr), 6.78 (CH Tyr), 3.50 (t, -CH$_2$- G5 PAMAM), 3.32 (m, -CH$_2$- G5 PA-MAM), 3.14 (t, -CH$_2$- G5 PAMAM), 2.87 (m, -CH$_2$- G5 PAMAM), 2.68 (m, -CH$_2$- G5 PAMAM), 2.49 (t, -CH$_2$- G5 PAMAM), 2.47 (m, -CH$_2$- G5 PAMAM). The ^{1}H NMR spectra of an unconjugated mixture of LFC131 and G5 PAMAM-AC without a crosslinker was provided for comparison (Fig. 5) in addition to G5 PAMAM. After conjugation with LFC131 peptide, the NMR profile of methylene chemical shifts of an ethylene diamine moiety was changed within the range of 2.45–3.49 ppm [1].

1.4. Mass spectrometry analysis of LFC131 peptide conjugated G5 PAMAM dendrimers

The molecular weights of LFC131 peptide conjugated G5 PAMAM dendrimers (Fig. 6B) and G5 PAMAM dendrimers (Fig. 6A) were 31,021.5 and 27,157.22, respectively. The molecular weight of the unconjugated mixture of LFC131 and G5 PAMAM-Ac without a cross-linker (Fig. 6C) was not different from that of G5 PAMAM-Ac.

2. Materials and Methods

2.1. Materials

PAMAM 5.0 G was purchased from Dendritech, Inc., (Mi, USA). Fluorescein Isothiocyanate was purchased from Bio-World (OH, USA). LFC131 peptide Tyr-Arg-Arg-Nal-Gly (MW 747.82) was synthesized and characterized by Pepmic Co., Ltd. (Suzhou, China).

2.2. NMR spectroscopy

G5 PAMAM (20 mg), partially acetylated G5 PAMAM dendrimers (20 mg), FITC labelled partially acetylated G5 PAMAM dendrimers (20 mg) were weighed in analytical balance and 500 μL of methanol-d4 was added to completely dissolve the sample. The solution was placed in a clean and dry resonance tube. To obtain the spectroscopic data of hydrogen nucleus (^{1}H), BRUKER AVANCE NANOBAY 400 MHz NMR spectrometer was used.

Partially acetylated G5 PAMAM dendrimers (1 mg), LFC131 peptide conjugated PAMAM dendrimers (1 mg) and unconjugated mixture of LFC131 peptide and partially acetylated PAMAM dendrimers (1 mg) were weighed in analytical balance and 500 μL of deuterated water was added to completely dissolve the sample. The solution was placed in a clean and dry resonance tube. The sample was analysed with a Bruker AVANCE III nuclear magnetic resonance spectrometer at 500 MHz.

2.3. Mass spectrometry

Partially acetylated G5 PAMAM dendrimers (1 mg), LFC131 peptide conjugated PAMAM dendrimers (1 mg), and unconjugated mixture of LFC131 peptide and partially acetylated PAMAM dendrimers (1 mg) were dissolved in 1 mL of water and measured by matrix assisted laser desorption ionization-time of flight (MALDI TOF) with Micro flex mass spectrometer. The MALDI spectra were acquired using a matrix solution of sinapic acid (10 mg/mL) in water.

CRediT Author Statement

Chuda Chittasupho: Conceptualization, Methodology, Investigation, Formal analysis, Writing original draft; **Chaiyawat Aonsri:** Investigation, Formal analysis, Visualization; **Witcha Imaram:** Conceptualization, Formal analysis, Writing review & editing, Supervision.

Declaration of Competing Interest

The authors declare that they have no competing financial interests or other relationships or affiliations that could have appeared to influence the work reported in this paper.

Acknowledgments

Supplementary Materials

Supplementary material associated with this article can be found in the online version at doi:10.1016/j.dib.2021.106849.

Reference

[1] C. Chittasupho, S. Anuchapreeda, N. Sarisuta, CXCR4 targeted dendrimer for anti-cancer drug delivery and breast cancer cell migration inhibition, Eur. J. Pharm. Biopharm. 119 (2017) 310–321 https://doi:10.1016/j.ejpb.2017.07.003.