The data presented here are related to the research paper entitled “PCL-b-P(MMA-co-DMAEMA)₂ new triblock copolymer for novel pH-sensitive nanocapsules intended for drug delivery to tumors” by Franco et al. [1]. Characterization data of PCL-diol, macroinitiator Br-PCL-Br, homopolymers (PMMA and PDMAEMA) and copolymers (batch 1 and batch 2) analyzed by FTIR, SEC and NMR, as well as, characterization of PCL-NS formulation by laser diffraction and DLS analysis, initial nanocapsule formulations and 1C-NC and 2C-NC formulations, including hydrodynamic diameter at different pH media, and DMSO cytotoxicity.

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Specifications Table

| Subject Area     | Chemistry, Biology, Pharmacy |
|------------------|------------------------------|
| More specific subject area | Copolymer synthesis and pH-sensitive nanocapsules |
| Type of data     | Tables and figures |
| How data was acquired | FTIR spectrometer (Varian 640 FT-IR, USA), SEC by GPCMax triple detector (Viscotek, Marvel Instruments Ltd, England, UK, columns of Styragel 10^4, 10^5, and 10^6Å), 1H NMR (300 MHz) and 13C NMR (75 MH) by INOVA-300 (Varian, USA), laser diffraction (Malvern Mastersizer® 2000, Malvern Instruments, UK), dynamic light scattering (DLS, Malvern Zetasizer instrument - NanoZS, Malvern Instruments, UK) and cytotoxicity in MCF-7 cells (ATCC®-HTB-22™ Rockville, MD, λ of 570 and 630 nm - SpectraMax M2, Molecular Devices) |
| Data format      | Raw, analyzed |
| Experimental factors | Synthesis and products isolation by filtration and purification by impurities dissolution. Nanocapsules were analyzed as produced, without pre-treatment |
| Experimental features | Chemical characterization and identification of modifications induced by synthesis procedures or by formulation of materials |
| Data source location | Commercial reagent: PCL, MMA and DMAEMA |
| Data accessibility | Data is provided with this article |

Value of data

- Characterization spectra of the materials were compared with data from other works when developing a similar delivery system or copolymer synthesis.
- SEC and NMR data provided information on the efficiency of the copolymer synthesis and were useful for their identification.
- Nanocapsules parameters and its response to different pH media is innovative for scientific community since the copolymer maintains its integrity and expands upon acid pH.
- The bromide end-group of the copolymer permit application as active targeting system after covalent binding with ligands.

1. Data

The data presented in Section 1.1 is the 1H NMR analysis of the homopolymers PMMA and PDMAEMA (Fig. 1). Section 1.2 involves the profiles by laser diffraction of PCL-NS and its parameters (Fig. 2, Table 1). The data presented in Section 1.3 includes the synthesis of the macroinitiator and the characterization by FTIR and SEC analysis of the PCL-diol and Br-PCL-Br (Fig. 3). 1H NMR (Fig. 4) and 13C NMR (Fig. 5). Section 1.4 brings data referent to the copolymers (batch 1 and batch 2) with FTIR, SEC (Fig. 6), 1H NMR (Fig. 7) and 13C NMR (Fig. 8). The data contained in Section 1.5 is related to the characterization of nanocapsules formulations, as size distribution profiles of initial nanocapsule formulations (Fig. 9) and 1C-NC and 2C-NC formulations (Fig. 10), including its parameters (Table 2) and the DLS profile (Fig. 11) and its behavior in different pH (Fig. 12). Section 1.6 presented the DMSO cytotoxicity data (Fig. 13).

1.1. 1H NMR spectra of homopolymers PMMA and PDMAEMA
1.2. Laser diffraction profiles expressed by volume and number of particles

Fig. 1. $^1$H NMR (CDCl$_3$, 300 MHz) spectra of PMMA (A) and PDMAEMA (B).
1.3. Macroinitiator synthesis and characterization

See Figs. 3–5.

Table 1
Laser diffraction analysis of NS formulation.

| Formulation | Diameters (µm) | Span<sub>v</sub> | Diameters (µm) | Span<sub>n</sub> |
|-------------|----------------|-----------------|----------------|-----------------|
|             | D<sub>4,3v</sub> | d<sub>0.1v</sub> | d<sub>0.5v</sub> | d<sub>0.9v</sub> | D<sub>4,3n</sub> | d<sub>0.1n</sub> | d<sub>0.5n</sub> | d<sub>0.9n</sub> |
| PCL-NS      | 0.130        | 0.071           | 0.122          | 0.203           | 1.077         | 0.130          | 0.041           | 0.071           | 0.122           | 1.142         |

Fig. 2. Size distribution profiles by laser diffraction analysis of PCL-nanospheres, expressed by volume (A) and by number (B) of particles.
Fig. 3. Macroinitiator Br-PCL-Br synthesis (A) and characterization of the product by FT-IR (B) and SEC (C).
Fig. 4. Macroinitiator Br-PCL-Br structure (A) and $^1$H NMR (CDCl$_3$, 300 MHz, D) spectra of PCL-diol (B) and Br-PCL-Br (C).
Fig. 5. Macroinitiator Br-PCL-Br structure (A) and $^{13}$C NMR (CDCl$_3$, 300 MHz) spectrum of PCL-diol (B) and Br-PCL-Br (C).
1.4. Copolymers characterization

See Figs. 6–8.

Fig. 6. Characterization of PCL-b-P(DMAEMA-co-MMA)$_2$ products (batches 1 and 2) by FT-IR (A) and SEC (B).
Fig. 7. PCL-b-P(DMAEMA-co-MMA)₂ structure (A) and ¹H NMR (CDCl₃, 300 MHz, D) spectra of PCL-b-P(DMAEMA-co-MMA)₂ batch 1 (B) and batch 2 (C).
Fig. 8. $^{13}$C NMR (CDCl$_3$, 300 MHz) spectra of PCL-$b$-P(DMAEMA-co-MMA)$_2$ batch 1 (A) and batch 2 (B).
1.5. Characterization of nanocapsules formulations

See Figs. 9–12 and Table 2.

Fig. 9. Size distribution profiles by laser diffraction of nanocapsule formulations: A-NC, B-NC and C-NC, expressed by volume of particles (left) and by number of particles (right).
Fig. 10. Size distribution profiles by laser diffraction of nanocapsule formulations: 1C-NC and 2C-NC, expressed by volume of particles (left) and by number of particles (right).
Table 2
Diameters and polydispersity (Span) determined by laser diffraction analysis of formulations 1C-NC e 2C-NC.

| Formulation | Diameters (μm) | Span<sub>v</sub> | Diameters (μm) | Span<sub>n</sub> |
|-------------|----------------|----------------|----------------|----------------|
|             | D<sub>4,3v</sub> | d<sub>0,1v</sub> | d<sub>0,5v</sub> | d<sub>0,9v</sub> | D<sub>4,3n</sub> | d<sub>0,1n</sub> | d<sub>0,5v</sub> | d<sub>0,9v</sub> |
| 1C-NC       | 0.600 ± 0.49   | 0.103 ± 0.00   | 0.120 ± 0.01   | 0.174 ± 0.09   | 0.781 ± 0.64   | 0.125 ± 0.06   | 0.061 ± 0.00   | 0.085 ± 0.00   | 0.131 ± 0.00   | 0.795 ± 0.04   |
| 2C-NC       | 0.794 ± 0.53   | 0.08 ± 0.00    | 0.134 ± 0.02   | 0.318 ± 0.18   | 1.682 ± 0.95   | 0.143 ± 0.03   | 0.053 ± 0.01   | 0.079 ± 0.02   | 0.127 ± 0.00   | 0.973 ± 0.33   |
**Fig. 11.** Size distribution profiles by DLS analysis of nanocapsule formulations: 1C-NC and 2C-NC, expressed by intensity (%).

**Fig. 12.** Hydrodynamic diameters (z-average) by dynamic light scattering of 1C-NC and 2C-NC in different media: ultrapure water, potassium phosphate buffer pH 7.4 and potassium phosphate buffer pH 5.5.
1.6. DMSO cytotoxicity

Fig. 13. DMSO cytotoxicity assessed by MTT assay, after 24 hours of treatment in MCF-7 cells (n=1, quadruplicate). The culture medium was used as control. The symbol (*) represents the statistical differences between the sample and the control (ANOVA, $F = 45.34$, $F_{crit} = 2.57$, $p = 6.00 \times 10^{-11}$ and HSD = 11.22).

Table 3
Nanocapsules initial formulations and formulations.

| Materials and quantities$^a$ | Initial formulation | Formulations |
|------------------------------|---------------------|--------------|
|                              | A-NC    | B-NC    | C-NC    | 1C-NC | 2C-NC |
| PCL-\(b\)(P(MMA-co-DMAEMA)$_2$ | 0.0103 | 0.0103 | 0.0320 | 0.0341 | 0 |
| PCL-\(b\)(P(MMA-co-DMAEMA)$_2$ | 0     | 0     | 0     | 0     | 0.0304 |
| Sorbitan monostearate (g)    | 0.0042 | 0.0042 | 0     | 0     | 0 |
| CCT (oil) (g)                | 0.0163 | 0.0163 | 0.0540 | 0.0574 | 0.0521 |
| Acetone (mL)                 | 25     | 25     | 15     | 15     | 15 |
| Ethanol (mL)                 | 0      | 0      | 0      | 0      | 0 |
| Polysorbate 80 (g)           | 0.0084 | 0.0008 | 0.0520 | 0.0538 | 0.0514 |
| Water (mL)                   | 53     | 53     | 53     | 60     | 60 |

$^a$ Final volume after evaporation = 10 mL.

2. Experimental design, materials and methods

The methodologies to obtain the data exposed here are described in [1] and in cited references. PCL-NS was prepared using PCL (14,000 g mol$^{-1}$, 0.0301 g) solubilized in 30 mL of acetone:ethanol 1:1 (v/v) and injected into an aqueous dispersion (60 mL) of polysorbate 80 (0.0518 g), having its volume reduced to 10 mL.

Copolymers synthesis was performed with Br-PCL-Br macroinitiator (1.75 g, 0.14 mmol), DMAEMA (1 g, 6.36 mmol), MMA [65 mg, 0.65 mmol for batch 1 or 19 mg, 0.19 mmol (1 drop) for batch 2] in 2 mL of anisole were added in a bottle flask and stirred under argon at room temperature for 15 min. Then, a mixture of 0.1 mL of (CuBr (1) (6 mg, 0.04 mmol), PMDETA (140 mg, 0.81 mmol) in 1 mL of...
anisole) was added by a syringe at once. After 2 min, a solution of Tin (II) 2-ethylhexanoate (32 mg, 0.08 mmol) in anisole (2 mL) was drop-wised using a syringe. Then, the temperature was raised to 90 °C and the reaction was maintained under stirring for 24 hours. After cooling to room temperature, THF (5 mL) was added and the copolymer was precipitated in cold cyclohexane (50 mL). To remove the catalyst and non-reacted monomers, the crude solid was dissolved in 2-propanol (5 mL) and precipitated in cold cyclohexane (50 mL). The precipitate was isolated by filtration and dried under vacuum (Edwards Weg ® C56 0698, Brazil).

Initial nanocapsule formulations and 1C-NC and 2C-NC formulations are described in the Table 3.

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Reference

[1] C. Franco, M.B. Antonow, A. Beckenkamp, A. Buffon, T. Ceolin, M.L. Tebaldi, G.P. Silveira, S.S. Guterres and A.R. Pohlmann, PCL-b-P(MMA-co-DMAEMA)2 new triblock copolymer for novel pH-sensitive nanocapsules intended for drug delivery to tumors React. Funct. Polym.119, (2017), 116–124, http://dx.doi.org/10.1016/j.reactfunctpolym.2017.08.010.