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Fabricating core (Au)-shell (different stimuli-responsive polymers) nanoparticles via inverse emulsion polymerization: Comparing DOX release behavior in dark room and under NIR lighting

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ABSTRACT

Different core-shell nanoparticles with Au as core and stimuli-responsive polymers such as poly(acrylic acid) (PAA), poly(methacrylic acid) (PMAA), poly(*N*-isopropylacrylamide) (PNIPAAm), poly(*N,N'*-methylenebis(acrylamide)) (PMBA), poly(2-hydroxyethyl methacrylate) (PHEMA) and poly((2-dimethylamino)ethyl methacrylate) (PDMAEMA) as shells were fabricated via inverse emulsion polymerization. Dynamic light scattering (DLS) was used to investigate particles sizes and particle size distributions and transmission electron microscopy (TEM) was applied to observe the core-shell structure of Au-polymer nanoparticles. Also, surface charge of all samples was studied by measurement of zeta potentials. Synthesized core-shell nanoparticles were utilized as nanocarriers of DOX as anti-cancer drug and drug release behaviors were investigated in dark room and under irradiation of near-infrared (NIR) light. Results showed that all core-shell samples have particle sizes less than 100 nm with narrow particle size distributions. Moreover, amount of drug loading decreased by increasing zeta potential. In dark room, lower pH resulted in higher cumulative drug release due to better solubility of DOX in acidic media. Also, NIR lighting on DOX-loaded samples led to increasing cumulative drug release significantly. However, DOX-loaded Au-PAA and Au-PMAA showed higher drug release at pH = 7.4 compared to 5.3 under NIR lighting.

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1. Introduction

Gold nanoparticles (AuNPs) are used in many biomedical applications such as sensing [1], cellular imaging [2], drug delivery [3], and cancer therapy [4] because of their strong surface Plasmon resonance (SPR) absorption band in visible range [5]. Position of SPR depends on size and shape of nanoparticles (NPs) [6], their environment [7], aggregative state [8] and interparticle distance [9]. The optimal wavelength to use for best tissue penetration is 800 nm (near infrared, NIR) where light has its maximum depth of penetration in tissue [10,11]. SPR in the NIR region is useful for biological applications because of transparency peak of skin, tissues, hemoglobin, and blood in that spectral region [12]. Beside this, core-shell nanostructures have been developed in recent years due to their potential applications in various fields of biomedical and nanomedicine like drug delivery [13,14], bioimaging [15], cancer treatment [16], reducing cytotoxicity [17], etc. They can be fabricated with different shapes and sizes of cores as well as shell widths with different morphologies [18–20].

AuNPs absorb the incident energy and convert it into heat [21] where temperature increases high enough to cause immediate cancerous cells death by disrupting the cell membrane [22]. For example, Hirsch et al. [23] demonstrated photothermal destruction of human breast carcinoma cells as well as solid tumors using NIR-absorbing silica-gold core-shell particles. In some cases, surface modification of AuNPs has been reported to be destructive respecting colloidal stability of nanoparticles [21]. Thus, one-pot fabrication of polymer-AuNPs core-shell structures seems to be necessary to prevent aggregation of AuNPs. Besides, polymers used in drug delivery systems are mostly thermoresponsive and pH-sensitive ones where polymers are soluble in water in variety of temperatures and pH values [24–27]. Therefore, aqueous systems such as emulsion polymerization cannot be used for encapsulation of AuNPs with water soluble polymers to fabricate core-shell structures while AuNPs are aqueous colloidal systems. Conclusively, the continuous medium should be chosen organic systems such as inverse emulsion systems [28].

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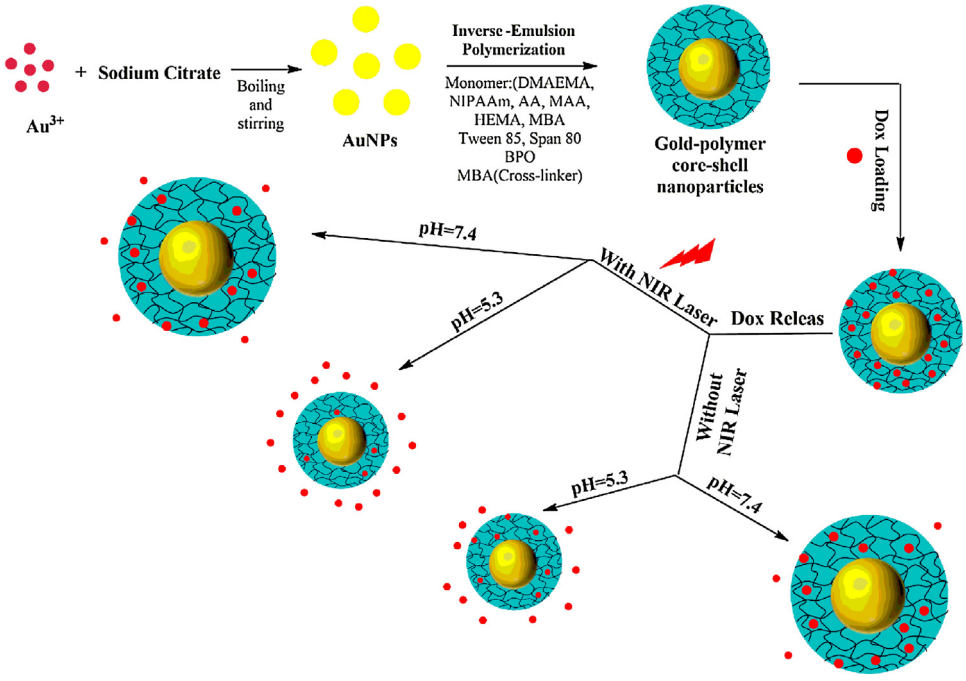

Scheme 1. Fabrication process of gold–polymer core–shell nanoparticles used as DOX nanocarriers.

Table 1
The amounts of initiator and monomer for the synthesis of gold-polymer core-shell nanoparticles by inverse emulsion polymerization.

Sample	Amount of initiator	Amount of monomer
Au-PAA	0.0605 g, 0.0003 mol	1.37 mL, 0.02 mol
Au-PMAA	0.058 g, 0.0003 mol	1.69 mL, 0.02 mol
Au-PNIPAAm	0.058 g, 0.0003 mol	2.26 g, 0.02 mol
Au-PMBA	0.0605 g, 0.0003 mol	3.08 g, 0.02 mol
Au-PHEMA	0.0605 g, 0.0003 mol	2.43 mL, 0.02 mol
Au-PDMAEMA	0.061 g, 0.0003 mol	3.03 mL, 0.02 mol

The aim of this study is synthesis and development of novel core-shell nanoparticles as anti-cancer drug carriers. To this end, after synthesis of aqueous colloidal AuNPs, they were used as seeds in inverse emulsion polymerization of poly(2-dimethylamino)ethyl methacrylate) (PDMAEMA), poly(N-isopropylacrylamide) (PNIPAAm), polyacrylic acid (PAA), poly(methacrylic acid) (PMAA), poly(2-hydroxyethyl methacrylate) (PHEMA) and poly(N,N'-methylenebis(acrylamide)) (PMBA) to obtain Au-PDMAEMA, Au-PNIPAAm, Au-PAA, Au-PMAA, Au-PHEMA and Au-PMBA core-shell nanoparticles respectively. Synthesized core-shell nanoparticles were used as nanocarriers of doxorubicin (DOX) as an anti-cancer drug. To investigate the effect of NIR light on drug release behavior of nanoparticles, DOX-loaded core-shell nanoparticles were subjected to drug release in different media with different pH values in dark room condition or under IR irradiation.

2. Experimental section

2.1. Synthesis of gold-smart polymer core–shell nanoparticles

Firstly, AuNPs were synthesized via Turkevich approach as described in section S2 (see supporting information). To fabricate core-shell nanoparticles, inverse emulsion polymerization of different monomers was conducted in cyclohexane as continuous media and 250-mL three-necked round-bottomed flask at 65 °C. In all batches, monomer/cross-linker molar ratio was 80:20. A certain amount of monomer (0.02 mol) and methylenebis(acrylamide) (MBA) as cross-linker (0.771 g, 0.005 mol) were added into a mixture of cyclohexane/colloidal AuNPs (w/w, 100/25, 128 mL cyclohexane and 25 mL colloidal AuNPs), benzoyl peroxide (BPO) as initiator (0.061 g, 0.0003 mol) and surfactants (3.03 mL Span80, 1.95 mL Tween80). Reactions were performed for 24 h under a nitrogen atmosphere. After several times of centrifugation and

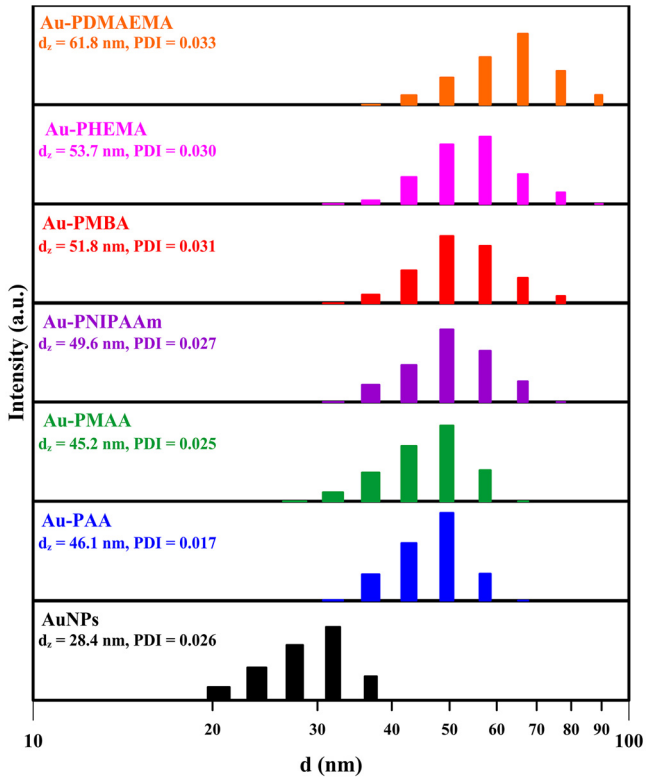

Fig. 1. DLS results of colloidal AuNPs, Au-PAA, Au-PMAA, Au-PNIPAAm, Au-PMBA, Au-PHEMA and Au-PDMAEMA nanoparticles.

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