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Preparation and characterisation of triangular pyramid-shaped puerarin and aspirin microparticles with nanostructures

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A new class of triangular pyramid-shaped microparticles of puerarin and aspirin were prepared on copper substrate by using oil-in-oil microemulsion method which is simple and economical. The morphology and microstructure of the pyramid-shaped microparticles were characterised by field emission scanning electron microscopy. The results show that pyramid-shaped puerarin and aspirin, with coral reef-like morphology, resulted from the self-assemble of nanoparticles or nanorods. A possible formation mechanism was proposed and discussed.

Keywords: triangular pyramid-shaped puerarin and aspirin; synthesis; nanosphere; nanorod

1. Introduction

In the past few decades, much attention has been attracted by the special properties of nanomaterials, which are controlled by their size, shape and surface structure. It has been found that nanoparticles with different shapes have different properties and applications due to their different surface-size ratios. Therefore, nanoparticles with various shapes and morphologies have been synthesised for various potential applications [1–3], especially in biological and pharmaceutical applications. Likewise, nanodrugs with different shapes should also have different bioactivities. To date, most researchers have focussed on the synthesis of nanomaterials by using metals or inorganic compounds as crude materials, such as Au nanothreads, nanorods and mesoflowers, carbon nanotubes, silver nanoprisms and nanocubes, MgO fishbones, SnO_2 nanoribbons [4–8] and so on. However, it is rarely reported that using a small molecular organic compound as the precursor material synthesise can the nanoparticles with different shapes.

Puerarin, a bioactive component from a plant named *Pueraria thomsonii* Benth, has significant effect in reducing myocardial oxygen consumption and improving micro-circulation [9]. But puerarin has a very low oral bioavailability (BA), about 4%, owing to its poor water solubility. In our previous work [10], puerarin with a higher BA, about 38%, was obtained by means of the oil-in-oil (O/O) type of nanoemulsion. In this work, a class

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Table 1. Preparing conditions of the triangular pyramid-shaped nanodrugs.

Sample	Drug	Substrate	Fabrication approach	Thermal treatment condition
1	Puerarin	Copper	Stir	180°C for 8°h
2	Puerarin	Copper	Stir	100°C for 5°h, then 180°C for 8°h
3	Puerarin	Platinum	Stir	100°C for 5°h, then 180°C for 8°h
4	Aspirin	Copper	Stir	100°C for 5°h, then 180°C for 8°h
5	Aspirin	Copper	Sonicate	100°C for 5°h, then 180°C for 8°h

of triangular pyramid-shaped microparticles of puerarin and aspirin was synthesised by using the O/O nanoemulsion method. A possible formation mechanism of the microparticles is proposed and discussed.

2. Experimental procedure

The preparation processes of the O/O blank microemulsion are the same as those found in our previous report [10]. In brief, 5 ml of soybean oil, 2.6 ml of oleic acid and 2.4 g of soy lecithin were mixed and magnetically stirred for 4 h, so as to obtain the O/O blank microemulsion. Then, 0.1 g of puerarin or 0.35 g of aspirin was dissolved in 2 ml of ethanol, and dropped in 5 ml of the above blank microemulsion under stirring. The obtained mixture was then stirred at 50°C or ultrasonated for 2 h. For the preparation of the pyramid-shaped nanodrugs, a drop of the above drug-loaded microemulsion was coated on the surface of a glossy copper substrate, and then dried in vacuum in order to remove ethanol and the other reagents. Finally, triangular and pyramid-shaped puerarin and aspirin were obtained. The morphology and microstructure of puerarin and aspirin were characterised by field emission scanning electron microscopy (FE-SEM, Sirion 200, operated at 5 kV). Detailed preparation conditions are summarised in Table 1.

3. Results and discussion

Figure 1(a) and (b) shows the SEM images of the final puerarin prepared on copper substrate. It is clear that large amounts of regular triangular pyramid-shaped products were obtained. Figure 1(b) presents an integrated triangular puerarin. The bottom side of the triangle is about 10 μm, and the height of it is about 4 μm. In addition, only a few nanoparticles were observed beside it, which suggests that almost all of the puerarin was assembled together, and formed the triangular pyramid-shaped products. The irregular side and the coarse surface of it imply that the products were resulted from the self-assemble of the puerarin. Figure 1(c) presents the surface of the puerarin micro/nanostructures. Many nanoparticles, with about 200 nm in diameter, were observed, and resulted in the formation of the coarse and porous nanostructures of the surface, as shown in Figure 1(d). The results further suggest the aggregation and the self-assembly of the puerarin in the process of the thermal treatment. In addition, no products were obtained after heating the blank nanoemulsion, as shown in Figure 1(e), which confirms

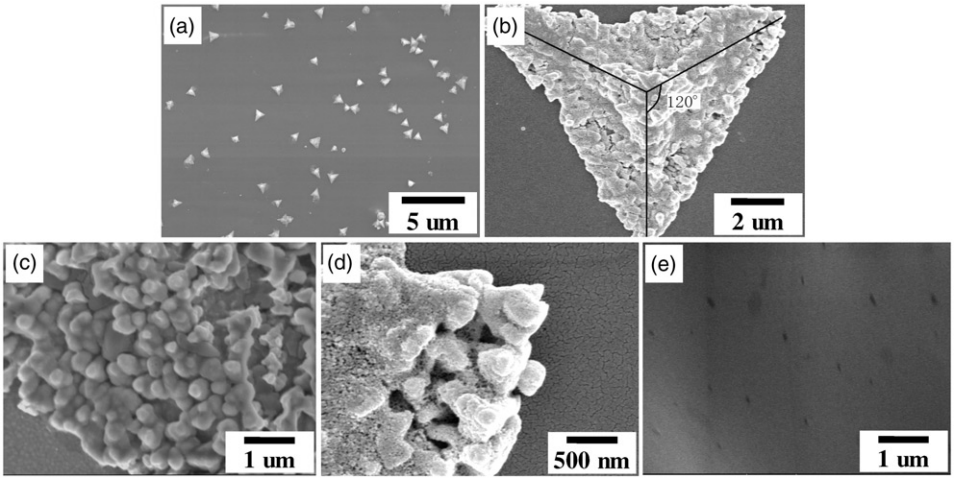

Figure 1. FE-SEM images (a)–(d) of triangular pyramid-shaped puerarin prepared on copper substrate, and SEM image (e) of the product after heating of the blank nanoemulsion.

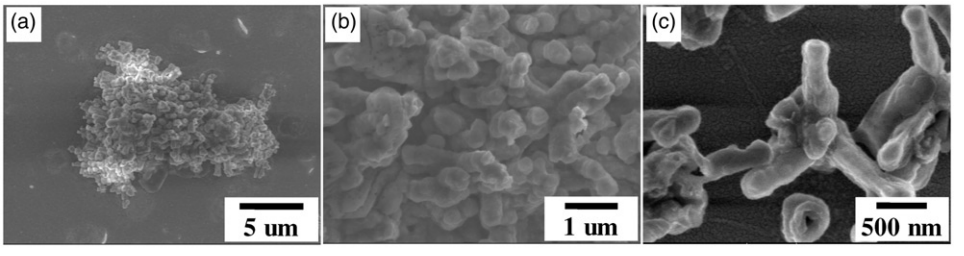

Figure 2. FE-SEM images of products after drying the puerarin-loaded microemulsion at 100°C for 5 h, and then at 180°C for 8 h.

that the obtained micro/nanostructures were the self-assembled puerarins and also proved that the volatilisation of the nanoemulsion was complete.

In order to investigate the formation mechanism of the triangular pyramid-shaped products, the effect of the thermal treatment conditions was studied. Figure 2 presents the SEM images of the final products after drying the puerarin-loaded microemulsion at 100°C for 5 h, and then 180°C for 8 h (sample 2). Compared with the sample 1, although no regular triangular pyramid-shaped puerarin was obtained, the obtained products still have similar morphology. From Figure 2(b) and (c), it is clear that the obtained products resulted from the agglomerate of the puerarin nanorods. The diameter of the nanorods was about 200–300 nm, and the length was 600–1000 nm. The results suggest that the thermal treatment conditions had a great influence on the microstructure of the products. Before thermal treatment, puerarin was loaded in an O/O type of nanoemulsion. In the process of the thermal treatment, the congregate and the volatilisation of the nanoemulsion take place simultaneously. The low temperature was propitious to the

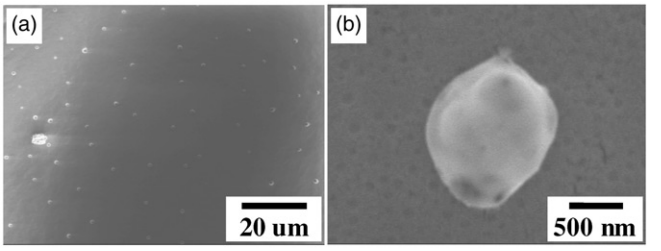

Figure 3. FE-SEM images of puerarin nanospheres prepared on platinum substrate.

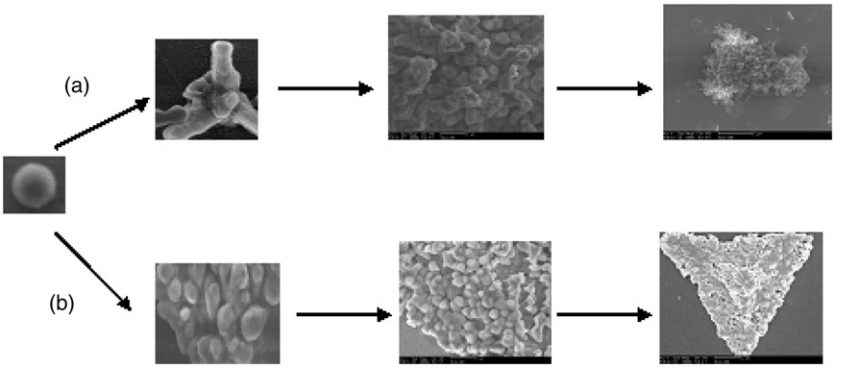

Figure 4. Growth mechanism of the triangular pyramid-shaped puerarin: (a) the self-assembly of the puerarin nanorods prepared at low temperature and (b) the self-assembly of the puerarin nanodots obtained at high temperature.

congregate of the nanoemulsion, and the high temperature resulted in fast volatilisation. When the nanoemulsion is directly heated to 180°C, the rapid volatilisation of it will result in the formation of the puerarin nanoparticles. The obtained nanoparticles were assembled together and formed the regular triangular pyramid-shaped products, with many nanoparticles on its surface. However, at a lower thermal treatment temperature of 100°C, the volatilisation rate was slower than that of the 180°C treatment. As a result, the nanoemulsion, loaded with puerarin, was congregated and formed bead-like intermediate products. With further volatilisation of the nanoemulsion at 180°C, the loaded puerarin in it was merged together, and resulted in the formation of the puerarin nanorods, as shown in Figure 2. The diameter of puerarin nanorods was similar to the puerarin nanoparticles obtained at high temperature. The results further imply the puerarin nanorods resulted from the congregate of the nanodrugs, which were loaded in the nanoemulsion. Furthermore, the role of the copper substrate was also investigated. When the copper substrate was replaced with platinum, only large amounts of puerarin nanospheres with 200–600 nm diameters were obtained, as shown in Figure 3 (sample 3). The results suggest that the copper substrate plays a very important role in the formation of the puerarin micro/nanostructures. The reason for this is not clear, and more research is in progress. Based on the above discussion, the growth mechanism of the puerarin micro/nanostructures on copper substrate is schematically presented in Figure 4.

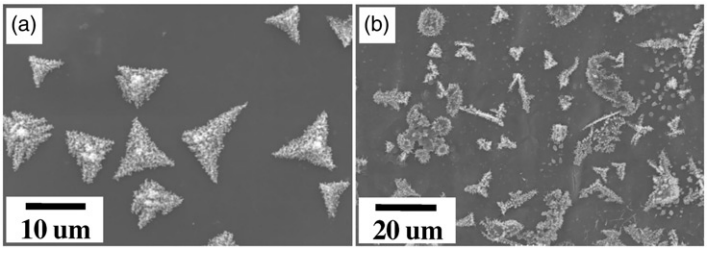

Figure 5. FE-SEM images of the aspirin micro/nanostructures prepared by using stirring (a) and sonicating (b) methods.

In addition, aspirin was also loaded in the O/O-type nanoemulsion. After a similar loading method and thermal-treatment process, the obtained products are shown in Figure 5(a) (sample 4). It is clear that similar aspirin micro/nanostructures were prepared on copper substrate. However, when the sonicate method was employed to load the aspirin in nanoemulsion, only a small quantity of triangular pyramid-shaped products were obtained, as shown in Figure 5(b) (sample 5). The result suggests that stirring is a more effective method to prepare the drug-loaded nanoemulsion, as well as the triangular pyramid-shaped micro/nanostructured drugs.

4. Conclusions

Triangular pyramid-shaped puerarin and aspirin micro/nanostructures were prepared on copper substrate by heating the loaded O/O-type nanoemulsion. The prepared micro/nanostructured drugs were resulted from the self-assembly of the enwrapped drugs after removing the nanoemulsion. A possible growth mechanism was also proposed and discussed. The results provide a new route for the preparation of nanodrugs with peculiar microstructures.

Acknowledgements

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