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ANALYSIS OF THE PROPERTIES OF POROUS CHITOSAN/SILICA HYBRID MICROSPHERES

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ABSTRACT

In the past few years, the synthesis of various inorganic/organic hybrid materials has gained much attention. Chitosan, being a base polysaccharide in nature and non-toxic, biocompatible, biodegradable, insoluble either in water or in organic solvents, has great potential applications in adsorption and isolation of protein, enzyme immobilization and controlled drug release in the form of microspheres and capsules. The hybrid materials exhibit better mechanical performances than pure chitosan, hence various hybrid microspheres, with different structure and catalytic properties have been synthesized. Here, the properties of the porous chitosan/silica hybrid microspheres are analysed, which are important for their successful biomedical application.

Keywords: Chitosan, Chitosan/Silica hybrid microspheres, Mechanical properties, Microfluidic methods,Production technique

I. INTRODUCTION

Recently, microfluidic methods have been developed for the synthesis of various organic and inorganic materials, highly mono-dispersed droplets with spherical polymer microparticles[1], various inorganic / organic hybrid microspheres e.g. Zeolite A/chitosan[2], chitosan/silica hybrid microspheres[3] etc. These microspheres are small spherical particles with diameters in the micrometer range (typically 1µm - 100µm) and can be manufactured from various natural and synthetic materials. They have a wide range of applications e.g. in creating porous ceramics used for filters, in drug delivery, in thermal insulation, as retro-reflective coating to increase night visibility of road signs, as thickening agent, adsorption of metal ions etc.

In the production of these microspheres, chitosan plays an important role due to its specific properties. It is the only base polysaccharide in nature. It is insoluble in a weak base, water or organic solvents, while soluble in dilute aqueous acidic solution. Moreover, it is non-toxic, bio-compatible, bio-degradable and of low cost.

II. PRODUCTION TECHNIQUE

To prepare mono-dispersed chitosan microspheres with small size and controlled structures, types of capillary-embedded T- junction microfluidic device [4,5] were developed. This type of device could form larger shear force than other structures [1]. A T-junction generation and formation of droplets is shown in Fig 1(a) &(b) [4]. The chitosan droplets formed in the tube were consecutively collected in a solidification bath. Chitosan reacted with glutaraldehyde diffused into the droplet and water was simultaneously extracted out of the droplets by the

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n-octanol in the solidification bath when the droplets were shaken in it [1]. Thus, the droplets gradually solidified from the surface to the inside with the residence time increasing.

Fig. 1 (a) T-junction generation (b) A photograph showingformation of the droplets in a fabricated device {See ref. [4]}.

The structure of microsphere could be controlled by regulating the solidification time. The chemical crosslinking reaction between glutaraldehyde and chitosan and solvent (water)extraction method can be used to produce microspheres of decreased size with increasing solidification time [1]. For the solidification time of 10 min, the microspheres are porous both on the surface and on inside. The pores are mainly formed due to extraction of water from the microspheres to the solidification bath. As the solidification time was increased to 20 min., microspheres are formed with a solid shell and a porous core [1]. This is mainly due to the diffusion ofwater from microspheres into the solidification bath and the diffusion of glutaraldehyde molecules into the inside of the microspheres and crosslinking with the chitosan molecules. For solidification time of about 35 min., glutaraldehyde molecules diffused into the core of the microspheres and the crosslinking reactions occurred everywhere in the microspheres. Thus, a solid structure was formed [1].

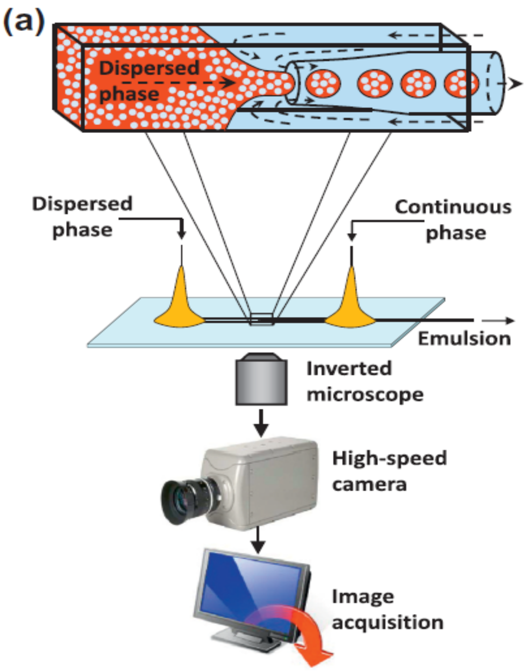

Fig. 2 (a): A schematic diagram of experimental setup with a magnified view of the emulsion formation in the collection capillary of the microfluidic device [5].

III. FORMATION OF POROUS CHITOSAN/SILICA HYBRID MICROSPHERES

To prepare porous chitosan/silica hybrid microspheres [3], 0.5 - 2.0 wt.%tetraethoxysilane (TEOS) and 2.0 wt.% chitosan were added into the aqueous solution with 2.0 wt.% acetic acid and then stirred for 12h at room temperature to obtain a clear chitosan-silica solution, which served as the dispersed phase. This was injected into the micro-channel and broken up into microspheres by the shear force of the continuous flow{Fig. 2(a)} [5]. The microspheres were submerged in the n-octane for 24h to ensure the gelation of silica and then freeze-dried.The Scanning Electron Microscope (SEM) images [3] of porous chitosan microspheres and chitosan/silica hybrid microspheresare shown in Fig. 3(a) & (b) for comparison.

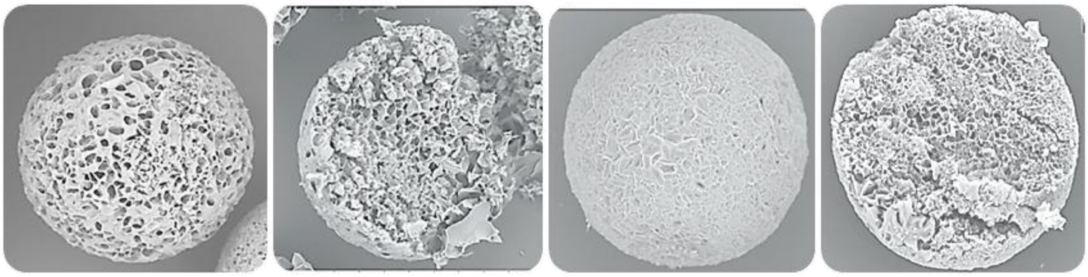

Fig. 3(a): SEM Images of porous chitosan microspheres [3]

Fig. 3(b): SEM Images of chitosan / silica hybrid microspheres [3]

The hybrid microspheres were prepared with different pre-solidification time. The porosity on the surface becomes smaller with the increase of pre-solidification time and is spongy in the interior. On further increasing the time, the specific surface area and the pores in the interior gradually become smaller.The difference in the formed structure of the microspheres leads to the slight variations in their properties.

IV. MECHANICAL PROPERTIES

The mechanical properties of the microspheres depend upon their formed structure. The mechanical intensity and adsorption capacity of hybrid microspheres with different solidification time was studied[3] and 3h was found to be the optimized pre-solidification time for the porous hybrid microspheres [3].The adsorption kinetic curvesof porous chitosan and chitosan/silicahybrid microspheres [3] are shown in Fig. 4(a). The adsorption rate of chitosan/silicahybrid microspheres was found to be two times faster than porous chitosan microspheres [3]. Also, chitosan/silicahybrid microspheres were found to have 25% more adsorption capacity than porous chitosan microspheres [3]. Theyshow better adsorption of heavy metal ions.

As the size and the structure of the produced microspheres could be controlled by regulating the solidification time, the adsorption properties of hybrid microspheres formed with different solidification time were also studied. The adsorption kinetic curvesof hybrid microspheres with differentsolidification time [3] are shown in Fig. 4(b). It can be seen that when the solidification time is of 3h,the adsorption capacity of the hybrid microspheres is maximum. Thus, 3h was found to be the optimized pre-solidification time for the porous hybrid microspheres [3].

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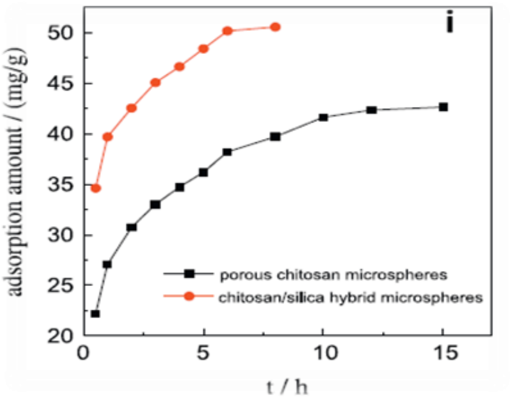

Fig. 4(a):The adsorption kinetic curves of porous chitosan and chitosan/silica hybrid microspheres [3]

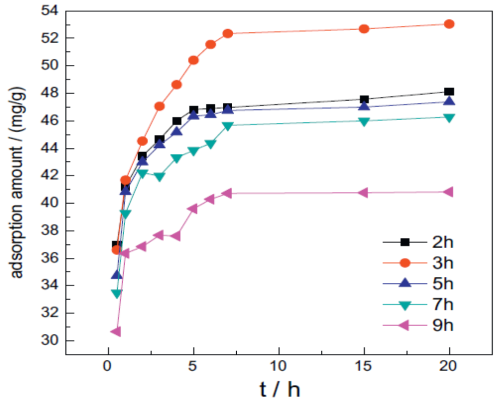

Fig. 4(b): The adsorption kinetic curves of hybrid microspheres with different solidification time [3]

V. RESULT AND DISCUSSION

The porous chitosan/silica hybrid microspheres [3] have the following advantages over porous chitosan microspheres:

- They have better mechanical property
- They have advantage in adsorption of heavy metal ions
- Their adsorption rate is two times faster than porous chitosan microspheres
- Their adsorption capacity is increased by 25%

VI. CONCLUSION

In comparison to the porous chitosan microspheres, the porous chitosan/silica hybrid microspheres [3] are better as they easily form spongy spheres and have large specific surface area which is helpful in the adsorption of heavy metal ions. They also exhibit higher mechanical intensity, faster adsorption rate and larger adsorption capacity.

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