Preparation of core TiO₂/ shell Poly(styrene-co-vinylacetate) nanoparticle using miniemulsion polymerization

Hyeon Jung Yoo and Soo min Park

Department of Organic Material Science, Pusan National University san 30 Jangjeon-dong Geumjeong-gu, Busan, 609-735, Korea E-mail: soominpark@pusan.ac.kr

1. INTRODUCTION

Inorganic core/organic shell hybrid composite particles have received considerable attention in a wide range of industrial fields, such as diagnosis, toner, and paint applications because of their superior functionality and dispersion stability[1,2]. Especially, Titanium dioxide has been widely applied in dispersed fields such as ink, cosmetic, catalysis, photocatalysis and antibacterial actions. Moreover, inorganic core/organic shell hybrid composite particles have better thermal, and optical properties and extend their range of industrial applications [3,4]. The encapsulation of inorganic particles in a polymer shell provides enhanced various aspects of their mechanical properties such as their shape, and chemical resistance [5-8] with the enhancement of their stability being of particular significance.

In present study the preparation and characterization of core TiO₂/shell poly(styrene-co-vinylacetate) nanoparticles were investigated.

We synthesized styrene and vinylacetate polymer hybrid composite particles containg titanium dioxide $(TiO_2/P(St-co-VA))$ by miniemulsion polymerization. Our aims are to verify the effect of different contents of initiator and the morphology of the nanoparticles formed styrene and vinylacetate miniemulsion polymerization were investigated.

Also, conversion ratio of miniemulsion polymerization in producing hybrid composite particles was estimated and we find adequate amount of initiatior by considering the surface characteristics of the composite particles. Besides, experiments were conducted to characterize the thermal stability of TiO₂ according to different component rates of shell material and the quantity of initiator.

2. EXPERIMENTAL

Materials

The liquids used in the tests were distilled water. Titanium dioxide as a core material, Styrene monomer and Vinylacetate as a shell material, AIBN as a initiatior and Sodium Lauryl Sulfate as a surfactant were used.

Furthermore Tetra Hydro Furan(THF) were used to remove shell material from TiO_2 core-shell capsule. The separated polymer was used for further analysis such as GPC. NaOH is used to hydrolysis the nanoparticle.

Preparation of nanoparticle

Shell materials were prepared using miniemulsion polymerization. To prepare water phase, 3g of TiO_2 , SLS were dissolved in distilled water using the sonicator. Next, styrene was mixed with vinylacetate to prepare the oil phase. The oil phase was added into water phase by stirring. The stirring speed was fixed at 3000rpm. And then, they were washed three times with distilled water to remove residual initiation.

The resultant nanoparticles were added in distilled solution and the system was then sonificated and magnetic stirred for 30min.

TABLE 1. Reaction parameters of the polymerization						
	matorial(a)	condition				

	material(g)			condition	
No	core	wall	initiator	Temp	stirring rate
			AIBN	Temp	ourning rate
	TiO ₂	St	(x10 ⁻²)	(°C)	(X1000rpm)
S1	3	9	1.25	70	3
S2	3	9	2.50	70	3
S3	3	9	5.00	80	3
S4	3	9	10.00	80	3

3. RESULTS AND DISCUSSION

Conversion ratio was sharply increased for 60min and almost all of the reaction was finished during 90min (Fig.1). So we set the reaction time as 2hour.

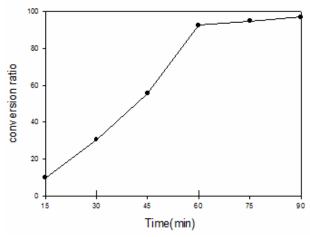
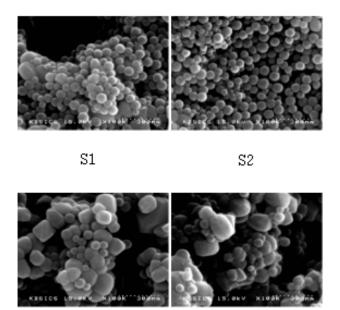


FIG1. Conversion ratio of nanocapsule

Fig.2 shows the SEM image of the nanocapsules formed in the styrene miniemulsion polymerization using different amount of initiator, AIBN. These images revealed that S2 particle had a regular morphology and smaller particle size.

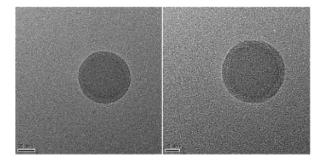


S3

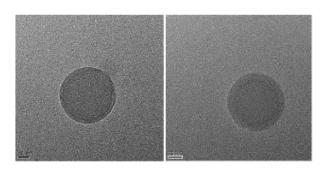
FIG. 2. SEM images of particles

S4

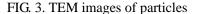
Fig.3 shows the TEM image of the nanocapsules with the same condition like Fig.1. Although S1, S3, S4 hadn't regular shape in Fig1, they showed core/shell structure in a particles.



S2



S3 S4



S1

In the FT-IR of the resultant particles, the characteristic peaks for polystyrene were observed at 1475 and 1600cm-1 belonging to the C=C bands of the phenyl ring, so we could confirm the shell formation.

4. REFERENCES

- [1] Y. Haga, S. Inoue, T. Sato, R. Yosomiya, Angew. Makromol. Chem.139 (1986) 49.
- [2] C.H.M. Caris, R.P.M. Kuijpers, A.M. Heck, A.C. German, Makromol.
- [3] M. Hasegawa, K. Arai, S. Saito, J. Polym. Sci., Part A: Polym.Chem. 25 (1987) 3231..
- [4] C.H.M. Caris, A.M. Herk, P.M. Louissa, A.L. German, Br. Polym.J. 21 (1989) 133.
- [5] S.N. Goyanes, J.D. Marconi, P.G. Konig, C.L. Matteo, G.H. Rubiolo, A.J. Marzocca, Polymer 42 (2001) 5267.
- [6] H. Kawaguchi, Prog. Polym. Sci. 25 (2001) 1171.
- [7] Y.N. Chan, G.S.W. Craig, R.R. Schrock, R.E. Cohen, Chem. Mater.4 (1992) 885.
- [8] P.K. Gupta, T.C. Hung, F.C. Lam, D.G. Perrier, Int. J. Pharm. 43 (1998) 167.