

Concentrated Emulsion Polymerization of Methyl Methacrylate/Butyl Acrylate Initiated by a Redox System

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Received 22 January 2004; accepted 6 October 2004

DOI 10.1002/app.21656

Published Online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Stable concentrated emulsions of methyl methacrylate/butyl acrylate were prepared with sodium dodecyl sulfate and cetyl alcohol as the compound surfactant and poly(vinyl alcohol) as the major reinforcer of the liquid film. With a redox system based on benzoyl peroxide/*N,N'*-dimethyl phenylamine introduced into the concentrated emulsions, polymer particles with different shapes and sizes were obtained by initiation of the polymerization at low temperatures. We investigated the kinetic behaviors of concentrated emulsion polymerization and drew linear regression diagrams of its time–conversion curves in a constant rate phase (conversions ranged from 20 to 70%), and the experimental results show that the variation of the concentrations of the compound surfactant and initiator, the cate-

gories of reinforcers of the liquid film, the temperatures, and so on were responsible for the polymerization stability and the polymerization rate. Finally, the kinetics equation and activation energy of the initiator were obtained. The particle size and distribution of particle diameters of latex particles were determined by photon correlation spectroscopy. The determination results reveal that concentrations of the compound surfactant, polymerization temperatures, and so on affected the shape and size of the polymer particles greatly. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 97: 1695–1701, 2005

Key words: emulsion polymerization; kinetics (polym.); stabilization

INTRODUCTION

A concentrated emulsion has a paste-like appearance and behavior. It has a large volume fraction of the dispersed phase (ϕ), larger than 0.74 and as large as 0.99.^{1–8} The dispersed phase is in the form of spherical or polyhedral cells separated by a network of thin films of continuous phase. Concentrated emulsions have three important characteristics: (1) a reduced mobility of monomer between the cells, because of the presence of a reinforced surfactant layer, can generate an earlier gel effect, which leads to a delay in the bimolecular termination reaction and, hence, to a higher molecular weight. (2) The size of particle can be controlled easily by the selection of a suitable kind and concentration of the surfactant. (3) A polymer containing a lower amount of water, which is easily formed from powdery resins, can be obtained through concentrated emulsion polymerization.

In the early 1980s, Ruckenstein and coworkers did plenty of meaningful studies in this field, and their investigations concentrated mostly on minority mono-

mer systems, such as styrene and styrene/acrylamide.^{9–17} The majority of polymerization reactions based on these systems were carried out with thermolysis initiators, such as azo compounds. Ruckenstein and Li¹⁸ used a redox system containing cumene hydroperoxide and ferrous sulfate/sodium metabisulfite to initiate the copolymerization of acrylonitrile/vinylidene in concentrated emulsions. The heat generated by the polymerization reaction increased rapidly with the temperature, and it accelerated the polymerization, but this led to instability in the concentrated emulsion.

To solve the heat conduction problem of polymerization systems by increasing the stability of concentrated emulsion polymerization, reducing the loss of monomers, and realizing a larger scale production, concentrated emulsion polymerization initiated by redox initiators has been studied at lower temperatures in recent years.¹⁹ In this study, first, a redox system based on benzoyl peroxide (BPO)/*N,N'*-dimethyl phenylamine (DMA) was used to initiate methyl methacrylate (MMA)/butyl acrylate (BA) polymerization in concentrated emulsions at lower temperatures. The polymerization kinetics of MMA/BA in concentrated emulsions is discussed in detail; their effects on the polymerization stability and polymerization rate were studied. A polymerization kinetic equation was obtained. Morphology and size of the latex particles

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Contract grant sponsor: National Natural Science Foundation of China; contract grant number: 20374019.

were observed by photon correlation spectroscopy (PCS); the effects of concentration of the compound surfactant and polymerization temperature on the morphology, size, and size distribution of the latex particles were investigated.

EXPERIMENTAL

Materials

MMA and BA (industrial products, Dong Fang Chemical Factory of Beijing, China) were distilled under reduced pressure to remove the inhibitor and were then stored in a refrigerator until they were used. Sodium dodecyl sulfate (SDS), hexadecanol (HD) and poly(vinyl alcohol) (PVA) were purchased from Japan (Shanghai Supply Depot, Shanghai, China) and were used directly. 1,4-Hydroquinone, hydroxypropyl cellulose (HPC), and poly(*N*-vinylpyrrolidone) (PVP; chemical grade, Shanghai Qunli Chemical Factory, Shanghai, China) were purchased and were used directly. BPO and DMA (chemical grade, The Factory of Hubei University, Wuhan, China) were recrystallized from methanol and dried at room temperature *in vacuo*. The water was deionized before use.

Concentrated emulsion preparation and polymerization

A small amount of an aqueous solution containing SDS and DA was placed in a single-necked flask (250-mL capacity) equipped with a mechanical stirrer. The compound monomers of MMA/BA containing the redox initiator were added dropwise to the upper aqueous solution, with the stirring rate controlled and a dropwise addition rate used to avoid phase separation. The whole preparation process of the concentrated emulsion lasted for 10–15 min at room temperature. The prepared gel-like emulsions were transferred to preweighed centrifuge tubes with capacities of 15 mL, which were sealed with rubber septa. Mild centrifugation (1300 rpm, <1 min) was used to pack the concentrated emulsions into the tubes when necessary. A small quantity of nitrogen was imported into the tubes with an upward exhausting method. Polymerizations were conducted in a temperature-controlled oven or water bath (the general reaction temperature was 30°C). Table I gives the typical recipes used in the preparation of the concentrated emulsion. The volume fraction of dispersed phase (ϕ) was 83.3%.

Measurement and characterization

Concentrated emulsion polymerization stability

The bulk phase separated was moved from the top of the concentrated emulsions prepared in the tubes and was then weighed out to an accurate quantity (ψ_1). ψ_0

TABLE I
Typical Recipes Used in the Preparation of the Concentrated Emulsion

Component	Amount
Dispersed phase: MMA/BA (1/1)	50.00 g
Continuous phase: H ₂ O	0.01 L
Surfactant: SDS/HD (2/1)	1.50 g
Initiator: BPO-DMA	0.54 g

denotes the monomer mass that was used for polymerization in the concentrated emulsion. Therefore, polymerization stability was calculated according to the following equation:

$$\text{Bulk phase separated } (\alpha \%) = (\psi_1 / \psi_0) \times 100$$

Polymerization conversion

After polymerization, a small quantity of a 5% 1,4-hydroquinone aqueous solution was added to the polymerization products to hinder further polymerization. Then, the products were dried at about 100°C for 12 h, and we obtained the weight of the MMA/BA copolymer (W_1), considering other fixed substances. W_0 denotes the monomer mass used for polymerization. Thus, the polymerization conversion equation was obtained as follows:

$$\text{Polymerization conversion } (\%) = (W_1 / W_0) \times 100$$

Polymerization rate

During the polymerization process, we took out about 0.5-g samples from the reaction vessel at regular intervals and put them in a preweighed culture dish containing hydroquinone. The samples were dried in a vacuum oven until to a constant weight. The polymerization conversion was calculated by the gravimetric method. The slope of the conversion verse time curves (dc/dt ; conversion = 20–70%, at which the polymerization rate, R_p , remained relatively constant) and linear correlation coefficient (R) were calculated from follow equation: $R_p = dc/dt \times [M]_0$, where $[M]_0$ represents the initial monomer molar concentration.²⁰ At the same time, R_p , $\log R_p$, $\log [I]$, $\log [E]$ (where $[E]$ is the emulsifier concentration), and $\log R$ were figured out; the plots of $\log R_p$ versus $\log [I]$, $\log [E]$, and $\log R$ were drawn out, and their beeline slopes were calculated.

PCS

The particle sizes and distributions of particle diameter were determined by Photon Correlation Spectroscopy (PCS) (Malvern Auto Sizer Loc-Fc963), where D_Z

TABLE II
Effect of Initiator on the Polymerization Stability

R	α (%)	Other conditions	[I]	α (%)	Other conditions
1 : 1	0.58	[E] = 0.55 mol/L	0.36	0.44	[E] = 0.55 mol/L
1 : 0.65	1.12	$m[\text{SDS}]/m[\text{HD}] = 2 : 1$	0.54	1.60	$m[\text{SDS}]/m[\text{HD}] = 2 : 1$
1 : 0.5	1.60	[I] = 0.296 mol/L	0.72	2.04	$m[\text{BPO}]/m[\text{DMA}] = 2 : 1$
1 : 0.2	3.56		1.08	4.31	

$\Phi = 0.83$; temperature = 30°C; time = 5 h.

(nm) is the average diameter of the particles and *poly* refers to the distribution of particles (the value of *poly* changes between 0 and 1).

RESULTS AND DISCUSSION

Polymerization stability

The polymerization stability of a concentrated emulsion shows the stability degree of its cells in the course of polymerization for concentrated emulsions, namely, the cells' intensity and penetrability of the cells versus the monomers. Many experiments have revealed that polymerization stability is connected with the concentration of initiator, the category of reinforcers of the liquid film, the concentration of the compound surfactant, the polymerization environment, and so on.

The ratio of oxidant to reducer and the concentration of initiator can have obvious effects on the stability of the concentrated emulsions (Table II, where α is the percentage of monomer phase-separated from the concentrated emulsions, and *m* is mass). The results show that the polymerization stability decreased with *R* and increasing [I] within the scope of *R* and [I] that were studied because of the inhibition of DMA on the concentrated emulsion polymerization, although it was the reducer of the system. When the amount of DMA was lower, the efficiency of initiation and the self-heated temperature within a certain period were higher, but this led to a lower stability in the concentrated emulsions. Moreover, when [I] was higher, the one-side polymerization rate increased, and the partial temperature increased, which led to a lower polymerization stability; for the other side, when the amount of the polar initiator in the system increased, the polymerization stability also decreased.²¹⁻²²

A series of stable concentrated emulsions were prepared with the compound surfactants of different concentrations, and the effect of the concentration on the stability of the concentrated emulsions is presented in Figure 1, which shows that within a certain scope of compound surfactant concentrations, the amount of bulk phase separated decreased with increasing surfactant concentration, which resulted in a higher stability in the polymerization. This could be explained by the fact that the aqueous viscosity and the intensity

of the interface film increased with increasing [E]; thus, the concentrated emulsion became more stable. Moreover, the study showed that stable concentrated emulsions were not obtained if the compound surfactant concentrations were too high. In our experiments, suitable compound surfactant concentrations were in the range 0.55–0.77 mol/L of H₂O.

Table III indicates the effect of the reinforcers of the liquid film and the polymerization environment on the amount of bulk phase separated after polymerization. As expected, the extent of the effect on the stability of polymerization was different for the three kinds of reinforcers of the liquid film. The result was arranged in big or small order as follows: PVA < PVP < HPC < none. The hydrophilicity of PVA was stronger than that of PVP and HPC, which was due to the higher content of hydroxy in PVA. Moreover, the stronger liquid film formed among cells increased the polymerization stability of the concentrated emulsions because of the association happening between hydrogen bonds. Table III also shows that different polymerization environments may have changed the polymerization stability of the concentrated emulsions. The efficiency of the conducting heat in the water bath was higher than that in oven because it was easier for the water medium to remove the reaction heat inside the polymerization system, which resulted in the partial temperature in the water bath being lower than

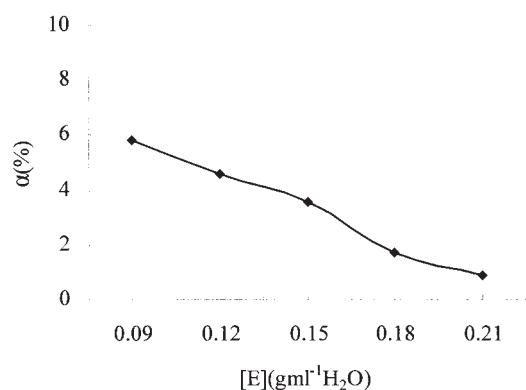


Figure 1 Relationship between various concentrations of surfactant and the phase separations ($m[\text{SDS}]/m[\text{HD}] = 2 : 1$; [I] = 0.296 mol/L; $m[\text{BPO}]/m[\text{DMA}] = 1 : 0.2$; $\phi = 0.83$; temperature = 30°C; time = 5 h).

TABLE III
Effect of the Categories of Reinforcers of the Liquid Film and the Polymerization Environment on the Polymerization Stability

Reinforcer	α (%)	Other conditions	Environment	α (%)	Other conditions
HPC	1.76	[E] = 0.55 mol/L	Oven	3.56	[E] = 0.55 mol/L
PVP	1.36	$m[\text{SDS}]/m[\text{HD}] = 2 : 1$			$m[\text{SDS}]/m[\text{HD}] = 2 : 1$
PVA	1.06	[I] = 0.296 mol/L	Water bath	1.21	[I] = 0.296 mol/L
None	3.56	$m[\text{BPO}]/m[\text{DMA}] = 2 : 1$			$m[\text{BPO}]/m[\text{DMA}] = 2 : 1$

$\Phi = 0.83$; temperature = 30°C; time = 5 h.

that in the oven. As a result, the amount of monomer loss decreased and the polymerization stability became higher.

Polymerization rate

Figure 2, which shows the time–conversion curves of MMA/BA prepared in concentrated emulsions of various initiator concentrations ([I] values), shows that polymerization rate increased with increasing [I] in a certain time (180 min or so); however, the curves expressing higher [I] values tended to be flat in the later period. This happened because the higher [I] was, the more free radicals were produced, which increased the polymerization rate. Furthermore, Figure 2 shows that higher conversions were obtained in a short time. The whole reaction time was about 300 min, and it was shortened greatly when the same high conversion was reached in comparison with the concentrated emulsion polymerization reaction initiated by heat initiators, and this was connected with a lower activity energy and higher initiation efficiency of the redox system based on BPO–DMA. The parameters obtained after the linear regression of their time–conversion curves in a constant rate phase are presented in Table IV. The interrelated coefficient (r) is 0.9962; thus, $R_p \propto [I]^{0.94}$ is obtained.

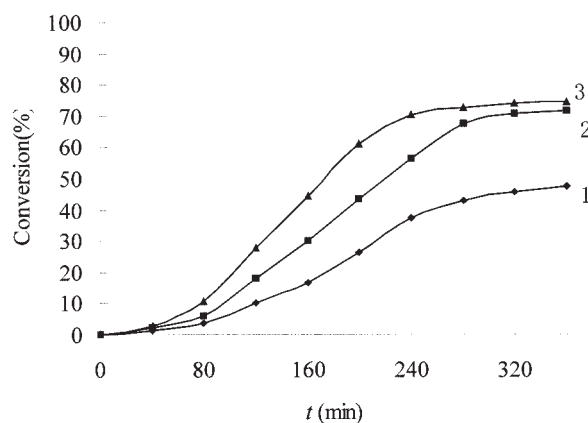


Figure 2 Conversion versus time (t) at various [I] values: (1) 0.198, (2) 0.296, and (3) 0.397 mol/L (water = 10 mL; $m[\text{SDS}]/m[\text{HD}] = 2 : 1$; [E] = 0.55 mol/L; $m[\text{BPO}]/m[\text{DMA}] = 2 : 1$; $\phi = 0.83$; temperature = 30°C).

Figure 3 indicates that the effect of R on the polymerization rate was obvious when [I] was fixed. Its time–conversion curve shows that the polymerization rate increased with increasing R . In theory, the mass ratio of the reaction happening between BPO and DMA is 2 : 1; the closer to the ratio is, the higher is the amount of free radicals that is produced,¹⁹ but here the amount of DMA was large, which led to an increase in the inhibition on the polymerization of the concentrated emulsions; on the side, the self-heated domino effect made the excessive BPO produce free radicals to participate in the reaction, so the polymerization rate increased. In the meanwhile, the study showed that the conversion exhibited a maximum 96.84% at BPO/DMA = 1 : 2 within 320 min; however, the polymerization rate began to decrease at BPO/DMA > 1 : 0.2. This happened because when the DMA concentration was too low, there were not enough free radicals produced for polymerization at low temperatures. The data obtained after the linear regression of the time–conversion curves in a constant rate phase are presented in Table V. The related coefficient was 0.9900, so, $R_p \propto R^{2.28}$.

Figure 4 compares the conversion as a function of time at various temperatures in concentrated emulsions of MMA/BA and shows that the rate of polymerization increased with increasing temperature, and the greater the temperature was, the shorter was the time needed to reach the highest conversion. The polymerization temperature could not exceed 40°C; otherwise, an explosion polymerization reaction would have easily happened, and the amount of monomer loss and the polymerization stability would have decreased, which was not suitable for the polymerization initiated by the redox system. Our experiments also showed that the difference in the final

TABLE IV
Effect of [I] on R_p

[I] (mol/L)	dc/dt (min^{-1})	r	R_p ($\text{mol/L}^{-1} \text{min}^{-1}$)	$\log R_p$	$\log [I]$
0.198	0.1867	0.9926	8.31	0.919	-0.703
0.296	0.2846	0.9998	12.66	1.102	-0.529
0.397	0.3915	0.9962	17.42	1.241	-0.401

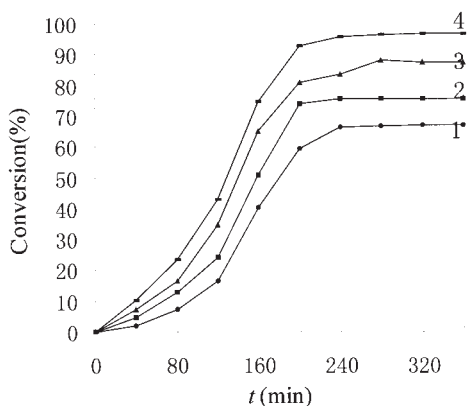


Figure 3 Conversion versus time (t) at various ratios of oxidant to reducer: (1) 1 : 1, (2), 1 : 0.65, (3) 1 : 0.5, and (4) 1 : 0.2 (water = 10 mL; $m[\text{SDS}]/m[\text{HD}] = 2 : 1$; $[\text{E}] = 0.55$ mol/L; $[\text{I}] = 0.296$ mol/L; $\phi = 0.83$; temperature = 30°C).

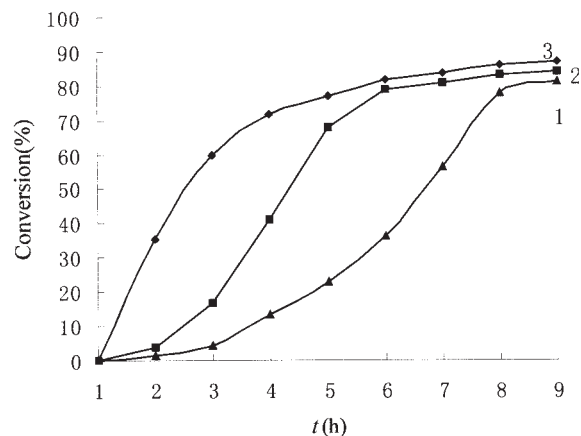


Figure 4 Conversion versus time (t) at various polymerization temperatures: (1) 20, (2) 30, and (3) 40°C (water = 10 mL; $m[\text{SDS}]/m[\text{HD}] = 2 : 1$; $[\text{E}] = 0.55$ mol/L; $[\text{I}] = 0.296$ mol/L; $m[\text{BPO}]/m[\text{DMA}] = 5 : 1$; $\phi = 0.83$).

conversions at 20 and 30°C was slight, and the latter time was about half of the former, so 30°C was the optical reaction temperature. The polymerization rate constant was obtained after linear regression of the time–conversion curves in a constant rate phase. The decomposing activation energy of the initiator ($E_d = 31.6$ kJ/mol) was obtained according to the Arrhenius experience formula.

Some high polymers, such as HPC, PVP, and poly(ethylene alcohol), that can dissolve in the aqueous solution may be taken for reinforcers of the liquid film. The conversion as a function of time is plotted in Figure 5. The time–conversion curves showed that there was a slight difference in the extent of the effect of the aforementioned reinforcers of the liquid film on the polymerization rate, among which the results obtained are arranged in big or small order as follows: PVA > PVP > HPC. A layer of strong film that formed around the cells after combination with water molecules increased the hydrophilicity of the reinforcers of the liquid film decreased the amount of monomer loss and transference between cells and reduced the termination frequency of double radicals, which resulted in an increase in the polymerization rate.

The polymerization conversion with changing concentration of surfactant was determined by a change in the concentration of the compound surfactant when

$[\text{I}]$, R , and the monomer concentration were fixed, which is presented in Figure 6. Figure 6 shows that within the suitable scope of concentrations of the compound surfactant, the polymerization rate increased with increasing $[\text{E}]$. When the concentrations of the compound surfactant increased, the volume of cells decreased, and the amount of cells increased; that is, the point of polymerization increased, where it could be expected that the crosslinking copolymer was increased. Furthermore, the film of cells thickened, and the intensity of cells increased, which led to the transference and monomer loss to decrease. In the meanwhile, the double-molecule termination frequency also decreased, and the initiation efficiency and the stability of the concentrated emulsions increased, which resulted in an increase in the polymerization

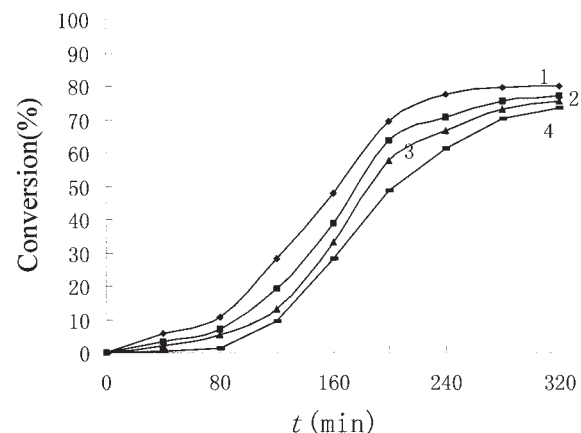


Figure 5 Conversion versus time (t) with various categories of reinforcers of the liquid film: (1) PVA, (2) PVP, (3) HPC, and (4) none (water = 10 mL; $m[\text{SDS}]/m[\text{HD}] = 2 : 1$; $[\text{E}] = 0.55$ mol/L; $[\text{I}] = 0.296$ mol/L; $m[\text{BPO}]/m[\text{DMA}] = 5 : 1$; $\phi = 0.83$; temperature = 30°C).

TABLE V
Effect of the Ratio of Oxidant to Reducer on R_p

R	dc/dt (min^{-1})	r	R_p ($\text{mol/L}^{-1} \text{min}^{-1}$)	$\log R_p$	$\log R$
1 : 1	0.3073	0.9819	13.67	1.14	0
1 : 0.65	0.3681	0.9986	16.38	1.21	0.19
1 : 0.5	0.4193	0.9943	18.66	1.27	0.30
1 : 0.2	0.4813	0.9942	21.42	1.33	0.70

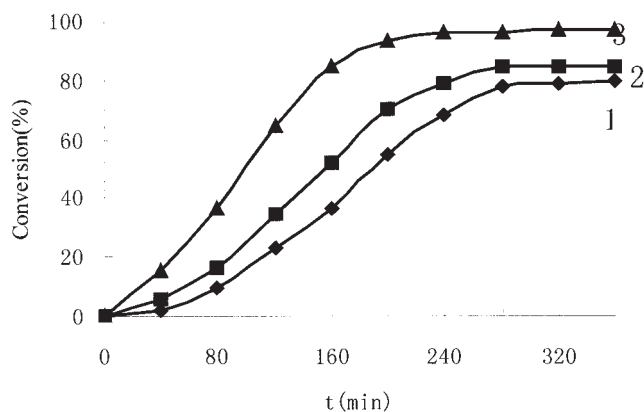


Figure 6 Conversion versus time (t) at various $[E]$ values: (1) 0.44, (2) 0.55, and (3) 0.66 mol/L (water = 10 mL; $m[\text{SDS}]/m[\text{HD}] = 2 : 1$; $[I] = 0.296$ mol/L; $m[\text{BPO}]/m[\text{DMA}] = 1 : 0.2$; $\phi = 0.83$; temperature = 30°C).

rate. The parameters obtained after the linear regression of their time–conversion curves in a constant phase are presented in Table VI. The related coefficient (r) is 0.9818; thus, $R_p \propto [E]^{0.17}$ is obtained.

Size of the latex particles and distribution of particle sizes

Several important parameters, including as the size and shape of the latex particles and the distribution of particle sizes in the polymerization of the concentrated emulsions, could affect the function of utilization greatly. The study showed that when the concentration of monomer was given, factors such as $[E]$, the mass ratio of SDS to HD, $[I]$, the polymerization temperature, and the polymerization environment could

have a great effect on the particle size and distribution of particle sizes, which is presented in Table VI.

Both the average particle size and the distribution of particle sizes increased with increasing $[I]$. This happened because the amount of the polar initiator increased with increasing $[I]$, and moreover, the self-heated temperature also rose, which led to an agglomeration of cells; thus, as a result, the latex size increased and the distribution of particle sizes became wider.

When the polymerization temperature increased, the latex average particle size and distribution of particle sizes increased because of the higher temperature results in the lower stability of concentrated emulsions and the higher agglomeration extent of cells. This could be regarded as a typical example to illustrate the effect of every factor on the shape of the latex particles. This shows that the shape of latex particles and the distribution of particle sizes became more irregular than before and that the agglomeration extent of the cells increased.

With the mass ratio of SDS to HD increasing, the average particle size and distribution of particle sizes decreased because of the increasing amount of the effective surfactant SDS with the increasing mass ratio of SDS to HD, which led to the average particle size decrease; however, $[E]$ was changeless, so the distribution of particle size decreased at all times.

With the alteration of the polymerization environment, the latex particle size and the distribution of particle sizes were obviously different. Both the average polymer particle size and the distribution of particle sizes decreased after the reaction in the water bath compared with that in the oven. This happened because the efficiency of the conducting heat of water

TABLE VI
Latex Particle Size and Distribution of Particle Sizes Under Different Conditions

Condition	Variable	D_z (nm)	Poly	Other conditions
$[E]$ (mol/L)	0.44	164.7	0.497	$m(\text{SDS})/m(\text{HD}) = 2 : 1$; $[I] = 0.296$ mol/L; $m(\text{BPO})/m(\text{DMA}) = 1 : 0.2$; $T = 30^\circ\text{C}$
	0.55	142.0	0.352	
	0.66	128.1	0.124	
	0.77	119.1	0.192	
	0.198	110.6	0.240	
$[I]$ (mol/L)	0.296	142.0	0.352	$m(\text{SDS})/m(\text{HD}) = 2 : 1$; $m(\text{BPO})/m(\text{DMA}) = 1 : 0.2$; $T = 30^\circ\text{C}$
	0.397	291.7	0.454	
T ($^\circ\text{C}$)	20	114.4	0.179	$m(\text{SDS})/m(\text{HD}) = 2 : 1$; $[I] = 0.296$ mol/L; $m(\text{BPO})/m(\text{DMA}) = 1 : 0.2$
	30	142.0	0.352	
	40	164.1	0.471	
$m(\text{SDS})/m(\text{HD})$	1 : 1	325.1	0.373	$[E] = 0.55$ mol/L; $m(\text{BPO})/m(\text{DMA}) = 1 : 0.2$; $T = 30^\circ\text{C}$
	2 : 1	142.0	0.352	
	3 : 1	132.0	0.315	
	4 : 1	123.4	0.171	
Environment	Oven	142.0	0.352	$m(\text{SDS})/m(\text{HD}) = 2 : 1$; $[E] = 0.55$ mol/L; $[I] = 0.296$ mol/L; $m(\text{BPO})/m(\text{BPO})/m(\text{DMA}) = 1 : 0.2$; $T = 30^\circ\text{C}$
	Water	106.3	0.156	
	Bath			

$m[\text{MMA}]/m[\text{BA}] = 1/1$; $\text{H}_2\text{O} = 10\text{mL}$; $T = \text{temperature}$.

is higher than that of air, and the self-heated temperature inside the system is lower, which leads to a higher stability in concentrated emulsions.

CONCLUSIONS

1. The stable concentrated emulsions of MMA/BA were prepared by a one-step method, and the suitable compound surfactant concentrations were in the range 0.55–0.77 mol/L of H₂O. The extent of the effect of the three kinds of reinforcers of liquid film on the stability of polymerization was arranged in big or small order as follows: PVA < PVP < HPC < none. The polymerization stability was higher in the water bath than in the oven. For the polymerization stability of the concentrated emulsions, the MMA content had to be limited under a certain scope.
2. The copolymerizations were carried out in a water bath or in an oven at various temperatures. Alterations in the amount of [E] and [I], the categories of liquid film, and the temperature led to a variation in the conversion and polymerization rates. The results are $R_p \propto [I]^{0.94}$, $R_p \propto R^{2.28}$, and $R_p \propto [E]^{0.17}$.
3. Both the average particle size and the distribution of particle sizes increased with increasing [I], with temperature increasing and the mass ratio of SDS to HD decreasing with reaction in the oven versus the water bath.

4. As expected, under certain conditions, the conversion was as large as 96.84%.

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