

On the Kinetics of Microemulsion Copolymerization of Acrylonitrile

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The o/w microemulsion polymerizations and copolymerizations of butyl acrylate (BA), styrene (St), and acrylonitrile (AN) initiated by a water-soluble initiator in the presence and absence of additives were investigated. Polymerizations and copolymerizations show two distinct nonstationary rate regions. The addition of isopropanol (IPA) was found to decrease the rate of polymerization and the decrease was more pronounced in the copolymerization runs. The rate of polymerization and the rate of particle formation decrease with increasing acrylonitrile concentration. The BA/AN copolymer latexes were more stable than the AN/St ones.

Generally, a microemulsion formed spontaneously by mixing oil and water in the presence of appropriate amount of emulsifier and coemulsifier is thermodynamically stable and transparent. Oil-in-water-type (o/w) microemulsion homopolymerizations of unsaturated monomers were already intensively investigated. A little has been done, however, on the kinetics of o/w microemulsion copolymerizations.

The phase diagram of the direct microemulsion allows to attain high volume fractions of the disperse phase. The direct microemulsion polymerizations and copolymerizations of oil-soluble monomers in polymer particles represent an attractive way to produce high-molecular mass polymers at a very high polymerization rate.

In the previous paper [1] we have reported on the effect of a partly water-soluble monomer acrylonitrile (AN) on the kinetics of microemulsion polymerization in the range of low and medium AN concentrations. A goal of this paper is to study the three- or four-component microemulsion (co)polymerizations of an oil-soluble monomer (butyl acrylate or styrene) with a partly water-soluble monomer (acrylonitrile) at a very high AN concentration. Acrylonitrile partitions between water and micelles (or particles), whereas butyl acrylate (BA) or styrene (St) is mostly located in the micelles (or the polymer particles). This results from the different solubility of monomers in water which is for AN 1.6 mol dm^{-3} , BA $0.011 \text{ mol dm}^{-3}$, and St $0.003 \text{ mol dm}^{-3}$ [2]. Thus, we intend to follow the effects of the solubility of monomer in water and the coemulsifier activity of monomers on the microemulsion copolymerization. The high concentration of emulsifier was used to get very small and more stable polymer particles and to suppress the agglomeration events.

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EXPERIMENTAL

Commercially available butyl acrylate (BA), styrene (St), and acrylonitrile (AN) monomers were purified by the traditional method. The initiator ammonium peroxodisulfate (the water-soluble initiator, APS, extra-pure from Fluka) was used as supplied. The emulsifier used was a reagent-grade sodium dodecylsulfate (SDS, from Fluka). Twice-distilled water and distilled isopropanol (IPA) were used.

The batch polymerizations were performed at 60°C . In all runs the recipe comprises 100 g of water, 20 g of SDS, 0.144 g of APS, and 0.025 g of NaHCO_3 . System A: the mass ratio $m(\text{AN})/m(\text{BA}) = 1.0$, 10 g of monomer, system B: the mass ratio $m(\text{AN})/m(\text{BA}) = 3.0$, 16 g of monomer, system C: the mass ratio $m(\text{AN})/m(\text{BA}) = 3.0$, 20 g of monomer, the BA system: 10 g of BA, and the system D: the mass ratio $m(\text{AN})/m(\text{St}) = 3.0$, 16 g of monomer. Amounts and type of additives varied as shown later.

The polymerization technique and the measurements of particle size and number (a light scattering method – LSM) were the same as described earlier [3–5]. Conversion of monomer was determined by dilatometric measurements (checked by gravimetry). The final conversion of each experiment was determined by both gas chromatography and dilatometry [6]. This approach showed a good reproducibility of all dilatometric experiments.

RESULTS AND DISCUSSION

Polymerization Rate

Figs. 1 and 2 show the conversion—time data of

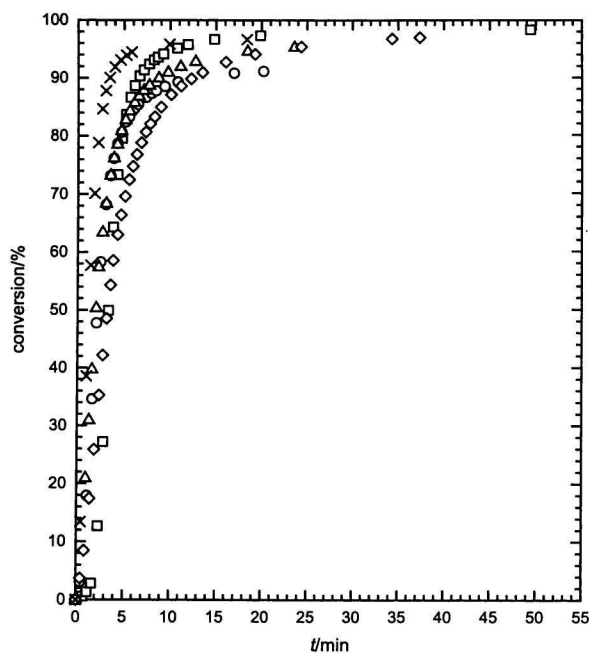


Fig. 1. Variation of monomer conversion in the microemulsion polymerization of BA initiated by APS with the IPA concentration and the reaction time. $[BA] = 0.92 \text{ mol dm}^{-3}$ $[IPA]/(\text{mol dm}^{-3})$: \times 0, \square 0.12, \circ 0.24, Δ 0.49, \diamond 0.98.

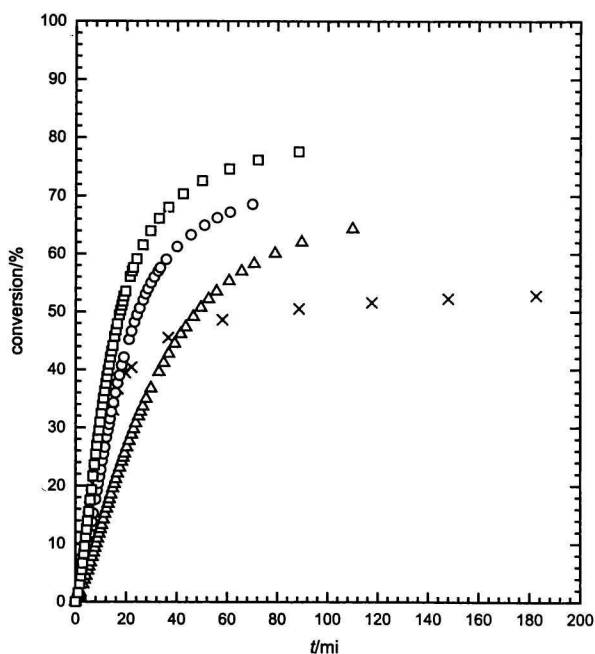


Fig. 2. Variation of monomer conversion in the microemulsion copolymerization of BA and AN initiated by APS with the IPA concentration and the reaction time. System B, the mass ratio $m(\text{AN})/m(\text{BA}) = 3.0$, $[\text{AN}] = 1.64 \text{ mol dm}^{-3}$, $[\text{BA}] = 0.226 \text{ mol dm}^{-3}$ $[IPA]/(\text{mol dm}^{-3})$: \times 0, \square 0.12, \circ 0.24, Δ 0.48.

the radical microemulsion (co)polymerization of butyl acrylate (BA) and acrylonitrile (AN) initiated by am-

monium peroxodisulfate (APS) in the presence of isopropanol (IPA). A "sigmoidal" shape of the conversion curves, typical for the conventional emulsion polymerizations, is somewhat suppressed in these runs.

Initially, the conventional emulsion polymerization is very slow. As the polymerization proceeds, the rate of emulsion polymerization gradually increases, reaching approximately a constant value of the rate of polymer formation within a long time period (interval 2) [7]. The initial increase in the rate is the direct consequence of the generation of new particles. In the interval 2 the number of particles and the concentration of monomer in particles are constant.

In the present microemulsion polymerization the variation of rate with conversion is quite different. This may be attributed to the rapid initial nucleation and the particle nucleation during the whole polymerization due to the very large number of monomer-swollen micelles (*ca.* $10^{22}/\text{dm}^3$). Indeed, the abrupt increase in the particle concentration was observed after the start of polymerization ([8], see below).

In all BA runs the final conversion close to 100 % was reached after *ca.* 10 min. In the copolymerization systems a very slow polymerization was observed giving above 60 % or 80 % conversion (the limiting conversion). The limiting conversion was reached in 1.5–3 h. The limiting conversion or the slow polymerization may be ascribed to the water-phase polymerization of AN, the chain-transfer events (mainly to isopropanol) in the aqueous phase, the participation of transferred IPA radicals in the water-phase termination, and to the occlusion events.

Variations of the rate of polymerization with conversion, the monomer feed composition, and the IPA concentration are expressed in Figs. 3 and 4, and Table 1, respectively. The polymerization rate at different conversions was determined by the nonlinear least-squares regression analysis. In both systems the polymerization rate—conversion curves show two distinct regions.

The maximum rate of (co)polymerization of BA and AN as a function of the IPA concentration ($[IPA]_0 < 0.5 \text{ mol dm}^{-3}$) obeys the following relationships (see Table 1)

$$\begin{aligned} R_{p,\max}(\text{BA}) &\propto [IPA]_0^{-0.2} \text{ and} \\ R_{p,\max}(\text{BA/AN}) &\propto [IPA]_0^{-0.65} \end{aligned} \quad (1)$$

These results show that IPA lowers the rate of polymerization and the decrease is more pronounced in the copolymerization. The presence of AN increases the retardation effect of IPA on the growth events. This finding may be attributed to the increased desorption of radicals, increased water-phase termination, and the high water solubility of IPA-transferred radicals. The accumulation of IPA-transferred radicals in water favours the termination events and depresses the radical entry ones.

Table 1. Variations of the Kinetic Parameters of Microemulsion (Co)Polymerization of BA and AN^a

[BA] ₀	[AN] ₀	[IPA] ₀	$R_{p,max} \cdot 10^3$	$R_{pp} \cdot 10^{21}$	Q	D	$N \cdot 10^{-18}$
mol dm ⁻³	mol dm ⁻³	mol dm ⁻³	mol dm ⁻³ s ⁻¹	mol particle ⁻¹ s ⁻¹	particle ⁻¹	nm	dm ⁻³
0.92	—	0.122	8.0	2.6	0.26	35	6.3
0.92	—	0.245	6.7	1.5	0.15	31	9.0
0.92	—	0.49	6.0	0.9	0.09	27	14.0
0.92	—	0.98	3.8	0.7	0.07	30	10.0
0.226	1.64	0.12	1.35	1.9	0.04	48	2.0
0.226	1.64	0.24	0.97	3.2	0.07	65	0.86
0.226	1.64	0.48	0.55	14.0	0.16	250	0.012

a) In all runs 16 g of monomer per 100 g of water.

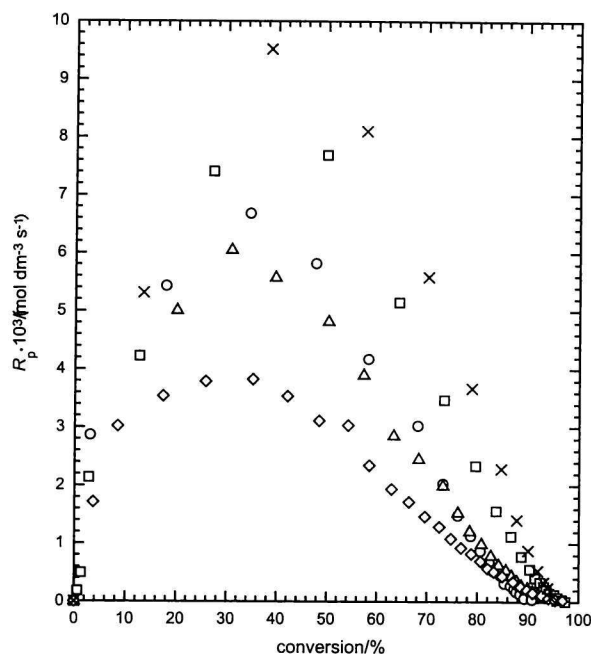


Fig. 3. Variation of the rate of polymerization in the microemulsion polymerization of BA with the IPA concentration and conversion. [BA] = 0.92 mol dm⁻³ [IPA]/(mol dm⁻³): × 0, □ 0.12, ○ 0.24, △ 0.49, ◇ 0.98. See legend to Fig. 1.

In the BA system the maximum rate of polymerization is observed at *ca.* 40 % conversion. The addition of IPA does not change the position of the maximum rate on the R_p —conversion curve, *i.e.*, it is observed at 30—40 % conversion. On the contrary, addition of AN to BA shifts the maximum rate strongly back to 10 % conversion. Fig. 4 shows that the rate of copolymerization of AN and BA strongly decreases with conversion. The addition of IPA to AN/BA copolymerization system decreases the maximum rate but the decrease of rate with conversion is much less pronounced than that without IPA. This may be attributed to the “short-stopped effect” of IPA-transferred radicals that decreases the consumption of monomer in particles. And therefore the maximum rate of polymerization appears at the higher conversion.

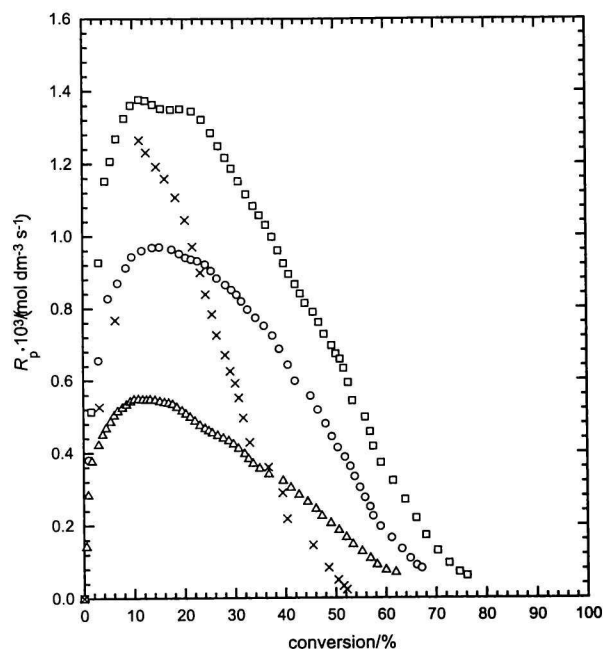


Fig. 4. Variation of the rate of polymerization in the microemulsion copolymerization of BA and AN with the IPA concentration and conversion. System B, [AN] = 1.64 mol dm⁻³, [BA] = 0.226 mol dm⁻³ [IPA]/(mol dm⁻³): × 0, □ 0.12, ○ 0.24, △ 0.48. See legend to Fig. 2.

The decrease of the rate of polymerization beyond *ca.* 20 % conversion may be attributed to the depletion of emulsified monomer droplets and the decrease of monomer concentration in the micelles and/or polymer particles. In the emulsion polymerization of BA, the monomer droplets disappear at *ca.* 40 % conversion, and the monomer/polymer ratio ($\omega_{m/p}$) in the particle is *ca.* 1.7 [9]. In our earlier paper we reported that the $m(\text{monomer})/m(\text{polymer})$ mass ratio for the BA microemulsion polymerization at 40—50% conversion is in the range 0.7—1.0 [8]. This indicates that the microemulsion polymerization of BA already proceeds at medium conversions under the monomer-starved conditions. Besides, the increase of the particle number with conversion (at medium and high conversions)

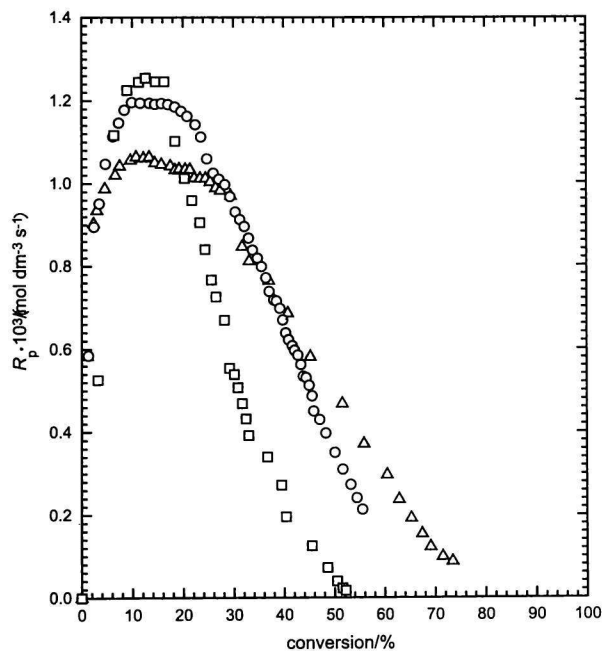


Fig. 5. Variation of the rate of polymerization in the microemulsion copolymerization of BA and AN with the monomer feed composition, n_{BA} and conversion. System B, the mass ratio $m(AN)/m(BA)$ changed from 7.3 to 16.9; n_{BA} : \square 0.059, \circ 0.087, Δ 0.121.

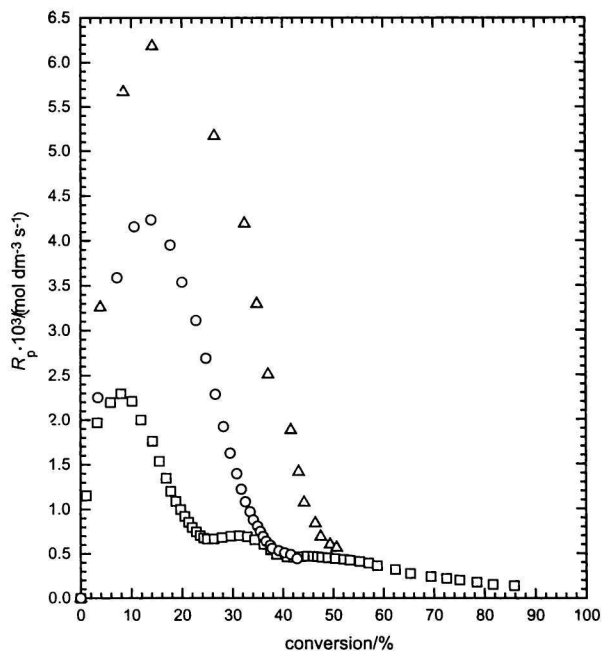


Fig. 6. Variation of the rate of polymerization in the microemulsion copolymerization of St and AN with the monomer feed composition, n_{St} and conversion. System D, the mass ratio $m(AN)/m(St)$ changed from 3.0 to 7.0; n_{St} : \square 0.068, \circ 0.105, Δ 0.145.

more pronounces the decrease of the monomer concentration in the polymer particles. Thus, any acceleration of polymerization can appear only at low conversion. Indeed, the abrupt increase in R_p is observed after the start of polymerization (Figs. 3 and 4).

In the microemulsion polymerization the new polymer particles are generated throughout the polymerization ([10], see later). This behaviour results from a much higher concentration of emulsifier required in the microemulsion polymerization. Thus, the three-interval concept typical for the classical emulsion polymerization is not valid for the microemulsion polymerization. At medium or high conversion the reaction loci are situated in the interface zone. Here, the polymerization is slower due to a lower concentration of monomer (a dilution approach [10]).

Variations of the rate of polymerization with conversion, the monomer feed composition, and the IPA concentration are expressed in Figs. 5 and 6, and Tables 1 and 2, respectively. Experimental results obey the following relationships

$$R_{p,\max}(\text{system B}) \propto [AN]_0^{-1.1} [BA]_0^{0.24} \quad (2)$$

$$R_{p,\max}(\text{system C}) \propto [AN]_0^{-3.7} [BA]_0^{0.8} \quad (3)$$

and

$$R_{p,\max}(\text{system D}) \propto [AN]_0^{-7.5} [St]_0^{1.5} \quad (4)$$

In all runs the rate of polymerization decreases with

Table 2. Variations of the Kinetic Parameters of Microemulsion (Co)Polymerization of St and AN^a

$[AN]_0$	$[St]_0$	$R_{p,\max} \cdot 10^3$
mol dm ⁻³	mol dm ⁻³	mol dm ⁻³ s ⁻¹
1.63	0.277	6.3
1.77	0.208	4.3
1.91	0.14	2.3

a) In all runs 16 g of monomer per 100 g of water.

increasing AN (or the total monomer) concentration and the decrease is more pronounced in the system with the higher total monomer concentration. Besides, the negative reaction order is much larger in the copolymerization systems with St. These findings may be attributed to several contributions: 1. the different coemulsifier properties of monomers (AN, BA, St), 2. the different partition of monomers between the reaction phases, 3. the different chain transfer and exit events, and 4. the different copolymerization parameters. The BA itself acts as a coemulsifier [8, 10] and therefore BA favours the growth events within the shell of micelles or particles where also takes part in stabilization events. The St monomer is situated mainly in the core of micelles or particles where takes part in growth events. The AN monomer is situated in water and in the core of micelles and particles and favours polymerization in water.

In the BA system the rate per particle decreases with [IPA]. This finding may be explained by all the monomers dilution, the radical desorption, and the increased (water phase) termination. In the BA/AN runs, however, the opposite is true. This may be attributed to the increased particle size due to an increased agglomeration of particles. The high water solubility of AN increases the portion of IPA in water, due to which the nature of the continuous phase strongly changes. Here, the coagulative nucleation, the water-phase polymerization, the radical desorption, and the agglomeration of particles influence the polymerization behaviour in a complex way. Desorption of radicals in the microemulsion polymerization of BA was experimentally proved by an increase of the particle number after the cessation of irradiation [11]. This process will be much more pronounced in the presence of AN or IPA.

The AN/BA copolymerizations are found to be much slower than the BA polymerizations. The values of k_p (see later) for AN and BA, however, should favour the opposite trend in the polymerization rates. This behaviour may be attributed to the high water solubility of AN, the decreased monomer concentration in particles, the increased monomer concentration in water, the high water-phase polymerization and termination, and the high desorption rate. Thus, the reaction loci are both in particles and in the aqueous phase. The polymerization in water is by 1 or 2 orders in magnitude lower than that in particles and therefore the addition of AN will decrease the total rate of polymerization (favouring the polymerization in water).

If the rates of polymerization are divided by the number of particles and the appropriate constants, the average particle radical number, Q , can be estimated according to the approach described earlier [12]. The values used were k_p (BA) = 1360 dm³ mol⁻¹ s⁻¹, k_p (AN) = 5000 dm³ mol⁻¹ s⁻¹, [AN/BA] = 8.5 mol dm⁻³ and τ_{AN} = 1.08, τ_{BA} = 0.816 [12, 13]. The estimated values for Q are shown in Table 1. These results indicate that the Q value for the AN/BA copolymer particles is much smaller than that for the poly(butyl acrylate) particles [11]. The low radical concentration in the copolymer microparticles results from both the higher radical desorption and water-phase termination rates as it was mentioned above.

Colloidal Parameters

Variations of the size of particles with the monomer feed composition, the comonomer type, conversion, and additives are summarized in Table 1 and Figs. 7–10. The monomer/polymer particle size is found to be a function of the monomer feed composition and conversion. In the system A the diameter of monomer/polymer particle is nearly independent of conversion. Here, the size slightly increases above the critical conversion (conv_{crit} ca. 80 %). With increas-

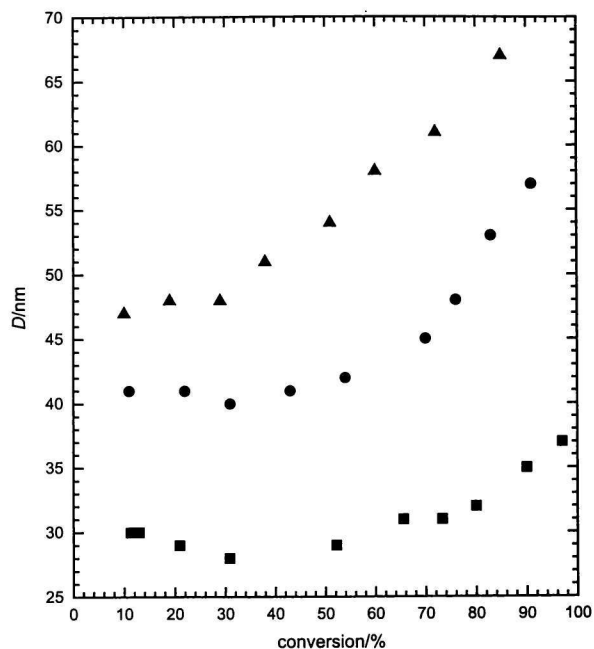


Fig. 7. Variation of the particle diameter (D) in the microemulsion copolymerization of BA and AN with the monomer feed composition and conversion. \blacktriangle system C, the mass ratio $m(\text{AN})/m(\text{BA}) = 3.0$, $[\text{AN}] = 2.0$ mol dm⁻³, \bullet system B, $m(\text{AN})/m(\text{BA}) = 3.0$, $[\text{AN}] = 1.64$ mol dm⁻³, \blacksquare system A, $m(\text{AN})/m(\text{BA}) = 1.0$, $[\text{AN}] = 0.72$ mol dm⁻³

ing AN concentration or the total monomer concentration the critical conversion is shifted to lower values. This finding is attributed to agglomeration of particles, which increases with increasing AN fraction in the comonomer feed. The presence of AN in water changes the kinetics of polymerization and the solvent power of the continuous phase. Fig. 7 shows that the critical conversion in the system B and C is 60 % and 30 %, respectively. Variations of the particle (the monomer-swollen polymer particle and the polymer particle (without monomer)) size with conversion shown in Fig. 9 indicate that the agglomerations are also more pronounced in the copolymer systems. The BA system is colloidally stable and therefore the particle size changes only slightly with conversion. The nucleation and growth events proceed within the monomer-swollen micelles. The premature polymer particles are colloidally stable and act as a monomer reservoir. In the runs rich in AN the particle size increases during the whole polymerization, especially at high conversion. Here, the polymer particles seem to take part in agglomeration during the whole copolymerization.

The dependence of N vs. conversion in the microemulsion AN/BA copolymerization is described by a curve with a maximum at 50 % conversion in the system A, at 40 % in the system B, and at 25 % in the system C (Fig. 8), respectively. In the BA sys-

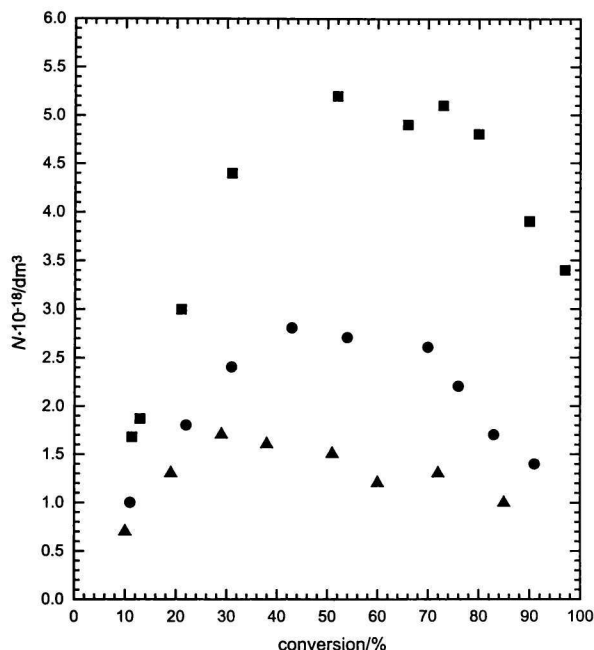


Fig. 8. Variation of the particle number (N) in the microemulsion copolymerization of BA and AN with the monomer feed composition and conversion. ▲ system C, the mass ratio $m(\text{AN})/m(\text{BA}) = 3.0$, $[\text{AN}] = 2.0 \text{ mol dm}^{-3}$, ● system B, $m(\text{AN})/m(\text{BA}) = 3.0$, $[\text{AN}] = 1.64 \text{ mol dm}^{-3}$, ■ system A, $m(\text{AN})/m(\text{BA}) = 1.0$, $[\text{AN}] = 0.72 \text{ mol dm}^{-3}$

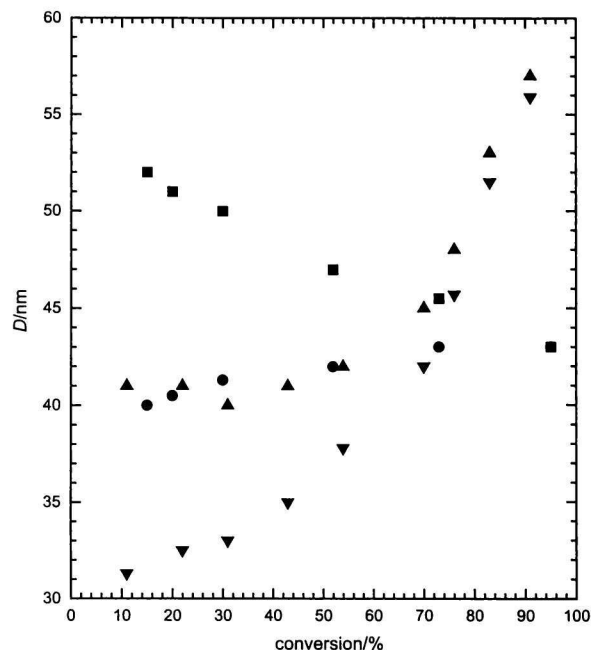


Fig. 9. Variation of the particle diameter (D) in the microemulsion polymerization of BA and copolymerization of BA and AN with the monomer feed composition and conversion. Homopolymerization; $[\text{BA}] = 0.6 \text{ mol dm}^{-3}$: ■ monomer-saturated polymer particles and ● polymer particles (without monomer). Copolymerization; the mass ratio $m(\text{AN})/m(\text{BA}) = 3.0$, system B, $[\text{AN}] = 1.64 \text{ mol dm}^{-3}$: ▲ monomer-saturated copolymer particles and ▼ copolymer particles (without monomer).

tem the number of particles, however, increases linearly with conversion (Fig. 10). This indicates that the nucleation proceeds during the whole polymerization. The decrease of the copolymer particle number with conversion, especially at high conversions, is caused by the increased agglomeration of polymer particles. This behaviour can be ascribed to the increased water-phase homopolymerization of AN, the change of water-continuous phase to an AN/water one, the change of copolymer composition on the particle surface, and the decreased interaction of the polymer particle surface with emulsifier.

At a very high conversion the comonomer-free mixture is rich in AN [3, 7]. Under such conditions the polymer chains or particle surfaces are rich in AN units. The reduced adsorption of SDS by the particles and the AN-saturated water phase are assumed to disfavour the colloidal stability of the polymer latex.

On the other hand, the microemulsion copolymerization of AN and St does not lead to the formation of the colloidally stable polymer particles. The difference in the polymerization behaviour between AN/BA and AN/St results from the difference in the coemulsifier activity between BA and St. This is due to the fact that BA is located mostly in the interphase zone while St is located in the core of micelles.

The surface area of the (initial) BA/SDS micelles

is by several orders in magnitude larger than that of the polymer particles [1, 8, 10]. The small fraction of micelles is nucleated or used for the stabilization of polymer particles. Thus, the polymer particles can only slightly compete with the monomer-containing micelles in capturing radicals. The each entry of radical to monomer-swollen micelle leads to the nucleation (formation of a primary particle). The very large number of micelles ($N_{\text{micelles}} \gg N_{\text{particles}}$) indicates that radicals are predominantly captured with the monomer-swollen micelles. This means that the polymer is mostly formed in the monomer-swollen micelles.

It is known that the variation of chain length of aliphatic alcohols strongly influences the stability and/or the interactions between micelles [14]. Besides, the chain transfer to alcohol and the formation of more stable radicals (with IPA) may also influence the polymerization kinetics [15].

The number of particles was found to vary in a complex way with increasing $[\text{AN}]$, $[\text{BA}]$, $[\text{St}]$, and $[\text{IPA}]$ (Tables 1 and 2). The exponents a , b , c , and d from the relationships $N \propto [\text{I}]^i$ for $[\text{I}]^i$: $[\text{AN}]^a$, $[\text{BA}]^b$, $[\text{St}]^c$, and $[\text{IPA}]^d$ are estimated to be

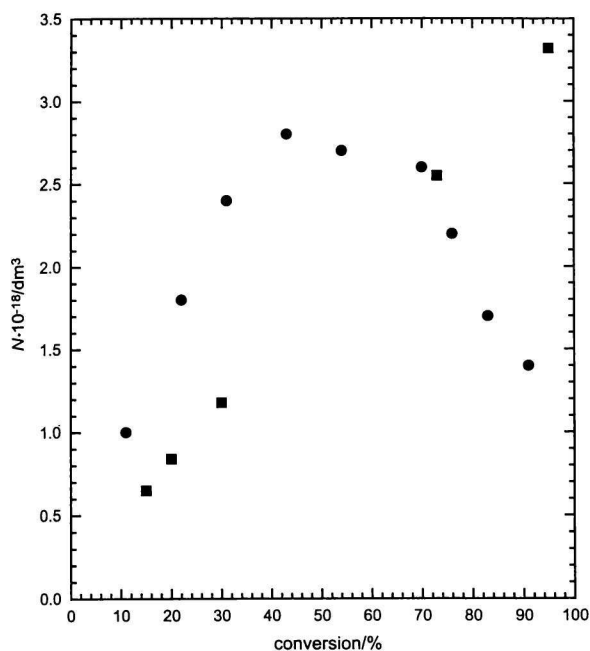


Fig. 10. Variation of the particle number (N) in the microemulsion polymerization of BA and copolymerization of BA and AN with the monomer feed composition and conversion. ■ Homopolymerization; $[BA] = 0.6 \text{ mol dm}^{-3}$; and ● copolymerization; the mass ratio $m(\text{AN})/m(\text{BA}) = 3.0$, system B, $[\text{AN}] = 1.64 \text{ mol dm}^{-3}$

the BA system (see Table 1):

$$b = 1.4 [14], d = 0.56 \quad (5)$$

system B (see Table 1):

$$a = -0.41, b = 9.3, d = -3.7 \quad (6)$$

$$\text{system C: } a = -3.7, b = 0.8 \quad (7)$$

$$\text{system D: } a = -7.5, c = 1.5 \quad (8)$$

These data show that IPA favours the formation of particles in the BA run but rather disfavors the particle nucleation in the AN/BA runs. In the AN/BA runs both the strong desorption of radicals and the water-phase termination strongly depress the entry rate of radicals to micelles and therefore decrease the rate of particle formation. The hydrophobic monomer BA favours the formation and stabilization of particles while the hydrophilic monomer AN favours the agglomeration events. The St monomer favours the growth events in the particle core.

In the former system (BA) the colloidal stability of the polymer dispersion increases in the presence of IPA. This may be attributed to the formation of low-molecular mass of poly(butyl acrylate)s which act as a coemulsifier. However, in the latter system AN/BA the radical desorption and the water-phase termination are assumed to decrease both the rate of poly-

merization and the colloidal stability of polymer dispersion.

CONCLUSION

Polymerizations of transparent o/w microemulsions of BA initiated by APS produce relatively stable microlatexes. The addition of AN more favours the agglomeration events. These latexes contain spherical AN/BA copolymer particles with diameter ranging from 40 nm to 60 nm. On the contrary, the microemulsion copolymerizations of AN with St do not lead to the formation of the polymer microemulsions. These findings were attributed to the high coemulsifier activity of BA and low or no surface activity of St and AN or their oligomers.

The dependences of the rate of polymerization and the number of particles *vs.* conversion are described by a curve with a maximum at a certain conversion. These results were discussed in terms of the variation of the monomer concentration, the degree of monomer (AN) saturated water phase, the chain length of precipitated oligomers, the nucleation of micelles, and the agglomeration of particles.

The IPA itself was found to increase the nucleation activity of the microemulsion polymerization of BA as a result of the decrease of molecular mass of poly(butyl acrylate) and/or the participation of the low-molecular mass products (as a coemulsifier) in the stabilization of polymer particles. On the contrary, the addition of IPA decreased the particle nucleation in the AN/BA runs as a result of a decreased entry rate of radicals to particles and the strong contribution of the water-phase polymerization.

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