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Continuous miniemulsification for the industrialization of miniemulsion polymerization

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ABSTRACT

Miniemulsion polymerization allows the synthesis of a broad range of materials, which are not obtainable by means of other polymerization processes in dispersed media such as conventional emulsion polymerization. However, the industrial implementation of the miniemulsion polymerization is being hindered by the miniemulsification process, which may require long times leading to unacceptable process times. This work explores the feasibility of using a continuous miniemulsification system that includes a static mixer and a high pressure homogenizer in series, in both loop arrangement and single pass. The proposed system allows the production at high rates, of high solids content miniemulsions containing a very viscous organic phase.

1. Introduction

Miniemulsion polymerization allows the synthesis of materials that are not obtainable by means of other techniques such as conventional emulsion polymerization. The reason is that in miniemulsion polymerization particles are mainly formed by droplet nucleation [1,2]. These materials include polymers from highly water insoluble monomers [3,4], polymer encapsulated inorganic materials as titanium dioxide [5] and other pigments and components [6], the controlled free radical polymerization (CRP) [7] and waterborne hybrid polymer particles [8–10].

Industrial production of waterborne polymer dispersions is mainly carried out by conventional emulsion polymerization in 15–60 m^3 reactors operating in a semicontinuous mode. Most likely, industrial scale miniemulsion polymerization will also be carried out in semicontinuous operation. The process time needed for each semicontinuous operation is typically in the range of 3–6 h, which is largely determined by the heat removal rate of the reactor. In comparison with conventional emulsion polymerization, miniemulsion polymerization involves an additional operation: the miniemulsification process. This process is the bottleneck for the industrial implementation of miniemulsion polymerization because, apart from the additional investment, the time required to prepare a miniemulsion of the required characteristics (droplet size, stability) may be that long that leads to an unacceptable increase of the time needed for the whole semicontinuous process. The problem is aggravated by the fact that in many practical applications, miniemulsification of highly viscous organic phases is required, and the higher the viscosity the longer the miniemulsification time. In the miniemulsification process, the coarse emulsion is subjected to a high energy source in order to break the droplets up to the submicron size (80–200 nm) with a narrow enough droplet size distribution. The methods/devices used in a lab scale to produce low droplet size miniemulsions are sonication [11,12], rotor–stator [13], membrane emulsification [14,15], static mixing [16,17] and high pressure homogenization [18–20]. Among these devices, high pressure homogenization (HPH) is the most suitable method to obtain small droplet sizes (even for systems dealing with highly viscous organic phase [21,22]) and it seems to be the most promising choice for industrial scale as large capacity (21,000 L/h at 400 bar) HPHs are available [23]. These devices would be able to perform industrial scale miniemulsifications in a reasonable time, provided that the required droplet size could be achieved in a single pass. However, a single pass of the coarse emulsion (droplet size > 1 μm) through the HPH may not be enough to obtain the desired miniemulsion droplet size (80–200 nm), mainly when the viscosity of the dispersed phase is high and/or small droplet sizes (≈100 nm) are desired. The use of two or more HPHs in series would likely allow achieving small droplet sizes in shorter times, but this option substantially increases the investment.

Another alternative is to use a single HPH in a loop arrangement. Working in a discontinuous way (Fig. 1), it has been demonstrated that, because of the mixing in the storage tank, 5–6 passes are typically needed to get to a small droplet size with a narrow droplet size distribution [21,22]. A pass is defined as the time needed to process the volume of the storage tank, V_s; namely V_s/Q_{HPH}, where

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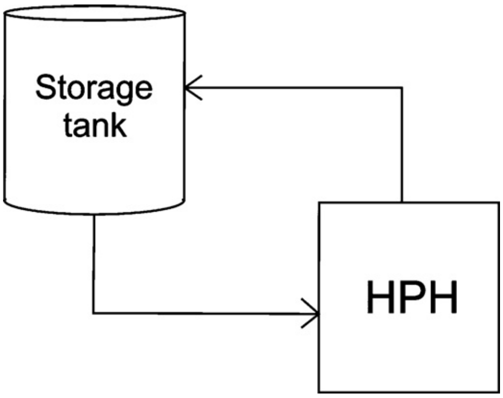

Fig. 1. HPH in discontinuous loop arrangement.

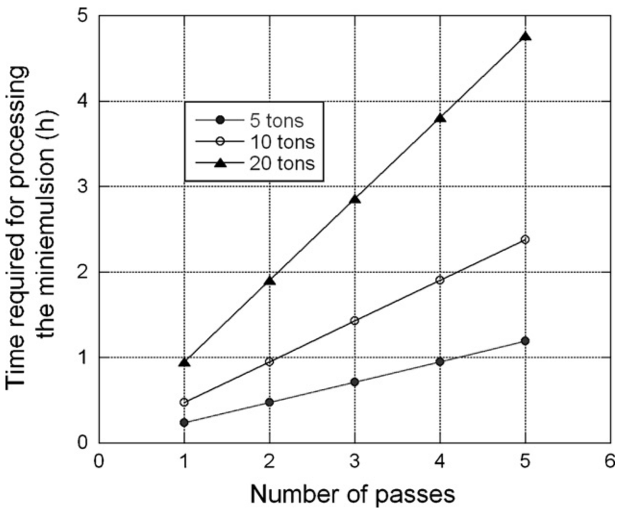

Fig. 2. Time required to process different production amounts, in a HPH with high processing flow (21,000 L/h at 400 bar) at different number of passes.

Q_{HPH} is the flow rate in the HPH. For industrial scale reactors, this leads to unacceptable long process times because the time needed to homogenize a certain amount of miniemulsion increases with the number of passes needed to achieve the desired droplet size (Fig. 2).

Although its use has not been reported, the continuous miniemulsification using a HPH in a loop arrangement (Fig. 3) may be a better option for the industrialization of this process. In this arrangement, the droplet size should decrease when increasing the recirculation ratio (Q_{HPH}/Q_1, being $Q_{HPH} > Q_1$). At first sight, this arrangement is affected by the same limitations than the discontinuous loop arrangement. Nevertheless, there may be a way to improve the efficiency of the continuous miniemulsification. In the case of the discontinuous loop miniemulsification, it has been reported that the decrease of the droplet size of the coarse emulsion substantially reduces the number of passes needed to achieve the minimum droplet size [21,22]. This may also be beneficial in the continuous miniemulsification. In the case of the discontinuous loop miniemulsification, the decrease of the droplet size should be achieved before the miniemulsification process, which adds another operation to the process. In addition to using mechanical means, phase inversion could be used to reduce the size of the coarse emulsion [24,25]. The production of small enough droplets by this procedure generally involves long stepwise addition times of the aqueous phase into the organic phase [26,27] and this may counteract the advantages of using a small size coarse emulsion. Therefore, for the continuous process, phase inversion is not a choice unless a fast procedure is developed.

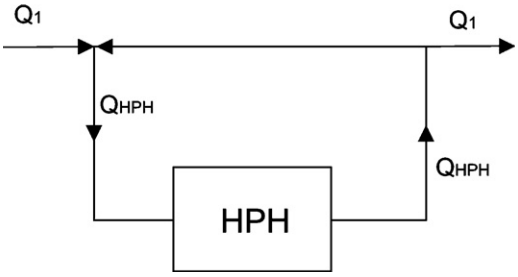

Fig. 3. HPH in a continuous loop arrangement.

Static mixers may be used to reduce the droplet size of emulsions, and amongst the other scalable devices, they represent a good alternative to the classical homogenization techniques [28] in terms of energy cost [29,30]. Ouzineb et al. [17] used two polyacetal static mixers in a continuous batch loop to obtain stable emulsions. However, the time needed to achieve the minimum size (297 nm with narrow DSD, at 28 wt% solids content) was long. Based on this work, the performance of static mixers in acrylics dispersions was further studied by Farzi et al. [31] and El-Jaby et al. [32,33] either using several numbers of mixing elements of different dimensions or using in situ surfactant generation (170 nm at 43 wt% solids content). These works demonstrated the feasibility of using mixers in generating miniemulsions with low viscosity organic phases. However, their performance in high solids content systems (≥50 wt%) and their usefulness for preparing miniemulsions with higher viscosity organic phase remains to be demonstrated.

In this work, an efficient continuous miniemulsification procedure was developed by combining a static mixer and a HPH in a loop arrangement. For the proof of concept, the formation of a 50 wt % solids content alkyd-rich acrylic miniemulsion was chosen. In this system, the organic phase contained 50% of alkyd resin dissolved in the acrylic monomers. This is a demanding case because of both the high viscosity of the miniemulsion (due to the high solids content) and the high viscosity of the organic phase (because of the presence of a high concentration of alkyd resin). For this system the relative viscosity, η_d/η_c, i.e., the ratio of the viscosity of the dispersed phase (η_d) and the continuous phase (η_c), is equal to 8 [22].

2. Experimental

2.1. Materials

Technical grade monomers, methyl methacrylate (MMA) and butyl acrylate (BA), supplied by Quimidroga, and acrylic acid (AA) and stearyl acrylate (SA) supplied by Aldrich were used without purification. SA was used both as monomer and costabilizer. A costabilizer is a low molecular weight water insoluble compound that enhances miniemulsion stability by avoiding Ostwald ripening. The alkyd resin used was supplied by Nuplex Resins with the commercial name of SETAL 293-XX (S293). This is a relatively hydrophobic resin (acid value = 11 mg KOH/g). Dowfax 2A1 (alkyldiphenyl oxide disulfonate, Dow Chemicals, 45 wt% solution) was used as emulsifier. Sodium bicarbonate ($NaHCO_3$, 99.7%, supplied by Riedel-de Haën) was employed to control the miniemulsion viscosity by reducing the intense electrostatic interactions between droplets. Deionized water was used throughout the work.

The formulation used in all experiments was as follows: acrylic monomers (BA/MMA/SA/AA = 47.6/47.6/3.8/1 wt%); alkyd resin 50 wt % based on the organic phase (wbop); $NaHCO_3$ at a concentration of 0.039 M in the water phase; and emulsifier at 6 wbop%. The organic phase content was 50 wt% in all cases.

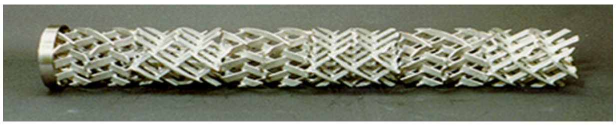

Fig. 4. Detail of the SMX static mixer.

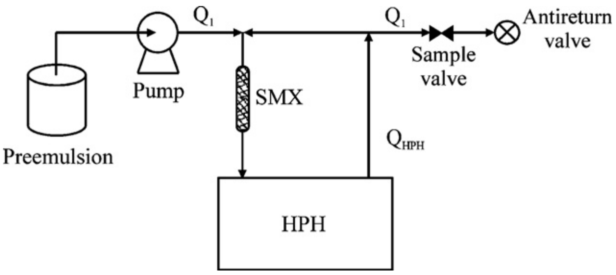

Fig. 5. Experimental set-up.

2.2. *Miniemulsification set-up*

A 19 mm length and 3.2 mm in diameter SMX DN3 static mixer (Sulzer Chemtech, Fig. 4) was used because it is particularly well suited for handling mixtures that exhibit large viscosity differences [34]. The SMX mixers consist of intermeshing and intersecting bars, which are positioned at 90° to the axis of the direction flow. The mixer was placed flush within a stainless steel tubular housing and was directly connected to a two valves PANDA 2 K Niro-Soavi homogenizer (Fig. 5).

The organic and the aqueous phases were mixed with magnetic stirring to produce a coarse preemulsion (droplet size in the order of thousands of nm). Then, the preemulsion, which was kept in a stirred vessel, was pumped through the system represented in Fig. 5. Because the flow rate in the HPH (Q_{HPH}) was greater than the flow rate in the pump (Q_1), the coarse preemulsion was mixed with the miniemulsion circulating within the loop and passed through the static mixer before entering into the HPH. The aim of this arrangement is to reduce the droplet size of the coarse emulsion before entering to the HPH. The recirculation ratio (R) was defined as Q_{HPH}/Q_1. The flow rate in the homogenizer was Q_{HPH} = 167 mL/min and it was kept constant, whereas the flow rate in the pump (Q_1) could be varied from 20 to 100 mL/min. The residence time (τ) was defined as the ratio between the volume of the recirculation loop (constant in all cases = 122 mL), and the pump flow rate ($\tau = V_s/Q_1$). The droplet size was measured at the exit of the system, taking samples every 2 min.

2.3. *Characterization*

Monomer droplet size (d_d) and droplet size distribution (DSD) were measured by dynamic light scattering, in a Zetasizer Nano Z (Malvern Instruments) immediately after sampling. The values given are *Z*-average values obtained through cumulants analysis and the droplet size distributions shown are intensity distributions.

Preliminary tests were carried out measuring the droplet size by diluting the sample in both deionized water and in deionized water saturated with monomer (to avoid monomer transfer to the aqueous phase). No significant differences between both measurements were observed. Therefore, droplet sizes were measured by diluting the samples in deionized water.

3. Results and discussion

In the miniemulsification using high pressure homogenizers, the droplet size is the result of two consecutive processes: droplet break up and coagulation, and the final droplet size is determined by the mechanism giving the largest size [21]. Droplet break up increases with the pressures used in the valves of the HPH. Coagulation decreases with the surfactant concentration. For a given concentration of surfactant, at low pressures, the droplet size decreases as pressure increases. However, there is a critical pressure beyond which no further decrease in the droplet size is observed. This is due to the fact that the droplet size is controlled by the coagulation, namely, by the concentration of surfactant in the system. Some preliminary experiments were carried out to determine the critical pressures for the system under study, and it was found that the critical pressures were 410 bar and 41 bar in the first and second valve, respectively. These pressures were used throughout the work.

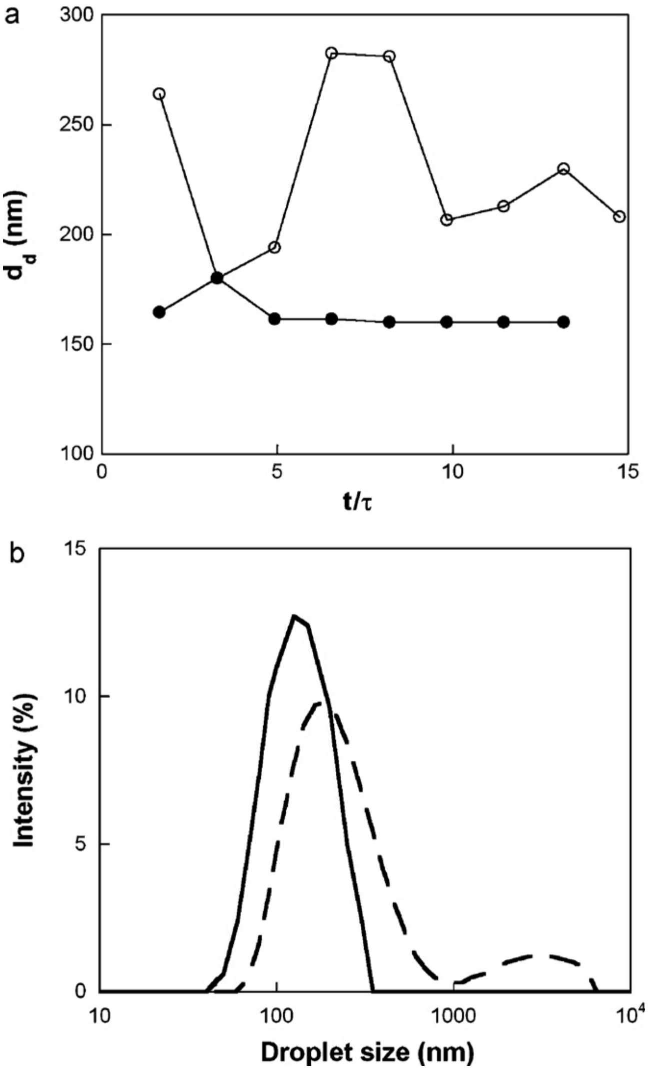

Fig. 6. (a) Droplet size evolution and (b) final droplet size distribution (•,—) with SMX and (○,––) without SMX. R = 1.66.

3.1. *Effect of the presence of the static mixer on the droplet size*

The effect of the presence of the static mixer within the circulation loop (Fig. 5) on the droplet size of the miniemulsion was studied at low (R = 1.66) and high (R = 8.3) recirculation ratios. It is worth pointing out that the production rate achieved by a given HPH is inversely proportional to the recirculation ratio. Figs. 6 and 7 present the time evolution of the average droplet size and the droplet size distribution under steady state conditions for R = 1.66

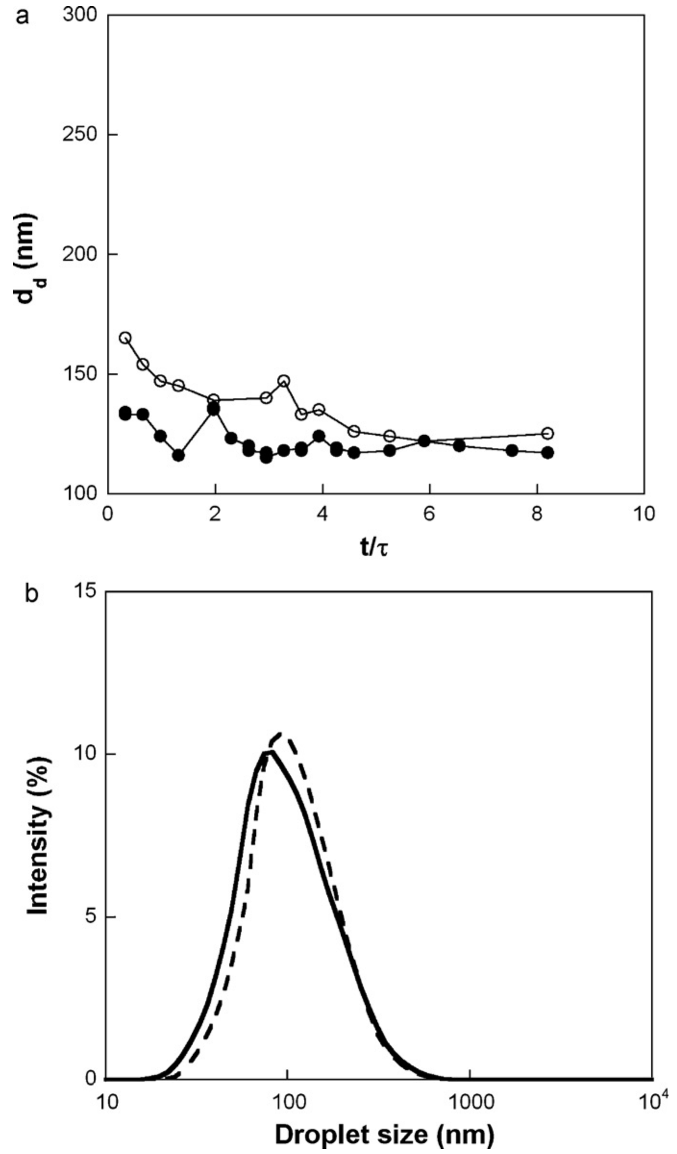

Fig. 7. (a) Droplet size evolution and (b) final droplet size distribution (•,—) with SMX and (o,−−) without SMX. R = 8.3.

and R = 8.3, respectively. It can be seen that the system requires some time to reach the steady state conditions. The reason was that the loop was initially filled with deionized water. Figs. 6 and 7 show that the static mixer helps to significantly reduce the droplet size when a low recirculation ratio (Fig. 6) was used, but at high recirculation ratios, the effect of the static mixer was marginal (Fig. 7). At R = 1.66, about 36% of the coarse emulsion passes only once through the HPH, and therefore, the droplet size of the coarse emulsion at the entrance of the HPH strongly affected the droplet size achieved at the exit of the system. When no static mixer was used, large droplets entered directly into the HPH. In the HPH, the droplets are elongated at the entrance of the high pressure valve and broken up by the turbulence caused at the exit of the valve [21]. Because plenty of new surface area is created, if there is not enough surfactant or the surfactant does not diffuse fast enough, the newly created droplets may not be well covered by the surfactant and droplet coalescence occurs. Therefore, the breakage of all droplets and their stabilization is difficult to achieve in a single pass. This is the reason for the large droplets (>1000 nm) found when no static mixer was used. In the presence of static mixer, which reduces the size of the droplets entering into the HPH, smaller droplets (160 nm vs. 208 nm) with a narrower droplet size distribution (PDI = 0.14 vs. PDI = 0.3) were obtained.

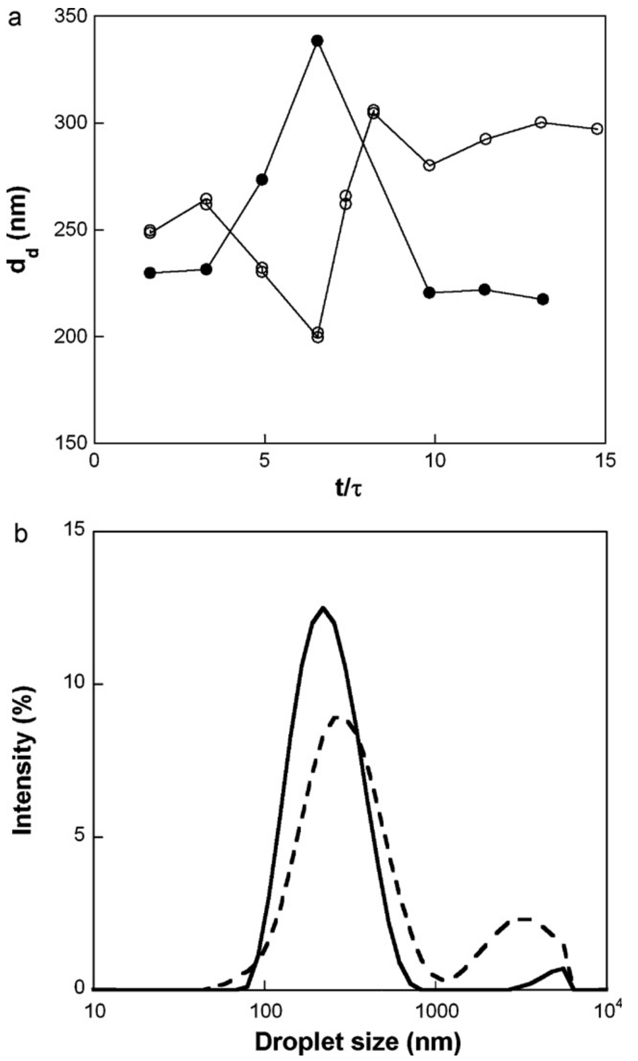

Fig. 8. Effect of the static mixer on (a) the droplet size evolution and (b) final droplet size distribution in a single pass miniemulsification, (•,—) with SMX a and (o,−−) without SMX.

At R = 8.3, most of the droplets passed several times through the HPH, and hence the contribution of the static mixer to the decrease of the droplet size was marginal (Fig. 7a). In this case, narrow droplet size distributions were obtained (Fig 7b). Comparisons between Figs. 6 and 7 shows that droplet size decreased as the recirculation ratio increased (but productivity decreased).

3.2. Single pass miniemulsification

The results presented in Figs. 6 and 7 show that the increase in production (decrease of the recirculation ratio) results in a broader droplet size distribution with larger average size, which often is considered to be a miniemulsion of less quality. The required quality of the miniemulsion (droplet size and size distribution) may depend on the application and in some cases broad droplet size distributions with a large average size may be acceptable. Therefore, it is interesting to explore the effect of using the static mixer at maximum production rate, namely, using single pass miniemulsification with no recirculation. Fig. 8 shows that placing the static mixer before the HPH is beneficial because as compared with using only the HPH, smaller droplets (217 nm vs. 297 nm) and a narrower

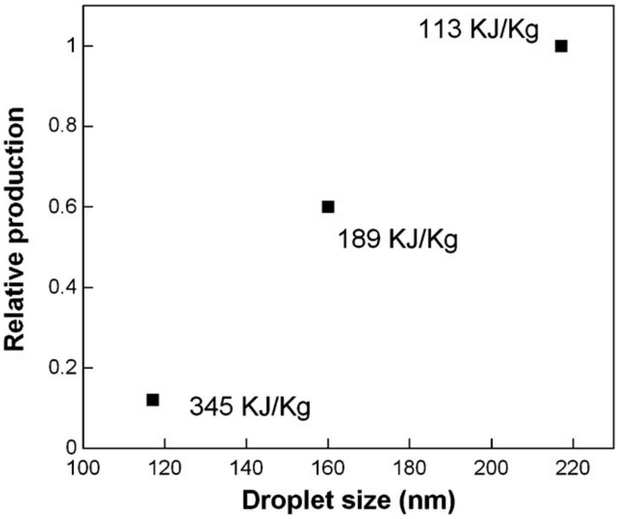

Fig. 9. Relationship between the production rate and the droplet size in systems that used a static mixer in combination with a HPH.

droplet size distribution (PDI = 0.2 vs. PDI = 0.35) were obtained, achieving the same production.

Fig. 9 shows the relationship between the production rate and the droplet size in systems using a static mixer in combination with a HPH, together with the relative energy input required in each case (calculated as the power consumed times the flow). In this plot, the relative production is defined as the production rate divided by the maximum production rate, which is the flow rate in the HPH. Fig. 9 shows that lower production at a higher relative energy consumption is the price for obtaining smaller droplet sizes. Compared to other miniemulsification devices such as a rotor–stator, a sonifier or a static mixer, the energy consumption of this system falls between the high energy requirements of the rotor–stator (average of 400 kJ/kg to obtain 190 nm droplets in a low viscosity system), those of the ultrasonication (around 300 kJ/kg to obtain 110 nm droplets in a low viscosity system) and the low energy requirements of the static mixers alone (160 kJ/kg to obtain 170 nm droplets in a low viscosity system) [32].

4. Conclusions

This work is an attempt to overcome the bottleneck for the industrial implementation of miniemulsion polymerization, which is the miniemulsification itself. For this purpose, the feasibility of using continuous miniemulsification to achieve a high production of miniemulsions with small size and narrow droplet size distribution was explored. For the proof of concept, a 50 wt% solids content alkyd-acrylic (50/50, wt/wt) miniemulsion was chosen. This is a demanding system because of the high viscosity of both the miniemulsion and the organic phase. The continuous miniemulsification system included a static mixer placed at the entrance of a high pressure homogenizer (HPH).

It was found that in the presence of the static mixer, whose main role was to reduce the droplet size of the preemulsion fed to the HPH, miniemulsions with smaller droplets and narrower droplet size distributions were obtained. It was also found that an increase in the production, which for a given HPH in a loop arrangement means a lower recirculation ratio, resulted in an increase in the size of the miniemulsion droplets. The use of a static mixer was beneficial even when single pass miniemulsification was used, namely, for maximum production. Thus, miniemulsions with a relatively small droplet size (≈200 nm) were obtained under these conditions. These results indicate that the continuous miniemulsification using a combination of a static mixer and a high pressure homogenizer may facilitate the industrial implementation of miniemulsion polymerization, at moderate energy consumptions.

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