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Development of an efficient emulsification process using miniaturized process engineering equipment[☆]

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ABSTRACT

In many cases, emulsification with highly viscous materials is performed batchwise using CSTR systems being heated in a conventional way. A more efficient process may be continuous emulsification using miniaturized equipment like micro heat exchangers and micro mixers. The advantages of such systems like reduced diffusion length and increased heat transfer capacities are widely known, as well as the disadvantages, namely increased pressure losses. The present study describes the development of a continuous oil-in-water emulsification process using microstructured devices. Based on the analysis on the rheological properties, several suitable micro mixer systems were developed and tested. Some experiments under different conditions have been carried out to study the influence of different parameters to the emulsification quality referred to the droplet size. The studied range of the total mass flow rates was from roughly 4 to 9 kg/h. The oil content varied from 52 to 72%. The oil passage was heated to 120 °C and the water passage to 88 °C. Characterized droplet sizes such as Sauter diameter were used as the main quality criteria. Results show that the higher the flow velocities are, the smaller the Sauter diameter of the emulsion droplets becomes. However, the pH of the products needed to be limited between 7 and 8. In this study, two water solutions were prepared, 1% (for S2–S9) or 2% (for S1) ammonia solution (25 vol.%) was added to the water phase. Large Sauter diameters or phase separation may be faced if the pH is not adjusted correctly. A high oil content or low emulsifier content may also reduce the quality of emulsions.

The combination of micro mixer emulsification units and micro heat exchangers was tested. Two micro heat exchanger devices have been tested to heat the water and to quench the emulsion and, therefore, to stabilize the emulsion. Both are 8 cm^3 active volume cross flow heat exchangers (each passage has a section area of $200 \times 200\ \mu m^2$/channel × 68 channels/foil × 34 foils). The viscosity of the generated emulsions was around 74 mPa s at 25 °C, measured at a shear rate of $100\ s^{-1}$. It shows only a weak temperature dependence. For example, it was reduced by 5 mPa s by increasing the temperature from 25 to 45 °C.

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The quenching shows positive effects to the emulsion stability by preventing droplet collapses, which, thus, normally take place at higher temperature. The integration of miniaturized heat exchanger and mixer devices into a system led to a very efficient emulsification unit reducing the total energy consumption of the process considerably.

1. Introduction

Batch production is flexible and suitable for versatile production schemes, it also shows advantages for high viscosity fluid, or a high solid content. It is suited for small or medium production capacities regarding related investment and operating costs (Grundemann et al., 2009), however, it is still profitable by transfer to micro process technology (Grundemann et al., 2012).

Continuous productions with microstructured devices can reduce hold-up, improve local energy dissipation, maintain constant product quality, etc., and attract much research attention for industrial application. Some successful examples have been reported (Garza-Garcia et al., 2013; Grundemann et al., 2009). Continuous emulsification processes have been continuously investigated since the last decade. The relevant processes work well due to the presence of the key devices such as membrane systems (Lloyd et al., 2015; Zawala et al., 2015), rotor–stator-systems (Han et al., 2012; Maa and Hsu, 1996), high pressure homogenizers (Floury et al., 2000; Schultz et al., 2004), mini (Belkadi et al., 2015) or microchannel systems (Khalid et al., 2015a,b,c; Liu and Yobas, 2015).

In most of the processes mentioned above, micro devices are amongst the key components for continuous processing. Generally, the same laws that describe the flow at a macro scale govern fluid flow in micro scale (Capretto et al., 2011; Schuchmann et al., 2009), but miniaturization provides some unique features. Micro devices are designed to give high surface-to-volume ratio, minimize the transport length, facilitate step control for exothermic or endothermic reaction, etc.

In this work, a currently running industrial batch process is transferred into a continuous process for producing oil-in-water emulsion paint. The influence of the parameters such as pH, oil content, flow rate and cooling effects was studied regarding the products quality.

2. Experimental setup

An emulsion is a dispersed system of two immiscible fluids. In this study, highly viscous oil was used. In the current batch process, the oil is heated up to 80 °C and then mixed with water, emulsifier, and pH adjusting agent. Additional to the energy consumption from the heating step, the mechanical stirring requires high electricity power input to reach high shear stresses. Apart from the convenience in operation brought by the continuous process, it is also expected that the energy consumption from the whole process can be reduced.

In a micro mixer, the two phases can be mixed in short time and limited space and the shear forces can be generated on the interfaces (Schuchmann et al., 2009), thus, generally micro mixer may give better defined shear stresses due to his homogeneous flow velocity. Some research related to micro mixer emulsification was given, especially in the food industries (Köhler et al., 2007, 2008, 2009) and cosmetics industries (Matsuyama et al., 2011). However, rarely highly viscous oil was employed for applications of micro devices. In a previous study, the rheological properties of the oil, the emulsifier, and their combination were studied (Li et al., 2013). The oil used in this study is a high viscosity product and its physical property is listed in Table 1.

The mixing order of the four components, water, oil, emulsifier and pH adjusting fluid was tested in a small-scale batch process. There was no evident difference found from the products created by different mixing order, i.e., the order of mixing steps is free of choice.

Fig. 1 shows the schematic for the continuous process. Due to the small amounts of the emulsifier and the pH adjusting agent, it would be much easier solve it directly in the low viscosity phase (water). Thus, in this flow diagram, only one mixing step is included. For at this time we had no efficiently working heat exchanger for the oil, the oil was heated in the container (~120 °C). The water was heated up with a cross-flow micro heat exchanger (μHX1) (Brandner et al., 2006) (to 88 °C). A V-type micro mixer (μMixer) was placed for creating high shear forces to the interfaces. A second micro heat exchanger (μHX2) was placed after the emulsification step to quench so that the coalescence could be reduced.

Micro channels were created on long metal foils by using precision milling. By diffusion bonding, the specially arranged micro channels stack was formed (Fig. 1 bottom left). A metal housing was developed to suit to the mixer stack and supply the inlet channels, mixing chamber, and outlet connection (Fig. 1 top right). Each channel has a cross sectional area of $100 \times 70\,\mu m^2$. Two foils were designed for the oil passage with 25 channels each, one foil for water passage with 5 channels only. The larger section area for oil passage aimed to reduce pressure losses, and the smaller section area was designed for the water passage in order to keep high kinetic energy.

Two micro heat exchangers of $8\,cm^3$ active volume (Brandner and Schubert, 2005) were used in this process. The micro channels were made by using the same process as for micro mixer manufacturing. Then, the channels of two passages were crosswise arranged and each foil of one passage is sandwiched by the other passage (Fig. 1 bottom left). Each passage has 34 foils, which contains 68 micro channels. The channel cross sectional area is $200 \times 200\,\mu m^2$, channel length is 2 cm. Four Swagelok male connectors were welded as fluid inlets and outlets. The pumps used in this work were displacement pumps from LEWA. The pH meter was the model C231 from Consort.

Table 1 – Physical properties of the oil used in this work (at 20 °C).

Viscosity (Pa s) (at shear rate $10\,s^{-1}$)	123
Density (kg/m^3)	1100
Thermal conductivity (W/(m K))	0.17
Specific heat capacity (kJ/(kg K))	1.84