

Comparative studies on processing facilities with special regard to the new Co-Twister system

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1. Introduction

In common used processing plants the homogenization process can be mainly influenced by varying the speed of the rotor with respect to the fixed stator or by concrete design of rotor and stator.

However, the maximum shear rate is limited by the dimensions of the rotor/ stator system, and the maximum rotating speed of the drive motor.

In this study a SYMEX CML 30 with a special designed homogenizer, the so-called "Co-Twister" system, has been used:



Fig. 1: Processing facility - Symex CML 30

Similar to many common used processing facilities this machine is equipped with CIP-nozzles and a slow rotating agitator.

In this picture a top-view of the homogenizer unit can be seen, too. The so-called Co-Twister is mounted below the bottom of the vessel and has a unique character.

Nominal Vialer Vessel	30L	Agitator rotation speed	18-162 rpm (-0.2 to 7 m/s)
Minimum Batch size	5L	Agitator (Power Consumption)	1.1 kW
Maximum Batch size	45L	Rotor (Power Consumption)	12.0 kW
Pressure (vessel)	0 to 3 bars	Dynamic Stator (Power Consumption)	9.8 kW
Pressure (double jacket)	1 to 5 bars	Rotor revolutions	300 - 9000 rpm
Temperature (vessel)	up to 133.5°C	Dynamic Stator revolutions	-9000 rpm to +9000 rpm
Temperature (double jacket)	up to 107°C	Resulting shear rate	0 to 57 m/s
Product contacting surfaces	Stainless steel	Pump rate (Flow*)	1800 l/min (at 10m/s)
Product connecting surfaces	316 Ti resp. 316 L		

Fig. 2: Processing facility - Technical data

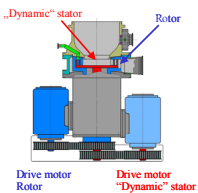


Fig. 3: Co-Twister unit

The maximum shear rate (57 m/s) of this Co-Twister system is much higher compared to conventional processing plants.

2. Investigation scheme

In principle always the same manufacturing process was performed during the series of experiments. At different temperatures model formulations were homogenized with different shear rates for a certain period of time. During and at the end of the production process the particle size distribution of the product was controlled via laser diffraction measurements.

In a pre-study the problem to detect the droplet size during homogenization and cooling was solved in that way, that a tube was directly brought into the vessel and performed the sampling with the aid of a peristaltic pump. After the necessary diluting the samples were characterized.

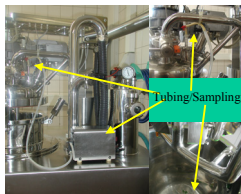


Fig. 4: Sampling

2.1 Model formulations

	„L1“- Formulations	„L2“- Formulations	„P“-Formulations
Medium chain triglycerides	10,0%	20,0%	10,0%
Lecithin	1,0%	2,0%	----
Poloxamer 188	----	----	3,5%
Glycerol	2,5%	2,5%	2,5%
Phenoxyethanol	1,0%	1,0%	1,0%
Water	ad 100,0%	ad 100,0%	ad 100,0%

Fig. 5: Model formulations

Special acknowledgements go to the company Phospholipid GmbH for supporting experiments by providing Phospholipon 90G

2.2 Manufacturing process

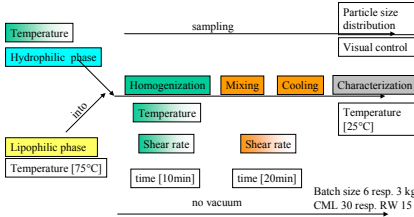


Fig. 6: Manufacturing process for the production of highly dispersed oil-in-water (O/W) emulsions

2.3 Critical process parameters

The critical process parameters were investigated using a 2³ - factorial design, which represents a rational method to study production processes. The effects of the tested parameters were calculated and the results were statistically evaluated for significance.

2.4 Comparative studies

Comparative studies using a BECOMIX RW 15 followed. The aim of these experiments was to clarify if Co-Twister technology allows to imitate common used processing devices, although having a different construction respective design of rotor and stator. Therefore emulsions were prepared at different shear rates in the two machines and the resulting particle size distributions were determined and compared. To finish the comparison experiments with a high pressure homogenizer (Type Panda, Co. Niro Soavi) were included additionally.

3. Results

3.1 Critical process parameters

The lecithin based "L1" and the poloxamer based "P" emulsions were prepared with the SYMEX CML 30 at different temperatures and moderate conditions in terms of shear rate and rotor/rotor revolutions (5000 rpm [22 m/s]).

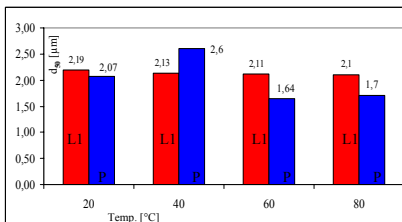


Fig. 7: Droplet size of different batches "L1 and P" produced under moderate manufacturing conditions

In order to judge which of the so far investigated parameters are decisive an investigation according to the 2³ factorial design was performed. The parameters investigated were

- A) Shear rate during homogenization (57 m/s or 22 m/s)
- B) Shear rate during cooling (57 m/s or 22 m/s)
- C) Temperature during homogenization (80°C or 20°C)

A statistical evaluation (d_{50} [µm]) according to Yates finished the study.

Batch	A Shear rate Homogenization	B Shear rate Cooling	C Temperature	d_{50} Lecithin	d_{50} Poloxamer
1	-	-	-	2,38	1,96
2	+	+	-	1,44	1,56
3	+	-	-	2	0,99
4	+	+	-	1,31	1,01
5	-	-	+	2,1	1,7
6	-	+	+	1,45	0,99
7	+	-	+	1,97	1,49
8	+	+	+	1,44	0,97

Fig. 8: Investigation according to 2³ factorial design

Evidence is given, that in the case of the lecithin based emulsions the effect of the shear rate during homogenization and during cooling is different from zero, but no investigated parameter has a statistical influence on the poloxamer based emulsions. Therefore all further investigations were based on lecithin formulations.

The homogenizer speed was found to have a high impact on the product quality. A strong dependency of the particle size on the shear rate and the formulation could be seen.

3.2 Comparative studies

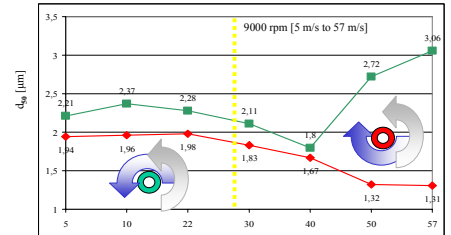


Fig. 9: Droplet size of different batches "L1 and L2" produced on CML 30 with shear rates from 5 to 57 m/s

The minimum droplet size for the L2 formulations is accomplished at a rotation speed of approx. 40 m/s in contrast to the L1 formulations containing 10 % lipids where the optimum shear rate is about 57 m/s.

Comparative studies using Becomix RW 15 were restricted through design of rotor and stator to 22 m/s in maximum.

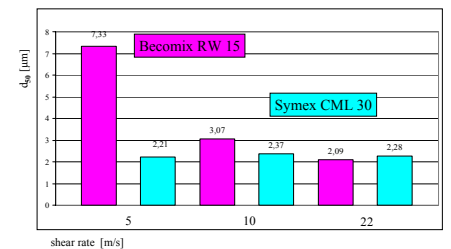
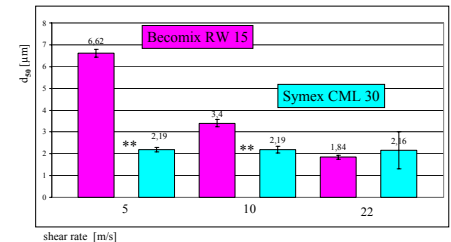


Fig. 10: Droplet size of different batches "L2" produced on CML 30 resp. BECOMIX RW 15 (shear rates from 5 to 22 m/s)



** : highly significant according F/t - test with $\alpha = 0,05$

Fig. 11: Droplet size of different batches "L1" (n = 6 each) produced at CML 30 resp. BECOMIX RW 15 (shear rates from 5 to 22 m/s)

The processing devices are different concerning their homogenization efficacy: d_{50} values for formulations produced on SYMEX CML 30 are even at lowest rotating speed comparable to the in minimum reachable d_{50} values on BECOMIX RW 15. On the other hand identical droplet sizes are available at certain "pump rate"/ "shear rate" - ratios. High pressure homogenization leads to droplets which are roughly in the same size range like these produced on CML 30 but distinctly smaller than those produced on the RW 15.

	L1	L2	Formulation
Pressure [bar]			
50	1,24	1,12	
100	1,15	1,11	
200	1,17	1,16	
	d_{50} [µm]		

Fig. 12: Droplet size of different batches "L1 and L2" produced with Panda high pressure homogenizer

4. Conclusions

Summarizing no general rule was found to prefer one of the homogenizer over the other. No full explanation of the differences in particle size at identical rotor speed was found but it is possible that the different constructions of the rotor/stator-units lead to a different homogenization efficacy. Nevertheless CML 30 and the high-pressure homogenizer are able to produce droplets in the 1 µm range and thus give comparable results.

References

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