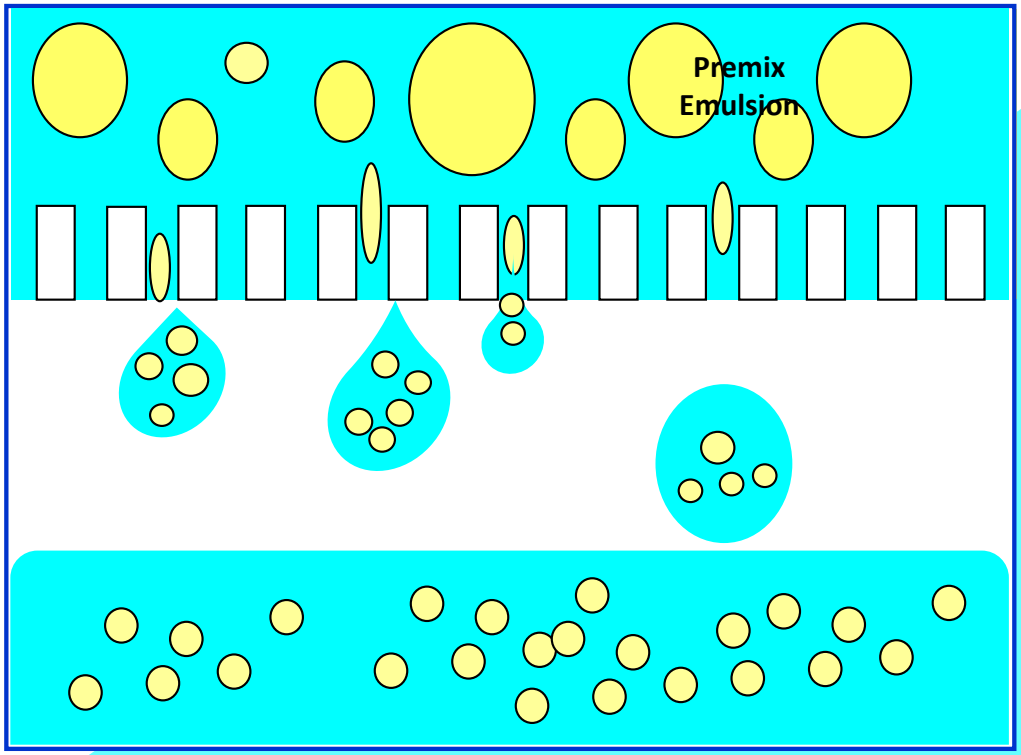
The effects of emulsification conditions on emulsion size and distribution (for High speed emulsification)

We confirmed how the following conditions affect the emulsion on high speed emulsification.

1. Premix emulsion distribution
2. Pass speed of SPG membrane (= Pass Pressure)
3. Number of pass

High speed emulsification (Premix emulsification)

"High-speed emulsification" refers to an emulsification method in which the emulsion is finely divided and monodispersed by passing the premixed (coarsely emulsified) emulsion liquid through an SPG membrane at a flow rate of a certain level or more.
Compared to "Direct emulsification", it is suitable for actual production because a large amount of emulsion can be prepared in a short time.



< Feature >

Emlsion size : SPG membrane pore size or less

Emulsification time : very Fast

It can be prepared 100 to 1,000 times faster than “Direct emulsification”

Monodispersed emulsion

Adjustment of emulsion particle size

Continuous production is possible

Use Device for test
: External pressure micro kit (MG-20)


外圧式マイクロキット
（MG-20）

Emulsified composition
: O/W (Oil in Water) emulsion
Dispersion phase (O) = Kerosene 3ml
Continious phase (W) = 0.5wt% SDS solution 7ml
＊SDS＝ sodium dodecyl sulfate (Hydrophilic surfactant)

Emulsion particle size measuring device
: Laser diffraction/scattering particle size analyzer
; Shimadzu Inc., SALD-2000

Average	: Average particle size
Mode	: Most frequent point (Most frequent particle size)
Median	: Particle size at which the integrated particle amount (volume basis) becomes 50%.
10%D	: Particle size at which the integrated particle amount (volume basis) becomes 10%.
90%D	: Particle size at which the integrated particle amount (volume basis) becomes 90%.
90%D/10%D	: Represents the width of the distribution. The smaller the value, the monodispersion.

1. The effects of “Premix emulsion distribution”

Using a premix emulsion prepared by ① polydispersion, ② slightly monodispersion, and ③ monodispersion, the distribution of the emulsion in permeation membrane emulsification was confirmed.

Sample		Average (μm)	Mode (μm)	Median (μm)	10%D (μm)	90%D (μm)	90%D /10%D
① Polydispersion	Premix	80.584	91.146	84.451	38.541	160.081	4.2
	After High speed emulsification	**12.235**	**11.354**	**12.024**	**7.811**	**17.768**	**2.3**
② Slightly Monodispersion	Premix	34.860	32.170	32.418	21.417	57.151	2.7
	After High speed emulsification	**11.858**	**11.354**	**11.675**	**8.432**	**15.516**	**1.8**
③ Monodispersion	Premix	42.744	39.619	42.443	33.271	53.930	1.6
	After High speed emulsification	**11.561**	**11.354**	**11.658**	**8.492**	**15.326**	**1.8**

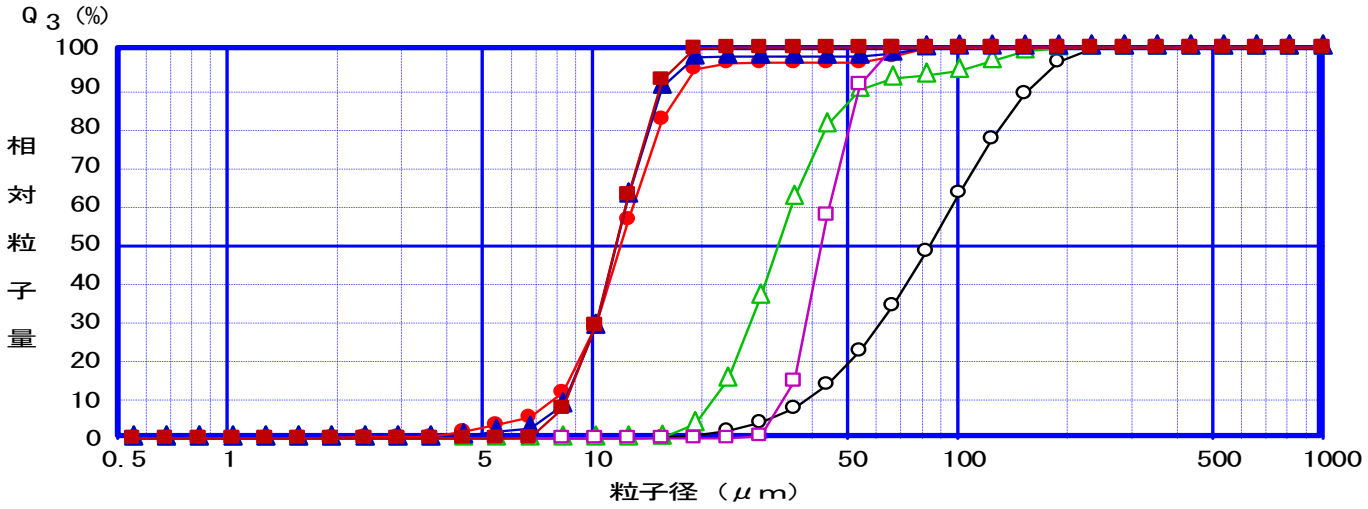
The narrower the premix distribution, the emulsion becomes monodispersed after high speed emulsification.

< Premix preparation method >
① Polydispersion ：Stirring with Stirrer. 400rpm, 1min
②Slightly Monodispersion： Emulsify at high speed. ①Polydispersed emulsion is prepared at high speed emulsification with a 30 μm SPG membrane.
③ Monodispersion ： SPG Direct emulsification. SPGmembrane 11 μm

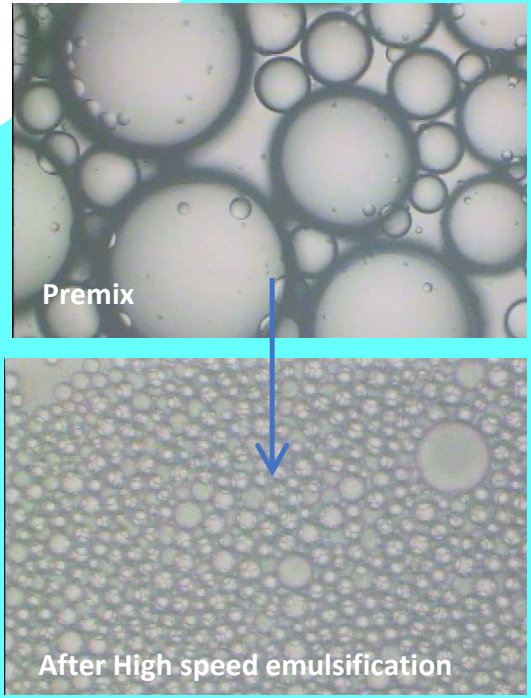
< Condition of high speed emulsification >
SPG membrane pore size = 11 μm, Pass Pressure = 40 kPa

1. The effects of “Premix emulsion distribution

1. ① Premix (Poly) 2. ① after SPG (Poly) 3. ② Premix (Slightly Mono) 4. ② after SPG (Slightly Mono)	5. ③ Premix (Mono) 6. ③ after SPG (Mono)	Sレベル : 0 分布関数:無 Dシフト : 0



<Polydispersion >



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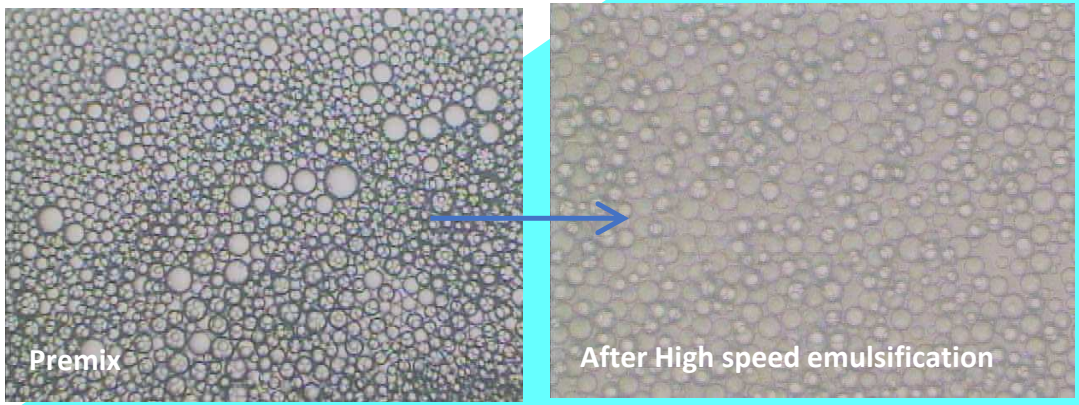
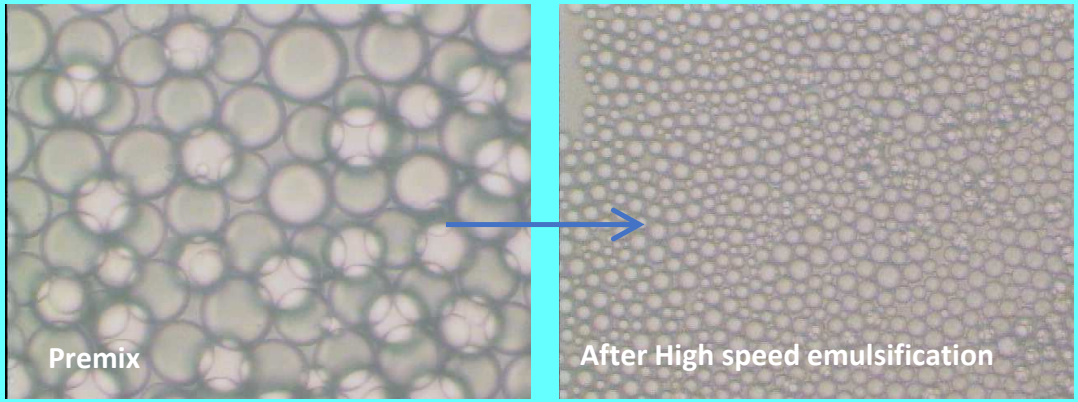


< Monodispersion >



2. The effects of “Pass speed of SPG membrane (= Pass Pressure)”

The emulsification (pass) pressure was set to ①20 kPa, ②40 kPa and ③80 kPa, respectively, and the distribution of the emulsion was confirmed.

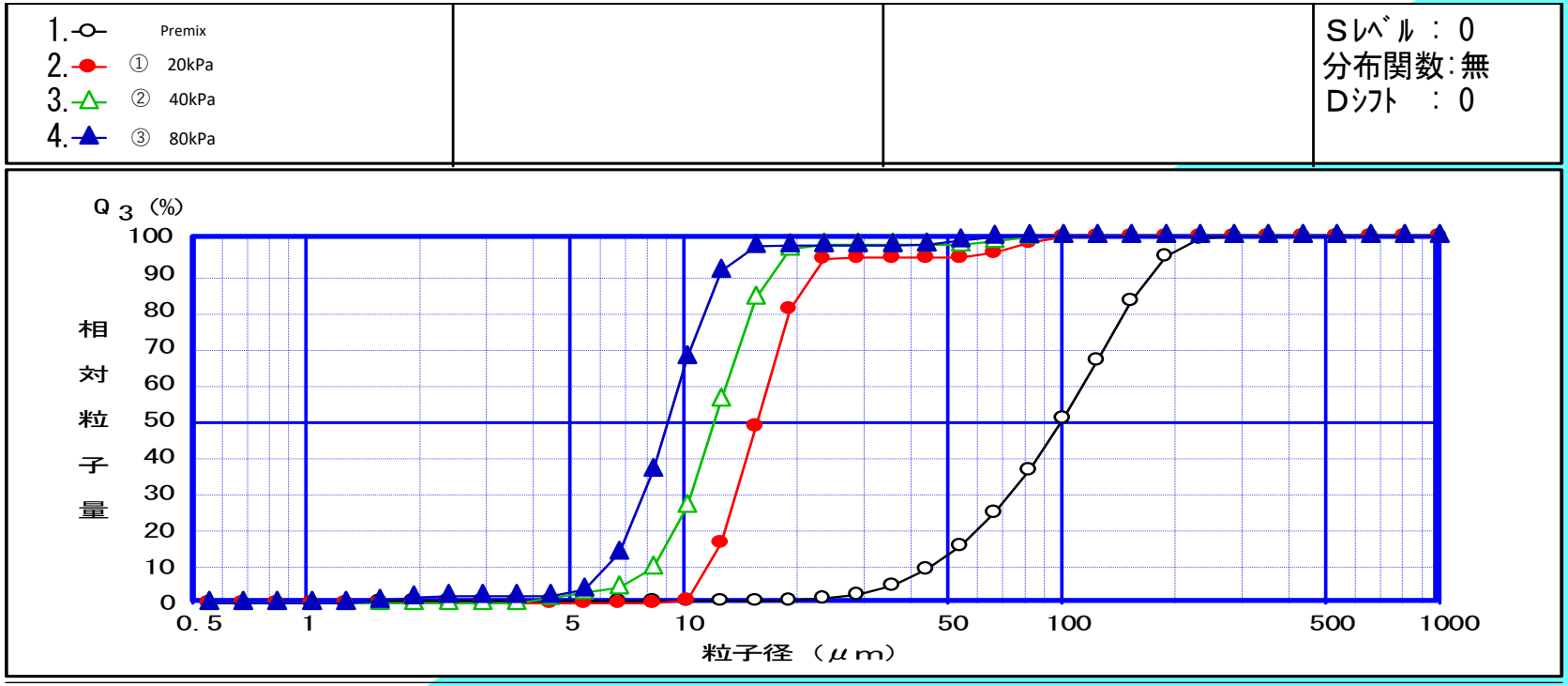
Sample	Average (μm)	Mode (μm)	Median (μm)	10%D (μm)	90%D (μm)	90%D /10%D
Premix	**91.634**	**138.246**	**100.407**	**44.865**	**174.321**	**3.9**
① 20kPa (5sec/10ml)	16.862	13.984	15.685	11.556	22.187	1.9
② 40kPa (4sec/10ml)	12.117	11.354	12.104	8.325	17.239	2.1
③ 80kPa (2sec/10ml)	9.083	9.219	9.109	6.244	12.521	2.0

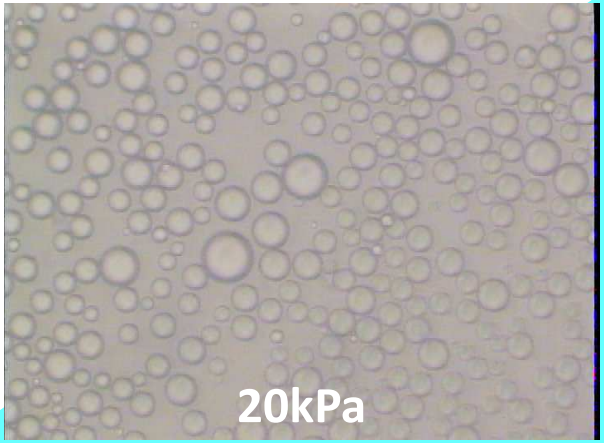
The higher the pressure (the faster the pass speed), the smaller the emulsion.
When the pressure is low, large particles are generated, and when the pressure is high, a fine emulsion is generated.

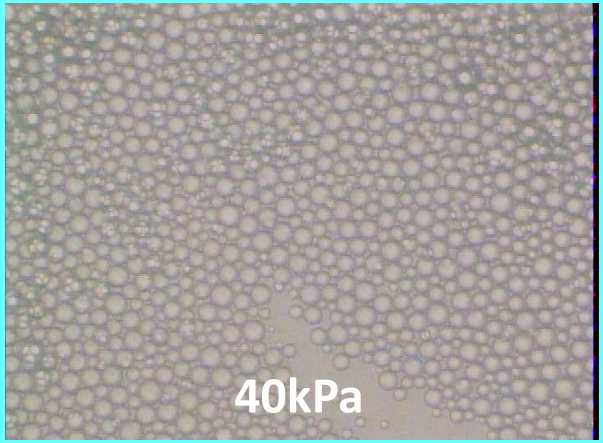
< Premix preparation method >
Polydispersion ：Stirring with Stirrer. 400rpm, 1min

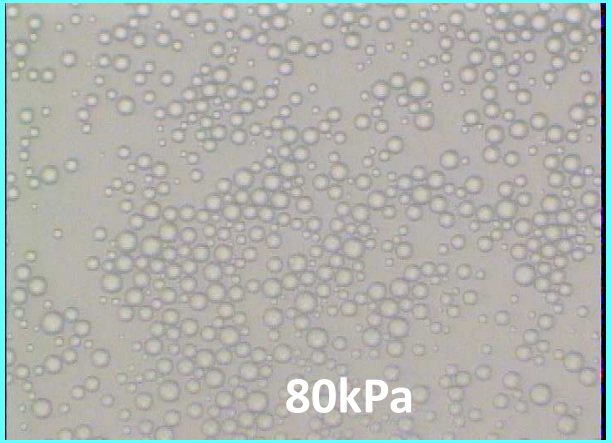
< Condition of high speed emulsification >
SPG membrane pore size = 11 μm

2. The effects of “Pass speed of SPG membrane (= Pass Pressure)”









3. The effects of “Number of pass”

We confirmed how the emulsion changes by multiple pass the same pore size SPG membrane.

Sample	Average (μm)	Mode (μm)	Median (μm)	10%D (μm)	90%D (μm)	90%D /10%D
Premix	**80.584**	**91.146**	**84.451**	**38.541**	**160.081**	**4.2**
1 pass （4sec/10ml）	12.235	11.354	12.024	7.811	17.768	2.3
2 pass （2sec/10ml）	10.434	11.354	10.301	7.034	14.661	2.1
3 pass （2sec/10ml）	8.930	9.219	9.102	6.652	12.051	1.8

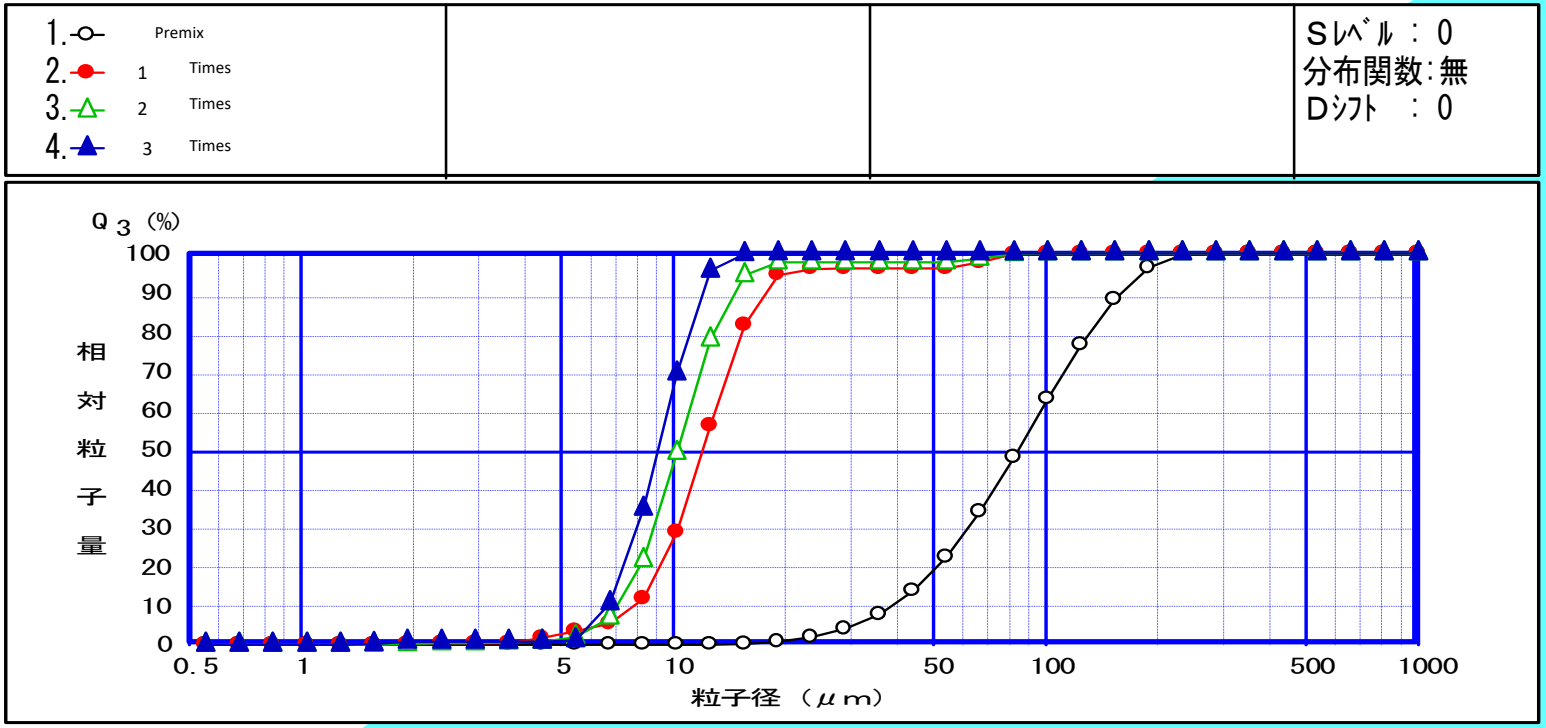
The distribution became narrower as the number of pass increased. This is because large particles decreased. In addition, as the number of pass increased, the emulsion particle size smalled.

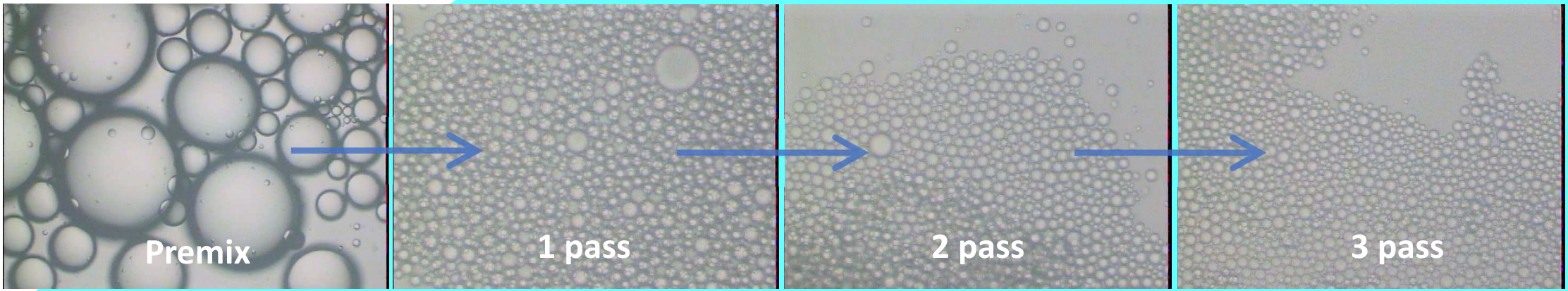
< Premix preparation method >
Polydispersion : Stirring with Stirrer. 400rpm, 1min

< Condition of high speed emulsification >
SPG membrane pore size = 11 μm, Pass Pressure = 40 kPa

High speed emulsification pass pressure 40kPa	1 pass pass speed = 4sec/10ml
	2 pass pass speed = 2sec/10ml
	3 pass pass speed = 2 sec/10ml

3. The effects of “Number of pass”





< Consideration >

As the above results, it was found that the each emulsification conditions affect the emulsion after high speed emulsification.
In particular, in order to obtain a monodispersed emulsion, the following factors are considered important.

: The premix emulsion uses monodispersed emulsion.

: Consider the optimal pass speed (emulsification pressure).

In addition, we have found that it becomes monodispersed by the following **"Multistage Emulsification"**.

: Premix (Stirring with Stirrer)

→ SPG 30μm (High speed emulsification)

→ SPG 11μm (High speed emulsification) *by "1. The effects of "Premix emulsion distribution" ②Slightly Monodispersion"

: Multiple pass the same pore size SPG membrane *by 3. The effects of "Number of pass"

When performing high-speed emulsification, it is necessary to comprehensively search each emulsification condition suitable for the purpose in consideration of the above.