

Dispersing Carbomers, Mixing Technology Matters!

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Mixing Setups

High Shear Mixer

For the industrial high shear mixing trial, a Silverson Flashmix FMX50 was used. The setup is shown in Figure S1. This particular model is suitable for mixing powders and liquids and can be used for a wide range of materials. In addition, the FMX50 is able to operate at high temperatures and high viscosities.^{1,2} The Silverson Flashmix was operated as follows. First, the mixer recirculates liquid from the process vessel through the Flashmix at high velocities. Next, the powder valve is opened to admit the carbomer powder into the liquid stream. In the high shear zone of the mixer, where the powder and the liquid are then instantaneously combined. The resultant mix is transferred back to the vessel.

All parts of the Silverson FMX50 are constructed from 316L stainless steel. The motor is of the type TEFV (Totally Enclosed Fan Ventilated), with a maximum power consumption of 11 kW. Tri-clamp fittings are used for the inlet and outlet connections. Hygienic (EHEDG approved) single mechanical shaft seals are used. The valves are manual butterfly valves. The powder feed hopper is also constructed from stainless steel. For these type of Carbopol® experiments, a rotor-stator workhead with big, round holes was selected.

A 150 L stainless steel vessel was used as secondary mixing tank. Stirring of the vessel was achieved using a Techtop Type T3A 802-4 motor (SIMOTOP N.V.; Arnhem, The Netherlands) with FLAI BG 080 industrial ventilation (WISTRO; Langenhagen, Germany) and Optidrive E3 frequency drive (Invertek Drives; Welshpool, United Kingdom). The stirrer contained four blades. In addition, an OPTIFLUX 6000 flowmeter (KROHNE Ltd; Wellingborough, United Kingdom) was incorporated into the setup to measure and monitor the flow through the system.

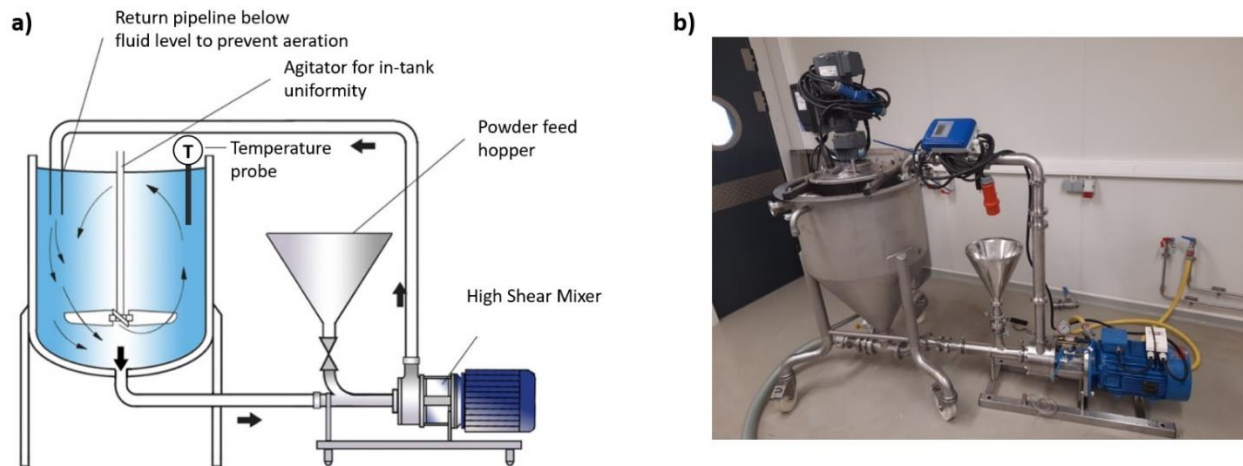


Fig. S1 a) High shear mixing setup (Silverson Flashmix FMX50 device).^{1,2} The mixture is recirculated between storage vessel and high shear mixer at a velocity of 12 m³/h. Carbomer powder is introduced via the feed hopper. The temperature of the mixture is monitored using a Testo 110 temperature probe, which is placed inside the tank. **b)** Image of the actual HSM setup used.

Magnetohydrodynamic Mixer

The magnetohydrodynamic (MHD) mixing setup used is shown in Figure S2. The mixer with Venturi inlet for feeding powders was provided by M4E “Magnets for Emulsion” company. The setup makes use of a Seepex eccentric screw pump (1.5 kW) with gland packing (Seepex; Herentals, Belgium) to recirculate the fluid.

Similar to the high shear mixing setup, stirring of the secondary 150 L mixing tank was achieved using a Techtop Type T3A 802-4 motor (SIMOTOP N.V.; Arnhem, The Netherlands) with FLAI BG 080 industrial ventilation (WISTRO; Langenhagen, Germany) and Optidrive E3 frequency drive (Invertek Drives; Welshpool, United Kingdom). The stirrer contained four blades. In addition, an OPTIFLUX 6000 flowmeter (KROHNE Ltd; Wellingborough, United Kingdom) was incorporated into the setup to measure and monitor the flow through the system.

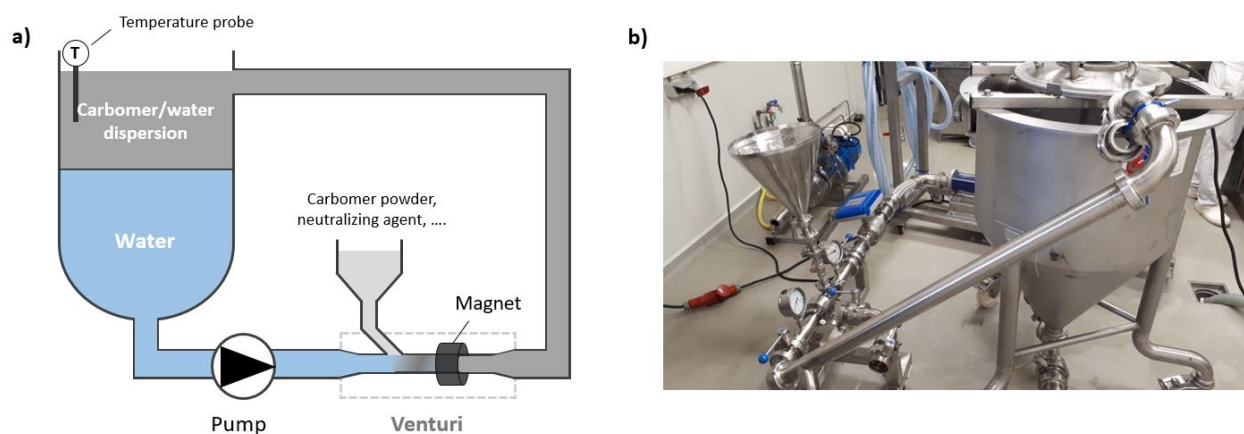


Fig. S2 a) Schematic representation of the MHD mixing setup used to disperse the Carbopol® powder. The aqueous mixture is circulated at 8 m³/h using a screw pump. The MHD effect is generated by forcing the liquid through a narrow slit in the conduct to create a turbulent flow ($Re > 4,000$) while applying a permanent magnetic field. The local pressure reduction at the restriction in the conduct creates a Venturi effect enabling to dose both powders and liquids to the recirculating fluid. The temperature of the mixture is monitored using a Testo 110 temperature probe placed inside the tank. **b)** Image of the actual MHD mixing setup used.

Process Description

Preparation of 1.22 wt.% carbomer dispersion

The experimental procedure for preparing a 1.22 wt.% Carbopol® 980 NF (Lubrizol Corporation) dispersion is summarized below. The pH of the samples was measured immediately after sampling using a Testo 205 pH meter.

Components	Dosage	Actions
H ₂ O (kg)	35.28	Carbopol® powder addition via hopper + rinse with Carbopol® dispersion. 50 g sample taken after 1 and 3 minutes of mixing.
Carbopol® (g)	435.84	

Rheological Characterization

Rheological properties of the hydrated carbomer samples collected after 3 minutes of mixing were determined at 22 °C with a MCR 702e rheometer (Anton Paar). Measurement protocols for small amplitude oscillatory shear (SAOS) and flow behavior testing are:

- Small amplitude oscillatory shear (SAOS) testing:
 - Time sweep to make sure sample is at steady-state: 0.1 % strain, 10 rad/s, 1 min
 - Frequency sweep: 0.1 % strain, 100-0.1 rad/s
 - Strain sweep: 0.01-1000 % strain, 10 rad/s
 - Time sweep to check recovery after partial structural breakdown due to strain sweep: 0.1 % strain, 10 rad/s, 5 min
 - All measurements are conducted at 22 °C.

- Flow behavior testing:
 - Flow sweep: 0.01-100 /s followed by reverse sweep
 - All measurements are conducted at 22 °C.

Nuclear Magnetic Resonance Spectroscopy: ^1H direct excitation and ^1H - ^1H 2D EXSY measurements

NMR measurements were performed on a Bruker Avance III 500 MHz spectrometer equipped with a 4 mm H/X/Y triple resonance probe with ^1H Larmor frequency at 500.87 MHz. The samples were packed in disposable, 4 mm Kel-F rotor inserts. Spectra were acquired at 4 kHz using a 90 degree pulse with an RF strength of 83 kHz, a recycle delay of 5 s and 32 transients. Spectra were referenced with respect to TMS, using adamantane as secondary reference with a ^1H resonance at 1.81 ppm. ^1H - ^1H 2D-exchange spectroscopy (EXSY)³ was performed with a mixing time of 3s. The two -dimensional spectra was collected with 400 t_e increments of 5 ms and 16 transients in the direct dimension.

Supplementary Figures and Tables

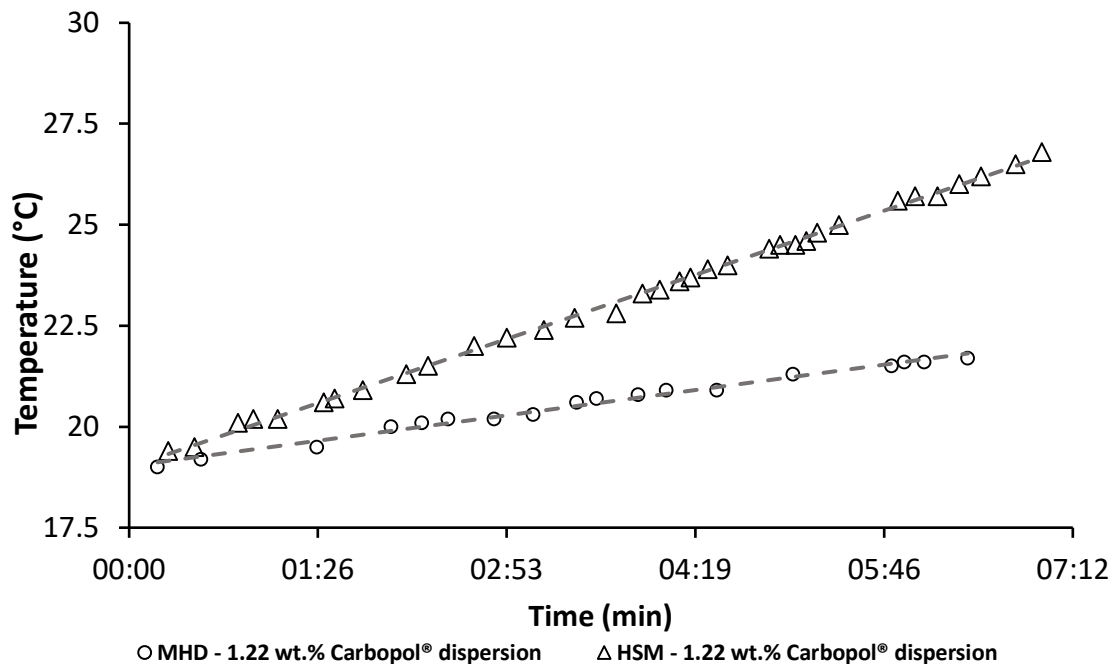


Fig. S3 Temperature evolution of the aqueous carbomer mixture with increasing mixing time in the high shear mixer (hollow triangle) and in the magnetohydrodynamic mixing setup (hollow spheres).

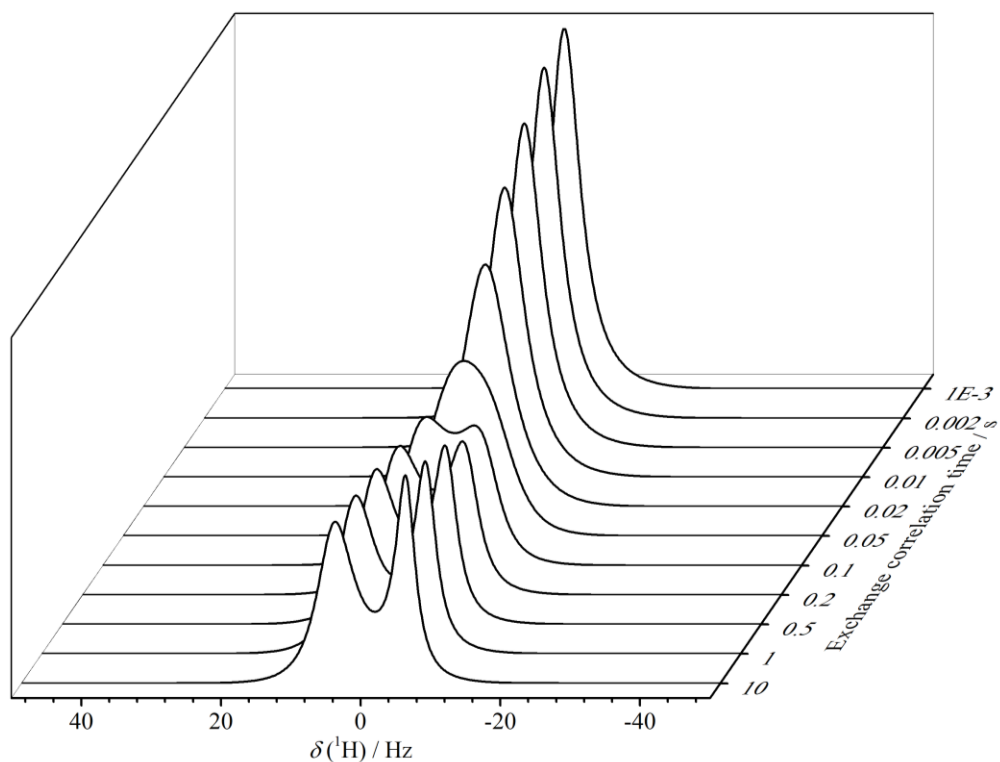


Fig. S4 ^1H line shape simulations assuming different chemical exchange rates for HSM - 1 min sample using Bloch-McConnell equations.⁴

Table S1. Overview of the ^1H signal area (in %) for each of the signals (CH_3 , CH_2 and CH) originating from polyacrylic acid backbone of the carbomer.

^1H chemical shift (ppm)	^1H Signal Area (%)			
	HSM - 1 min	HSM - 3 min	MHD - 1 min	MHD - 3 min
2.4 – 2.5	36.2	33.1	33.5	34.5
1.9 – 2.0	14.1	14.1	14.8	14.7
1.5 – 1.9	47.6	45.4	47.5	47.1
1.2 – 1.4	2.1	7.4	4.1	3.7

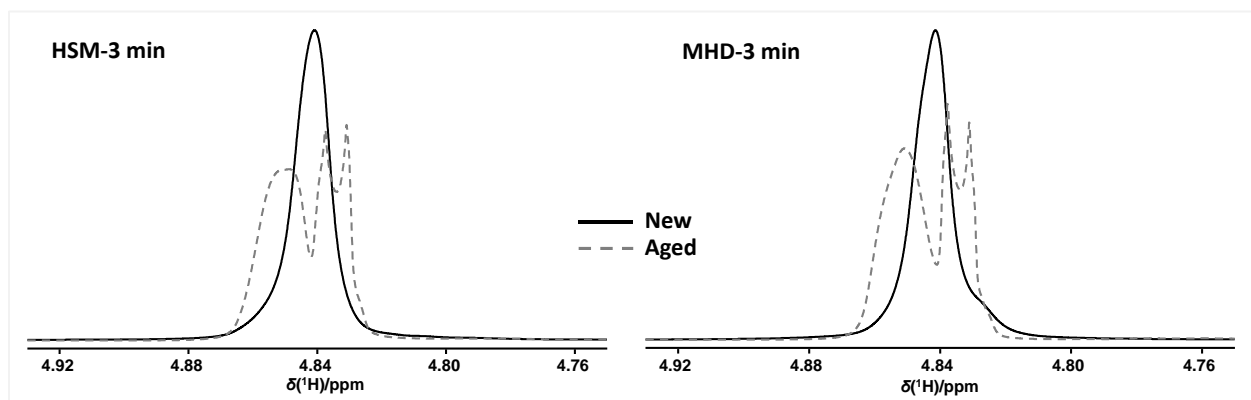


Fig. S5 ^1H NMR spectra of HSM-3 min and MHD-3 min samples as prepared (full line) vs. ^1H NMR spectra of the same samples recorded after 18 months of storage (dotted line). With time, the aqueous phase de-mixes and different water populations re-emerge similar to the ^1H NMR spectrum recorded for the HSM-1 min sample.

References

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Author Contributions

J.A.M and E.B designed and supervised the research. M.H., L.V. and F.V. carried out the experiments and processed the results. C.V.C., K.D. and S.R. performed and interpreted the NMR experiments. E.B. designed and supervised the NMR research and guided interpretation. All authors contributed to writing the manuscript.