Magneto-Hydrodynamic Mixing: a New Technique for Preparing Carbomer Hydrogels

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0. Abstract

Magnetohydrodynamic mixing was evaluated as an alternative to conventional high shear mixing in the preparation of carbomer hydrogels containing 1.22 wt.% Carbopol® 980 NF. Neutralization of the carbomer dispersion (pH = 2.74) with triethanolamine (TEA) enabled to adjust the pH of the mixture and tune the viscosity of the hydrogel. Using high shear mixing, this approach was limited to 0.2 wt.% TEA (pH = 3.83) as the gel became too viscous and the recirculation flow dropped from 12 to 0.3 m3/h. Magnetohydrodynamic mixing enabled to reach TEA concentrations up to 1.0 wt.% (pH = 5.31). Apparent viscosity measurements on samples having 0.2 wt.% TEA revealed lower viscosities for carbomer hydrogels prepared with high shear mixing, i.e. 6,800 mPa·s versus 8,800 mPa for magneto-hydrodynamic mixing. Based on ^{1}H NMR evidence, this decrease in apparent viscosity was attributed to structural damage to the carbomer backbone in combination with mechanochemical degradation of the added TEA.

1. Introduction

Carbomers are high molecular weight cross-linked polyacrylic acids that swell in water to form hydrogels with tunable viscosity. These polymers are commercially available under different trade names and in various grades depending on the degree of crosslinking and manufacturing conditions.[1] Because of their unique elastoviscoplastic properties and their low toxicity, carbomer gels are widely used in the cosmetics, pharmaceuticals, paint, and food industry as a thickening, suspending, dispersing, and stabilizing agent.[2,3] Carbomer hydrogels have been in high demand since the start of the COVID-19 pandemic as they are often used for preparing hand sanitizing hydro-alcoholic gels. These hydrogels are typically prepared using high shear mixers which are energy-intensive and typically require long mixing times to achieve homogenization.[4,5]

Here we report on the application of magneto-hydrodynamics as an alternative mixing principle to prepare viscous carbomer hydrogels in a time and energy-efficient manner. Experiments were performed with Carbopol® 980 NF (Lubrizol)[6], a synthetic homopolymer of polyacrylic acid cross-linked with allyl pentaerythritol. Carbopol® 980 NF is supplied as a dry, white powder of primary particles averaging around 0.2 µm in diameter. Each particle is composed of tightly coiled linear polymer chains, which are soluble in polar solvents such as water.[2,6] The acrylic acid groups render the polyacrylic acid backbone responsive to changes in pH and ionic strength. Upon initial mixing with water, the polymer forms an acidic dispersion (pH ≈ 3) of partly unfolded polymer chains following hydration and deprotonation of the carboxylic acid groups. After the initial hydration step, neutralization of the aqueous dispersion with a base finally yields a hydrogel that becomes increasingly more viscous with increasing pH (up to a pH of 10). The added base neutralizes the carboxylic acid groups lining the acrylic acid backbone, resulting in negatively charged polymer chains. Electrostatic repulsion between these chains promotes further unfolding of the polymer, enhancing the viscosity of the hydrogel even further.[7–10] This mechanism is sketched in Figure 1. At pH values higher than 10, viscosity starts to decrease again as the excess base screens the charges, dampening the internal electrostatic repulsion driving polymer chain unfolding.[11]

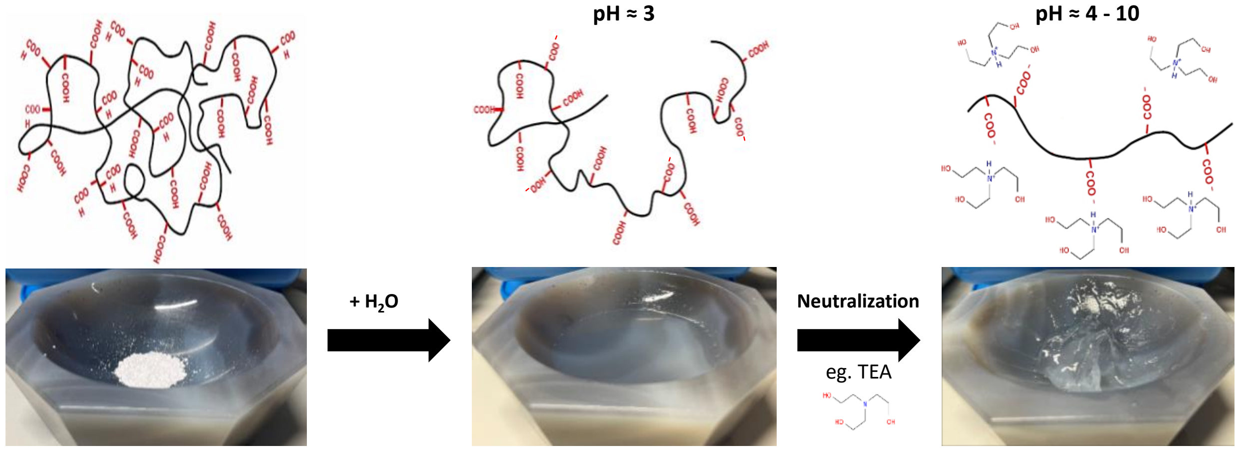


Figure 1. Schematic representation of the evolution in carbomer polymer structure under different conditions. Initial mixing of the carbomer powder (*left*) with water yields an acidic dispersion consisting of partly unfolded polymer chains (*middle*). Neutralization of the carboxylic acid groups with a base (here: triethanolamine or TEA) results in a negatively charged polyacrylic acid polymer backbone. Internal electrostatic repulsion causes the polymer chains to unfold even more, yielding a viscous hydrogel (*right*).

Next to the pH, other parameters such as carbomer concentration, temperature, solvent choice, and mixing method impact the viscosity of the final hydrogel.[12] In industry, high shear mixers (HSM) are commonly used for preparing carbomer hydrogel.[13] High shear mixing is based on the rotor-stator principle to generate shear forces and turbulence within the fluid to effectuate mixing. Important drawbacks of this mixing method are high energy intensity and heat generation near the rotor. The latter can lead to thermal degradation of thermolabile compounds, especially when longer mixing times and viscous mixtures hindering heat dissipation are involved.[13,14] With carbomers, shear may physically and chemically damage the hydrated polymer chains, resulting in a permanent viscosity loss.[15] In some cases the viscosity loss can be as high as 50%.[14] High shear mixers have some drawbacks when used for preparing carbomer hydrogels, but it remains the industrial standard for lack of other alternatives.[12,13,15,16]

Magneto-hydrodynamic mixing (MHD) is based on the application of a magnetic field on a fluid in turbulent flow regime. Turbulent flow is characterized by an irregular mixing pattern in randomly moving eddies. The impact of a permanent magnetic field on turbulent flowing colloidal system is very complex,[17–19] and even today there is no comprehensive scientific description of the phenomena. Experimental observations reveal that the magnetic field is responsible for additional shear, electrostatic, tensile and oscillatory forces.[20–23] Pairing of turbulent flow with electromagnetic forces creates powerful conditions for dispersing powders, enhancing fragmentation, de-agglomeration, hydration and solubilization.[24] These mixing conditions can be advantageous to emulsification, scale prevention, dispersion, crystallization, and catalyst preparation processes.[24–29] Recently, it was shown that magnetohydrodynamic mixing can also be used disperse and hydrate carbomers.[30] The hydrated carbomer dispersions exhibited higher viscosity and higher storage modulus than samples prepared through conventional high shear mixing.[30] ^{1}H NMR revealed different water distributions depending on the mixing method and demonstrated significant structural damage to the carbomer backbone to be induced by high shear mixing.[30]

The current work expands on previous exploratory research investigating carbomer hydration and documents the potential of MHD mixing technology for application in sol-gel sciences, more specifically in the field of hydrogel preparation. The performance of magneto-hydrodynamic mixing for preparing highly viscous carbomer hydrogels was evaluated against conventional high shear mixing for gels containing 1.22 wt.% Carbopol® 980 NF and different concentrations of neutralizing agent. Key performance criteria include viscosity, pH and temperature. The results are discussed based on molecular level information gained from nuclear magnetic resonance (NMR) spectroscopy.

2. Mixing setups and Methodology

2.1 High shear mixing

High shear mixing (HSM) experiments were carried out using a Silverson Flashmix FMX50 device (Figure 2A).[31] Technical specifications of the experimental setup are provided in the 'Supplementary Material' section along with an image of the actual setup used (Figure S1). The carbomer powder was hydrated by dosing 435.84 g Carbopol® 980 NF powder via the feed hopper to 35.28 L demineralized water, resulting in a nominal composition of 1.22 wt.% of carbomer. The initial recirculation rate was 12 m^3/h. Triethanolamine (TEA) was then used as base for neutralizing the pH of the aqueous carbomer dispersion. TEA, like other polyols, has the benefit of forming hydrogen bonds and linkages between different polymer chains, resulting in a more rigid gel structure with higher viscosity compared to other neutralizing agents such as sodium or potassium hydroxide.[2,11] An amount of 73.59 g TEA, diluted in 1.10 L demineralized water, was added through the feed hopper yielding an overall TEA concentration of 0.2 wt.%. The dispersion was recirculated for an additional 4.5 minutes before taking a sample. The holding tank was continuously agitated at 30-45 rpm to ensure sample uniformity. In a final step, an additional amount of 78.29 g of TEA, diluted in 1.197 L demineralized water, was added to achieve an increased TEA concentration of 0.4 wt.%. After an additional 4.5 minutes of mixing, a final sample was taken. The different preparation steps are summarized in Figure 2C.

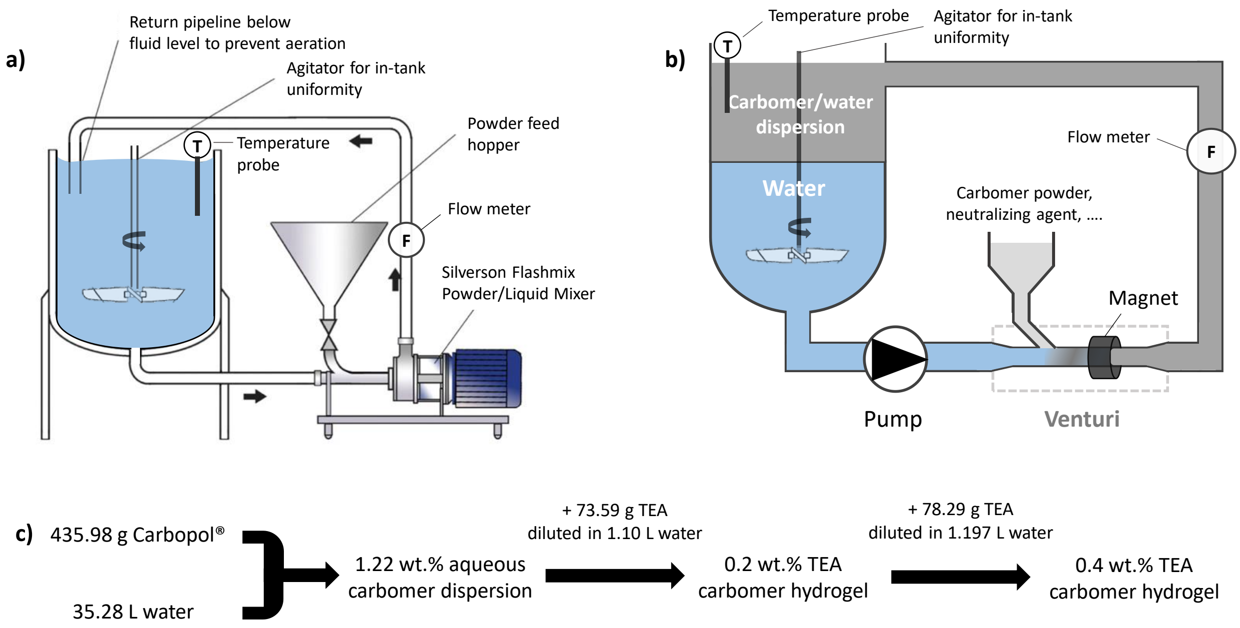


Figure 2. A. High shear mixing setup (Silverson Flashmix FMX50 device).[4,31] The mixture is recirculated between storage vessel and high shear mixer at a flow rate of 12 m^3/h. Carbomer powder is introduced via the feed hopper. **B.** MHD setup for the carbomer hydrogel preparation. The mixture is circulated at 8 m^3/h through a narrow duct with Venturi and permanent magnet. In both setups, the holding tank is agitated at 30-45 rpm to ensure sample uniformity. The temperature probe is positioned inside the tank to monitor the temperature evolution of the mixture. **C.** Overview of the different steps for preparing a 1.22 wt.% carbomer hydrogel with 0.2 and 0.4 wt.% triethanolamine (TEA).

2.2 Magneto-hydrodynamic mixing

A scheme of the magneto-hydrodynamic (MHD) mixing setup is presented in Figure 2B. Technical specifications of the setup along with a photo of the actual setup used (Figure S2) can be found in the Supplementary Material. A screw pump is used to recirculate the mixture. The MHD effect is generated by a slit-shaped restriction in the pipe creating a turbulent flow (Re > 4,000) in combination with a permanent magnetic field of *ca*. 0.4 T. Depending on the inner free diameter of the pipe restriction and the fluid flow rate, Reynolds numbers as high as 64,000 can be obtained.[27] The local pressure reduction at the restriction in the duct creates a Venturi effect enabling the dosing of carbomer powder as well as base to the circulating fluid.

A carbomer concentration of 1.22 wt.% was used in analogy to the HSM experiments. 435.97 g of Carbopol® 980 NF was added to 35.28 L of demineralized water circulating in the system at a flow rate of 8 m^3/h. Subsequently 73.59 g of TEA, diluted in 1.10 L demineralized water, was added to the dispersion as neutralizing agent, resulting in a TEA concentration of 0.2 wt.% similar to its HSM counterpart. A sample of the carbomer hydrogel was taken after 4.5 minutes of mixing. To ensure sample uniformity, the holding tank was continuously agitated at 30-45 rpm. An additional amount of 78.29 g TEA, diluted in 1.197 L demineralized water, was then added enhancing the TEA concentration to 0.4 wt.%. After an additional 4.5 minutes of mixing, a final sample was taken.

2.3 pH, apparent viscosity, temperature and flow rate measurements

The pH of the different samples was measured using a Testo 205 pH/temperature measuring instrument.

Apparent viscosities of the 0.2 and 0.4 wt.% TEA carbomer hydrogels were measured on samples at room temperature (22 °C) using a Brookfield Viscometer DV-II+Pro. This type of viscometer is operated with seven different spindles. Each spindle has a different diameter, which decreases with increasing spindle number. The higher the apparent viscosity of the hydrogel, the higher the spindle number that is required. Selection of the appropriate spindle yields a value between 10 - 100 on the instrument's %torque scale, along with the spindle speed and the corresponding mPa·s value.

Temperature evolution of the gel upon addition of TEA was monitored in real-time using a Testo 110 temperature probe which was placed inside the holding tank. Changes in the flow rate were measured using an OPTIFLUX 6000 flowmeter.

2.4 NMR measurements

The samples were loaded into disposable Kel-F inserts. The inserts were then closed and transferred to a 4 mm ZrO_2 rotor. Finally, the rotor was capped and inserted into the spectrometer. ^1H measurements were performed on a Bruker Avance Neo 800 MHz spectrometer equipped with a 4 mm H/C/N HRMAS probe having a ^1H larmor frequency of 801.25 MHz. ^1H NMR spectra were acquired at 8 kHz using a pulse with a pi/6 flip angle at 25 kHz RF field strength, a recycle delay of 5 s and 512 transients. Chemical shift referencing was performed with respect to TMS, using ethylbenzene (10% in chloroform-d) as secondary reference [$\delta(^1H) = 1.22$ ppm for ($-CH_3$)].

3. Results and Discussion

Addition of 0.2 wt.% TEA to the aqueous carbomer dispersion prepared using the high shear mixer resulted in a pH increase from 2.72 to 3.83. This pH change induced an increase in apparent viscosity resulting in a reduction of the flow-rate from 12 to 0.3 m^3/h, as measured at the outlet of the mixer as indicated in Figure 2. As the addition of an even higher concentration of TEA would have caused clogging of the piping and blocking of the mixer, the experiment was halted at this point. The apparent viscosity of the neutralized hydrogel was measured using spindle 6 and amounted to 6,800 mPa·s (Table 1).

Neutralization of the hydrated carbomer dispersion with 0.2 wt.% TEA in the MHD setup increased the pH from 2.72 to 3.98. The apparent viscosity of the sample was determined at 8,800 mPa·s (Table 1). In contrast with HSM, the flow in the MHD setup remained constant despite the high viscosity. Even at higher TEA contents of 0.4 and 1.0 wt.% and higher viscosities (Table 1), the MHD setup continued to perform consistently with a steady flow of 7.8 m^3/h.

Table 1. Overview of apparent dynamic viscosity data for 1.22 wt.% carbomer hydrogels neutralized with different TEA concentrations in HSM and MHD mixing setups.

Sample	pH	Spindle N°	RPM	%torque	Apparent viscosity (mPa·s)
HSM 0.2 wt.% TEA	3.83	6	100	70	6,800
MHD 0.2 wt.% TEA	3.98	6	100	84	8,800
MHD 0.4 wt.% TEA	4.62	7	100	30	13,430
MHD 1.0 wt.% TEA	5.31	7	100	41	16,520

During mixing and addition of 0.2 wt.% TEA, the temperature evolution of the carbomer mixtures was monitored in real time (Figure 3). Before addition of TEA, the temperature of the carbomer dispersion increased more quickly in the HSM as compared to the MHD experiment, respectively reaching 27.0 °C versus 22.0 °C after 7 minutes of mixing. Following the initial TEA addition, the temperature further increased to 29.5 °C in the HSM setup and to *ca*. 23.5 °C in the MHD experiment. During the subsequent increase of the TEA concentration to 0.4 wt.% (in the MHD experiment only) temperature further increased to *ca*. 24.5°C. The elevated temperatures observed in the HSM setup as compared to the MHD setup are likely caused by excessive shear at the rotor-stator interface. These shear forces create several high temperature 'hot spots', which ultimately heat up the entire mixture as the mixing goes on.[13] For samples with identical TEA concentrations (0.2 wt.%), MHD produced samples with significantly higher apparent viscosity as compared to HSM, *viz*. 8,800 versus 6,600 mPa·s (Table 1). In line with ^{1}H NMR observed structural damage induced by HSM during carbomer dispersion in water,[30] the lower apparent viscosity observed in the hydrogel samples prepared using HSM can be attributed to mechanochemically activated chain scission of the carbomer polymer.[2,32]

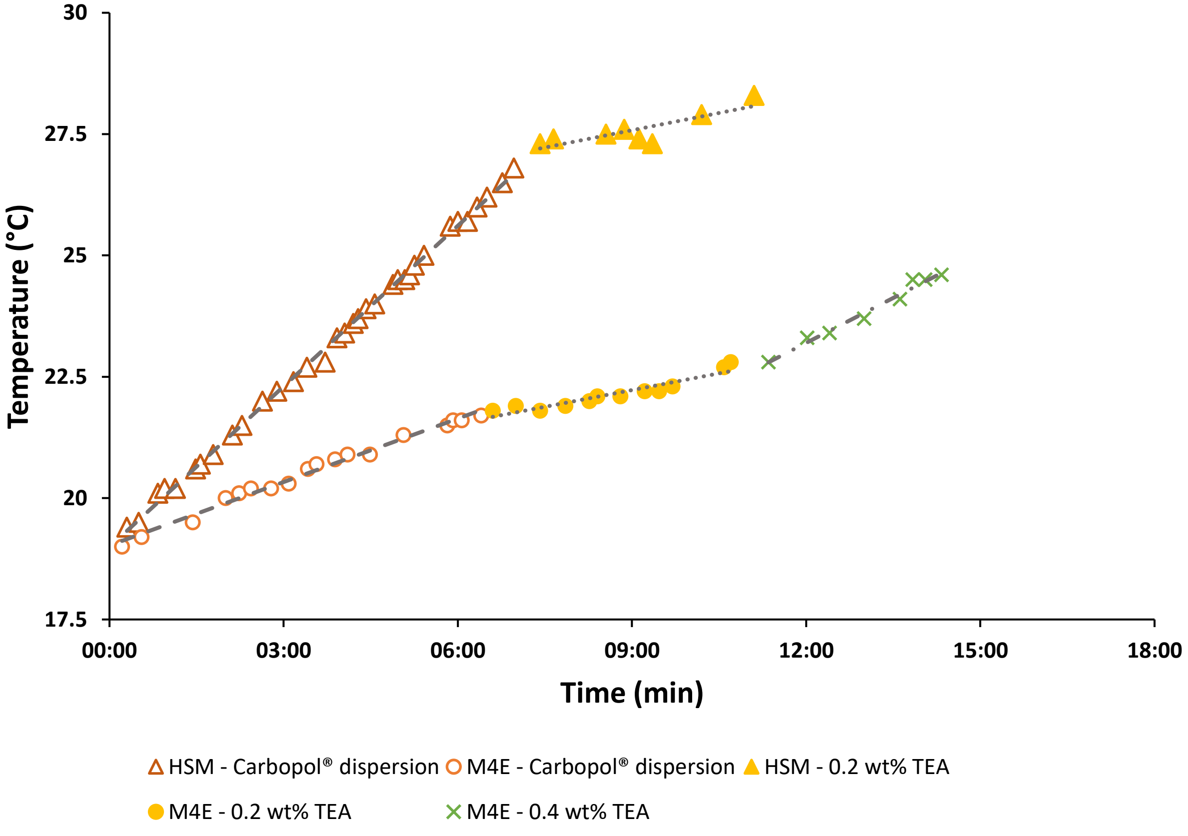
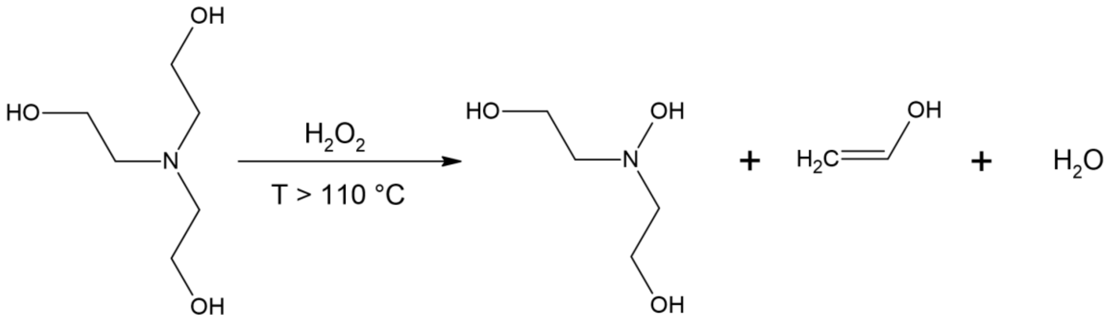
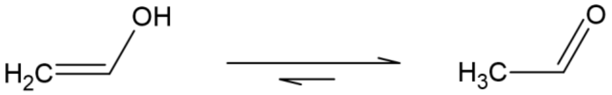


Figure 3. Temperature evolution in the HSM and MHD carbomer hydrogel preparation.

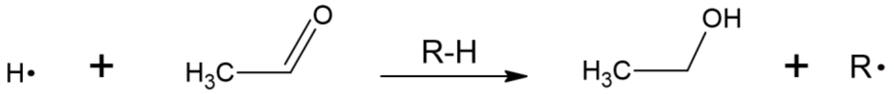
This degradation was confirmed by ^1H NMR spectroscopy performed on hydrogel samples containing 0.2 wt.% TEA prepared with HSM (Figure 4). The main signal around 4.7 ppm can be assigned to water. ^1H NMR signals of the polyacrylic acid and pentaerythritol linkers occur at chemical shifts between 1.5-2.5 ppm. Signals at 3.4 ppm and 3.9 ppm are assigned to protons of triethanolamine (TEA). The ^1H NMR spectrum of the HSM produced sample shows three additional resonances at 1.2, 2.1 and 3.6 ppm respectively. The signals at 1.2 and 3.6 ppm (Figure 4) are indicative for the presence of ethanol, whereas the 2.1 ppm signal demonstrates the presence of acetaldehyde. The emergence of these new resonances is accompanied by a simultaneous decrease in the intensity of the ^1H signals attributed to TEA (Figure 4), implying TEA is decomposed into acetaldehyde and ethanol. Such degradation pathway is known as the *Cope elimination* and typically occurs with tertiary amines in the presence of oxidants such as H_2O_2 at temperatures as low as 110 °C.[33] Although H_2O_2 is not actively added to the mixture, trace amounts of H_2O_2 are readily generated from airborne O_3 in heavily aerated aqueous mixtures undergoing shear.[34,35] In the present case, the entrapment of air bubbles in the viscous mixture would maximize the air/water interface. The aforementioned 'hot spots' would further provide the thermal energy required for the reaction to take place. Next to H_2O, the Cope elimination of TEA with H_2O_2 produces diethanolhydroxylamine and vinyl alcohol (eq. 1). The former readily evaporates at temperatures exceeding 23.5 °C.[36] The latter is unstable and tautomerizes into acetaldehyde (eq. 2),[36] which is easily converted to ethanol via a radical based reaction mechanism (eq. 3), as previously observed for gamma radiolysis of N,N-diethylhydroxylamine.[37] The different chemical reactions steps involved in the mechanochemical degradation of TEA are summarized below:



(1)



(2)



(3)

R-H being another organic molecule

Chain scission of the carbomer induced by mechanochemical activation could be a source of radicals,[32] and would also explain the increased degradation of the carbomer backbone as evidenced by the lower storage modulus G’ of hydrogels prepared with HSM (Figure S3). Such degradation already occurs during the initial dispersion and hydration the carbomer, as demonstrated by ^{1}H NMR.[30] During MHD mixing, however, TEA degradation seems to be negligible as no ^{1}H NMR signals for acetaldehyde or ethanol were observed in the spectra of the 0.2 wt.% TEA samples prepared with MHD (Figure 4). Overall, ^{1}H NMR evidence suggests the lower apparent viscosity observed for 0.2 wt.% TEA carbomer hydrogels prepared with HSM as compared to the hydrogels obtained with MHD mixing (Table 1) can be attributed to the combination of 3 effects: (1) decreased deprotonation of the carboxylic acid moieties during dispersion of polyacrylic acid,[30] (2) increased polyacrylic acid backbone degradation, and (3) a decreased hydroxyl content resulting from HSM induced degradation of TEA and subsequent evaporation of diethanolhydroxylamine.

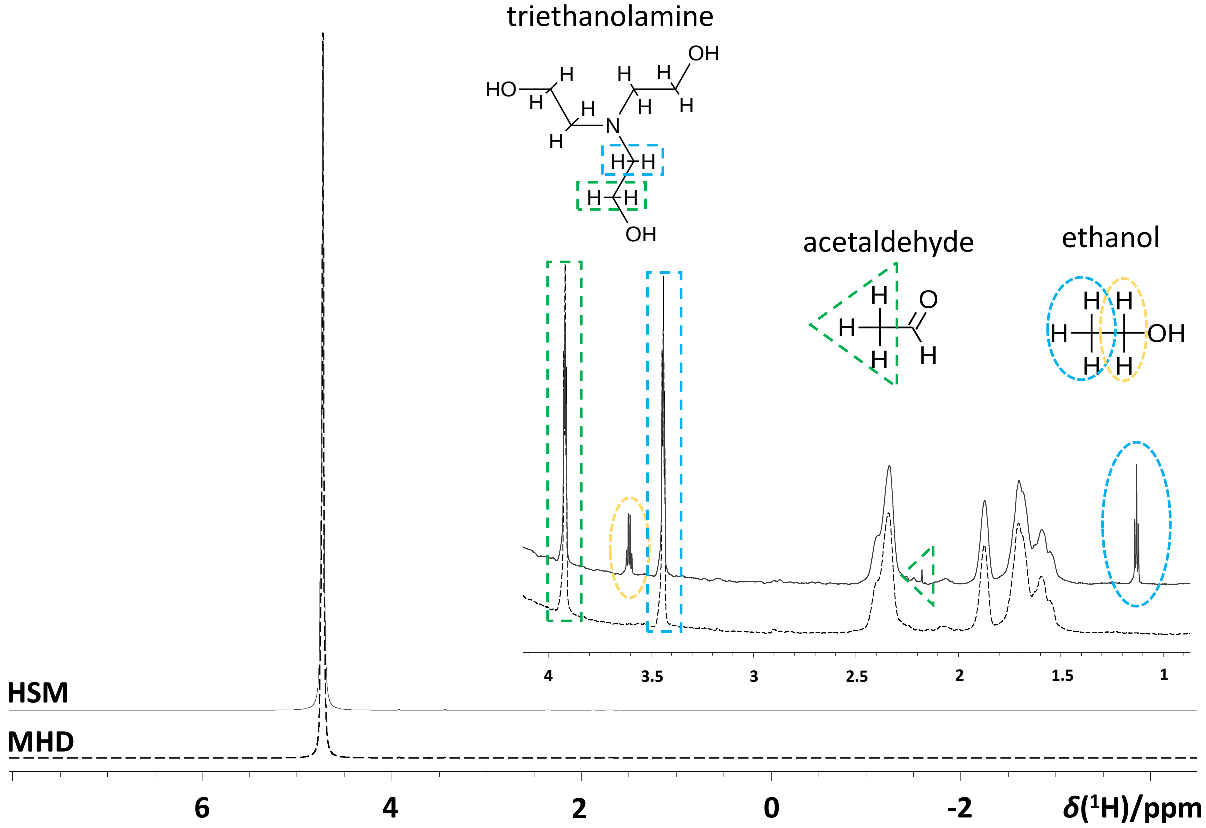


Figure 4. ^{1}H NMR spectra of carbomer hydrogels containing 1.22 wt.% Carbopol® and 0.2 wt.% TEA prepared using MHD (dotted line) and HSM mixers (full line). **Main display**: ^{1}H spectrum showing the large water signal at 4.7 ppm. **Inset**: Magnified view of the region between 1 and 4 ppm. ^{1}H NMR signals of the polyacrylic acid backbone and the pentaerythritol linkers in Carbopol® occur at chemical shifts between 1.5-2.5 ppm. Quantification of the TEA signal intensity reveals a TEA loss of 24.3%, which seems to be accompanied by the formation of acetaldehyde and ethanol in the case of high-shear mixing. TEA degradation appears to be negligible in the MHD samples.

4. Conclusion

In summary, we demonstrated the potential of magneto-hydrodynamic (MHD) mixing as an alternative mixing method to state-of-the-art high shear mixing (HSM) for preparing carbomer hydrogels with a carbomer content of 1.22 wt.%. Initial hydration of the carbomer with MHD proved to be more efficient. The increased apparent viscosity arising from the neutralization of the hydrated carbomer with 0.2 wt.% of TEA (pH ≈ 3.83) resulted in a non-continuous flow which dropped from 12.0 to 0.3 m^3/h for HSM. Unlike HSM, the MHD setup continued to perform consistently at a steady flow rate of 7.8 m^3/h, even at higher viscosities and TEA concentrations up to 1.0 wt.% (pH ≈ 5.31). NMR measurements showed that rotor induced shearing in HSM led to the mechanochemical degradation of ~25% of the added triethanolamine (TEA), ultimately resulting in the formation of acetaldehyde and ethanol. The absence of rotating parts in the MHD setup resulted in minimal shear and prevented the decomposition of TEA, leading to higher viscosities for similar amounts of added TEA, i.e. 8,800 mPa·s versus 6,800 mPa·s for HSM in the case of 0.2 wt.% TEA. The choice for magneto-hydrodynamic mixing over other conventional mixing methods might also be beneficial in other application areas and therefore merits further exploration.

Acknowledgments

The authors would like to extend their gratitude to Ms. Anja Vananroye for assistance with the revision of the manuscript. This work was supported by the ERC through an Advanced Research Grant under the European Union's Horizon 2020 research and innovation programme (No. 834134 WATUSO). NMRCoRe acknowledges the Flemish government, department EWI for financial support as International Research Infrastructure (I001321N: Nuclear Magnetic Resonance Spectroscopy Platform for Molecular Water Research). J.A.M. acknowledges the Flemish Government for long-term structural funding (Methusalem) and department EWI for infrastructure investment via the Hermes Fund (AH.2016.134). M.H. and E.B. also acknowledge FWO for an FWO-SB fellowship and a research grant (1.5.061.18N), respectively.

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