

Research Article

Demicellization of Polyethylene Oxide in Water Solution under Static Magnetic Field Exposure Studied by FTIR Spectroscopy

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Received 19 June 2013; Accepted 19 September 2013

Academic Editor: Sergej Tretiak

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FTIR spectroscopy was used to investigate the alterations of the vibration bands in the mid-infrared region of Polyethylene oxide in aqueous solution at 25 mg/mL concentration under exposure up to 4 h to a static magnetic field at 200 mT. FTIR spectroscopic analysis of PEO solution in the range 3500–1000 cm^{-1} evidenced the stretching vibrations of ether band, C–H symmetric-antisymmetric and bending vibrations of methylene groups, and the C–O–C stretching band. A significant decrease in intensity of symmetric and asymmetric stretching CH_2 vibration bands occurred after 2 h and 4 h of exposure, followed by a significant decrease in intensity of scissoring bending in plane CH_2 vibration around 1465 cm^{-1} . Finally, the C–O–C stretching band around 1080 cm^{-1} increased in intensity after 4 h of exposure. This result can be attributed to the increase of formation of the intermolecular hydrogen bonding that occurred in PEO aqueous solution after SMF exposure, due to the reorientation of PEO chain after exposure to SMF. In this scenario, the observed decrease in intensity of CH_2 vibration bands can be understood as well considering that the reorientation of PEO chain under the applied SMF induces PEO demicellization.

1. Introduction

Polymers are substances whose molecules have high molar masses, formed by repeated linking of small molecules called “monomers”.

Some natural polymers are proteins, starches, cellulose, and latex. Synthetic polymers are produced commercially on a large scale and have a wide range of properties and uses, such as plastics. Water-soluble polymers may be used in industrial and consumer products such as polymeric surfactant, pigment dispersants, or detergents, due to their properties of assembly in spherical micelles and vesicles that may be used as drug delivery vehicles. Indeed, several polymers can aggregate in micelles and can be considered good candidates for drug delivery purposes [1–4].

An important property of polymers is crystallization. The long chains of repeating monomers of a polymer form stacks of parallel chains called lamellae, that fold back on itself, as explained by [5, 6]. If crystallization is increased, the polymers tend to be stronger and more rigid, however this can lead to brittleness [7, 8].

Polymers structure have been accurately studied using neutron scattering techniques [9, 10].

In particular, the structure of polyethylene oxide (PEO) micelles is well described by the core-corona model [11–13]. PEO crystallinity was found by [14].

PEO is one of the simplest polymers, composed of chains of repeating $-\text{CH}_2-$ units, which is produced by the addition polymerization of ethylene; it is one of the most extensively studied water soluble polymers, because of the simplicity of its macromolecule's basic forming unit, the monomer $\text{CH}_2\text{CH}_2\text{O}$.

PEO is biocompatible, it can inhibit protein adsorption and can replace some biopolymers providing an insight to their behavior and functions [15–17].

The interest of researchers was also focused on the use of polymers in biomedical applications [18, 19], so that polymers can be considered to have a fundamental role in human life.

In the last few years, an interest in the effects of electromagnetic fields (EMFs) on the physicochemical properties of polymers has increased due to various functions attributed to these materials, often used as devices for insulation from

EMFs because their dielectrical properties. Indeed, although the conductivity of most polymers is low, more effects can be observed such as space charge and electrical breakdown strength [20].

Otherwise, the general population is commonly exposed to SMFs ranging from 0.1 to 10 mT, such as the magnetic flux density measured in proximity of the magnetic poles of conventional rail system DC traction motors, audio speaker components, battery-operated motors, refrigerator magnets, and headphones [21–23], whose effects on simple organic systems cannot be neglected [24–27].

It is only recently that the electrical characteristics of polymers have begun to be studied in detail, beginning an area of much interest besides their great mechanical properties. For instance, several elements of polymer materials are used in reactor materials and could be used in future fusion reactors, where magnetic confinement of plasma in a high induction static magnetic field (SMF) over 1 T have to be planned [28, 29]. In particular, the necessary security conditions for these future reactors give rise to concerns about the actual insulation capacity of polymers.

Furthermore, some effects of SMFs on crystalline and semicrystalline polymers such as creep and decrease of mechanical properties like the modulus of elasticity were observed, giving proof that the mechanical properties of polymers can be changed under SMFs exposure [30–33].

Hence, it may be important to study polymers behavior under the external stress of a SMF.

PEO can be considered a useful organic system to study the response to external SMFs because of the simplicity of its macromolecule's basic forming unit $\text{CH}_2\text{CH}_2\text{O}$.

The presence of an oxygen atom in PEO can change entirely the nature of the interactions when it is dissolved in various solvents.

PEO aqueous solution is characterized by hydrophobic and hydrophilic interactions as the CH_2 groups repel water, whereas hydrogen bonding of water molecules to the oxygen atoms on the polymer is formed when PEO is dissolved in water. Indeed, the main difference between the commonly studied polyethylene and PEO is water solubility, due to the polar nature of PEO.

PEO can dissolve in water because water molecules form a sheath around the PEO macromolecule by means of hydrogen bonding, whose model was extensively used to understand the mechanism of PEO solubility in water [34–40].

All these considerations led us to investigate the effects of external SMFs on the vibration bands of PEO using Fourier Transform Infrared (FTIR) spectroscopy. Indeed, infrared spectroscopy have already been proved to be a powerful tool in the investigation of the water structures inside polymeric materials [40–43].

2. Materials and Methods

2.1. Materials. Polyethylene oxide, with $M_w = 600000$, was purchased from Sigma Aldrich (Milan, IT).

The polymer was dissolved at 25 mg/mL concentration in bidistilled H_2O with gentle agitation at the room temperature of 20°C.

Samples of 250 μL of PEO aqueous solution were constituted at this concentration and immediately subjected to the following assays.

2.2. Experimental Design. The exposure system consisted of a couple of Helmholtz coils, with pole pieces of round parallel polar faces to produce a uniform magnetic field at the center of the coils distance, where the samples were placed, as described in [25]. A SMF was generated by coils powered by a DC generator, producing a uniform magnetic field intensity of 200 mT, corresponding to a half of the exposure limit for SMF recommended by the International Commission on Non-Ionizing Radiation Protection for public exposure [44].

Samples of PEO aqueous solution were placed at the centre of a uniform field area between the coils.

The magnetic field was continuously monitored by a magnetic field probe.

2.3. Infrared Spectroscopy. FTIR absorption spectra were recorded at room temperature by a spectrometer Vertex 80v of Bruker Optics.

PEO aqueous solution (25 mg/mL) samples were placed between a pair of CaF_2 windows separated with a 25 μm Teflon spacer. For each spectrum, 64 interferograms were collected and coadded by Fourier transformed employing a Happ-Genzel apodization function to generate a spectrum with a spectral resolution of 4 cm^{-1} in the range from 4000 cm^{-1} to 1000 cm^{-1} . IR spectra of water solution were subtracted from the spectra of PEO solution at the corresponding temperature.

Each measure was performed under vacuum to eliminate minor spectral contributions due to residual water vapor, and smoothing correction for atmospheric water background was performed. IR spectra were baseline corrected and area normalized for exposed and control samples, using vector normalization, calculating the average value of the spectrum, and subtracting from the spectrum decreasing the mid-spectrum. The sum of the squares of all values was calculated and the spectrum divided by the square root of this sum, as accurately described in [45, 46].

Also, interactive baseline rubberband correction was used to subtract baselines from spectra, using a rubberband which is stretched from one spectrum end to the other, and the band is pressed onto the spectrum from the bottom up with varying intensity. This method is performed iteratively, depending on the number of iterations in the algorithm and the baseline as a frequency polygon consisting of n baseline points. The resulted spectrum will be the original spectrum without the baselines points manually set and a subsequent concave rubberband correction. The values of 50 baseline points and 60 iterations were used.

2.4. Statistical Analysis. Exposures and measurements were repeated using 12 samples. Statistical analysis was carried out

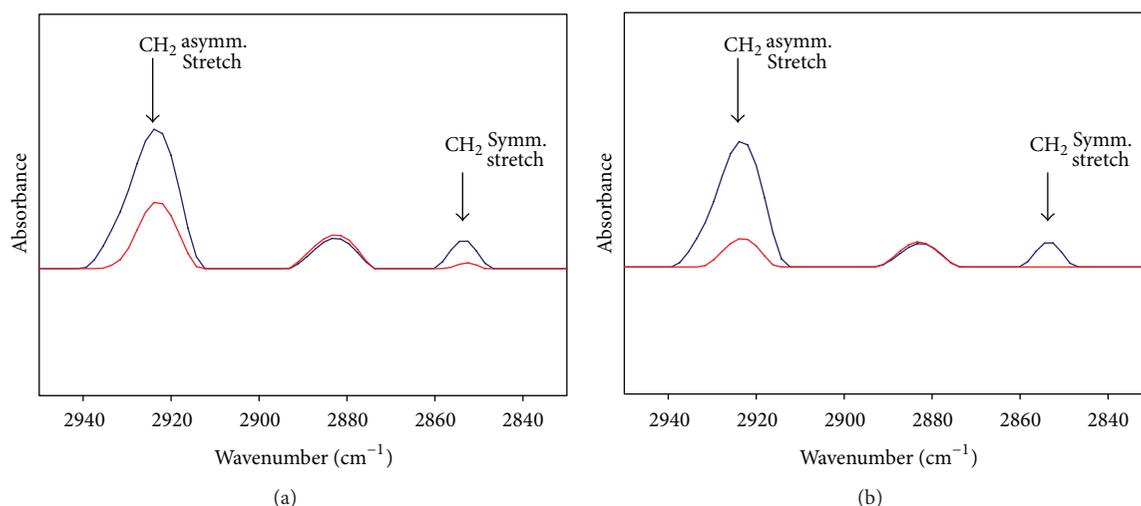


FIGURE 1: A representative spectrum in the region $3000\text{--}2800\text{ cm}^{-1}$ of PEO in bidistilled water solution. The vibration bands around 2850 and 2925 cm^{-1} (indicated by arrows) are due to the symmetric stretching and to the asymmetric stretching of the methylene group, respectively. Their intensity decreased significantly after exposure of 2 h (a) and 4 h (b) to a SMF at 200 mT . The spectra of exposed and unexposed samples are represented by red and blue lines, respectively.

using Student's t -test for comparisons between two groups, with P values less than 0.05 considered significant.

3. Results and Discussion

The vibration bands in the mid-infrared region of PEO in aqueous solution under SMFs exposure were studied using FTIR spectroscopic techniques to acquire further information about the dielectric properties of polymers as insulators from EMFs.

Different samples of $250\text{ }\mu\text{L}$ of PEO in bidistilled water aqueous solution, prepared as described in the preceding section, were exposed up to 4 hours to a SMF at 200 mT at the room temperature of 20°C . Analogue unexposed samples were used as control, at the same temperature.

FTIR spectra acquired after 2 h and 4 h of exposure showed significant changes of vibration bands in comparison to control, as described below. No further change occurred after exposure of more than 4 h.

Measurements were repeated ten times to ensure the statistical significance of results.

Representative exposed and unexposed FTIR spectra in the region $2800\text{--}3000\text{ cm}^{-1}$ obtained after 2 h and 4 h of exposure are shown in Figures 1(a) and 1(b), respectively.

Two relevant vibration bands appeared to change in that range: the bands around 2850 cm^{-1} and 2925 cm^{-1} , due to the symmetric stretching ($^s\text{CH}_2$) and to the asymmetric stretching ($^a\text{CH}_2$) of the methylene group, respectively [47–49].

The intensity of CH_2 stretching bands was observed to decrease significantly after exposure and to shift to lower frequencies; as can be observed in Figure 1, the decreases in intensity of CH_2 bands after exposure of 2 h (a) and 4 h (b) were significantly different in comparison to controls

($P < 0.05$). Exposed and unexposed samples spectra are represented by the red and blue lines, respectively.

Vibration bands of CH_2 group can be observed at higher wavelength. In fact, bending vibrations of CH_2 group are foreseen at lower frequencies than corresponding stretching vibrations, as both the stretching and bending vibrations of a molecule can be predicted mathematically (at least to a useful approximation by Hooke's law).

The strong vibration bands around 1465 cm^{-1} , represented in Figures 2(a) and 2(b), that appear splitted into two bands, can be assigned at CH_2 scissoring vibration bands [47–49]. Also this band was observed to decrease after 2 h and 4 h of exposure, confirming the decrease of CH_2 group due to exposure to SMF.

The strong band at 1349 cm^{-1} can be assigned to CH_2 wagging vibration, and it is related to the EO methylene wagging vibrations of the gauche conformation, whereas the weak band at 1250 cm^{-1} may be related to CH_2 twisting mode, both of which are characteristic of the amorphous state [50–52].

A weak increase in intensity of these vibrations was observed after exposure to SMF, as represented in the typical spectra in Figure 2.

This alteration indicated that gauche conformation of EO segments increased, suggesting a decrease of the PEO crystallization process during exposure.

Otherwise, the hydrogen-bond model has been extensively used to interpret the mechanism of the solubility of water in oxyethylene type surfactants and polymers [36, 39, 53, 54].

Hydrogen bonded C–O groups changed accordingly with methylene groups of gauche conformation, following Noda's rule [55, 56].

This hypothesis is confirmed by the observed increase in intensity of the vibration band around 1080 cm^{-1} (see

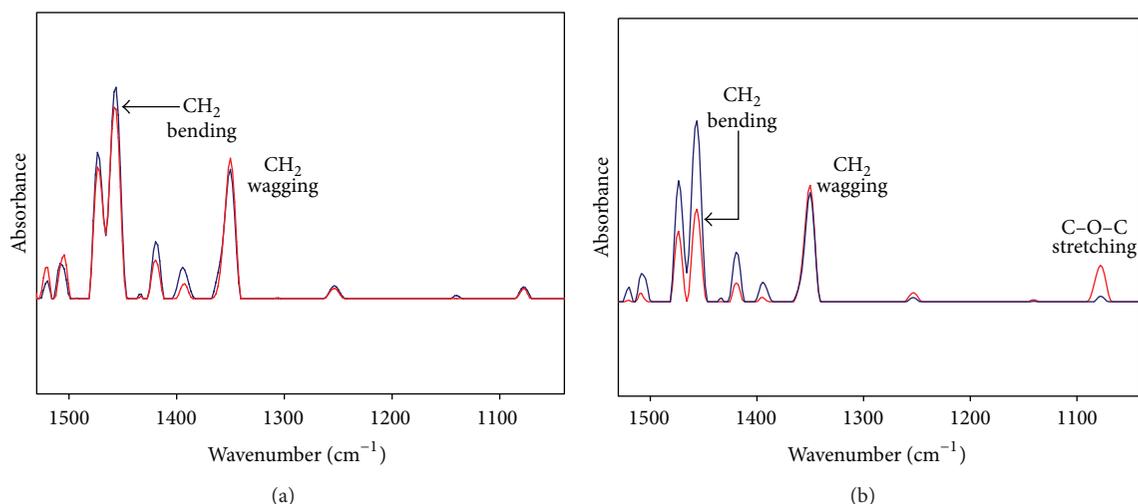


FIGURE 2: Representative spectra in the region $1500\text{--}1000\text{ cm}^{-1}$ of PEO in water solution after 2 h (a) and 4 h (b) of exposure to a SMF at 200 mT. The strong vibration band around 1465 cm^{-1} is splitted in two bands and can be assigned to CH_2 scissoring vibration bands. The 1349 cm^{-1} peak is related to the EO methylene wagging vibrations of the gauche conformation. The strong band around 1080 cm^{-1} can be assigned to the C–O–C stretching hydrated bond. Red and blue lines refer to exposed and unexposed samples, respectively.

Figure 2(b)), which can be assigned to the C–O–C stretching vibration [41, 42, 52].

The formation of a hydrogen bond Lone pair electrons on the oxygen atoms are withdrawn by the hydrogen atoms of water due to the formation of hydrogen bonds. This mechanism induces a reduction of electron density on the C–O bond which generates a red shift of the C–O–C stretching band from 1113 to about 1080 cm^{-1} [39, 52, 57].

Indeed, hydrogen bond weakens chemical bonds between atoms and therefore it produces shifts to lower frequencies and broadening in absorption bands.

The band at 1080 cm^{-1} represents the strongest band of the C–O–C linkages.

The bandwidth broadening of this vibration indicates that the polymer in aqueous solution have higher mobility [57]; its increase after exposure in comparison to unexposed samples showed that SMF increases PEO mobility in aqueous solution.

Also, C–O–C stretching band resulted in an to increase in intensity after exposure to SMF, and this change after 4 h exposure was significantly different in comparison to controls ($P < 0.01$).

In contrast, the dehydration in the micellization process could be inferred by the FTIR spectral analysis from a low shift of the frequency of the C–O–C stretching band toward higher wavenumbers.

The observed increase of the carbonyl stretching vibration C–O–C around 1080 cm^{-1} after exposure to SMF can be attributed to the increase of formation of the intermolecular hydrogen bonding that occurred in PEO aqueous solution after exposure.

This result is in well accordance with the increase of the weak band around 1140 cm^{-1} observed after 4 h of exposure to

SMF (see Figure 2(b)), that can be assigned to the dehydrated C–O–C bond [58, 59].

In this scenario, the decrease in intensity of stretching and scissoring vibrations of the methylene group that was observed after exposure to SMF can also be explained assuming that an unfolding of PEO chain occurred after exposure.

The application of a magnetic field on liquid-crystalline materials induces alignment of polymer molecules chains along the direction of a SMF [60].

Indeed, magnetic orientation can be applicable to materials such as macromolecules like polymers, composed by a relevant number of coupled molecules, so that a magnetic torque can induce molecular orientation effects [61].

Molecular dynamics simulations for the polyethylene chain was performed by [62] to study the polymer chain unfolding and reorientation processes under a high SMF, showing that it facilitates the unfolding of the flexible polyethylene chains and their reorientation in its direction.

Indeed, it was showed by [62] that the extra potential energy gained through the magnetic annealing of the chains can induce the reorientation of the polyethylene chains toward the direction of the magnetic field.

This “induced-unfolding” of PEO chain contrasts the micellization process through which the hydrophilic head of the polymer is in contact with solvent, and the hydrophobic tail (represented by the CH_2 group) is sequestered within the micelle core.

In fact, as water is added to form PEO aqueous solution, the hydrophobic block CH_2 is insoluble and collapses to form the micelle core, and it is encapsulated into the micelles.

Surfactants molecules which contain more than 16 $-\text{CH}_2$ groups are stated to coil around themselves [63]. This mechanism produces aggregation of unimers, and successively the

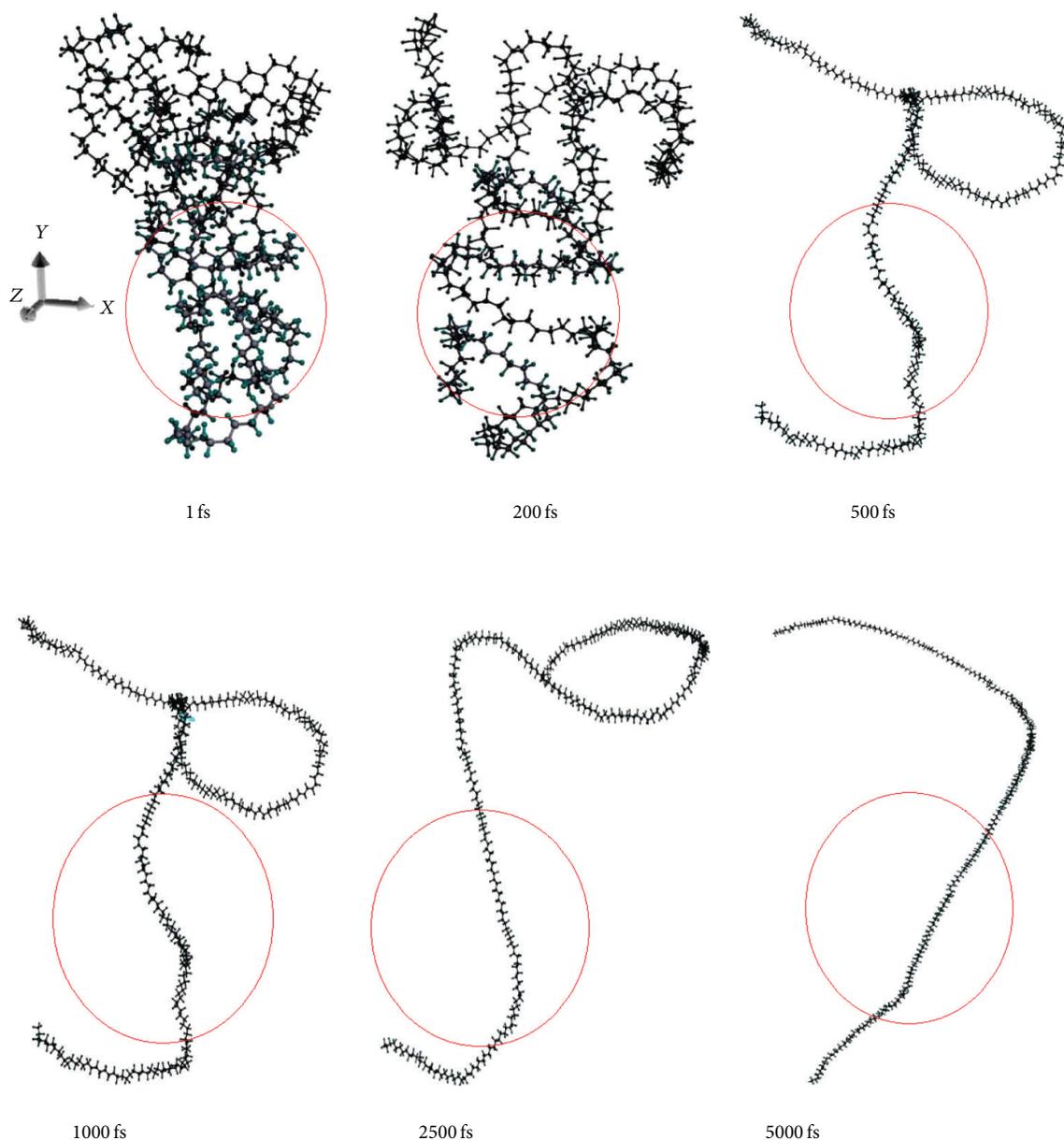


FIGURE 3: Molecular dynamics simulation of reorientation of polyethylene chains under a high magnetic field at different time steps. The red circles schematize a fixed volume to highlight that the population of CH₂ decrease as a function of time exposure. With kind permission of Taylor & Francis Ltd (<http://www.tandfonline.com/doi/abs/10.1080/08927020600887781>).

polar conformation of EO segments tends to transform to nonpolar conformation.

The dehydrated methylene groups approach through the hydrophobic interaction to form hydrophobic cores, leading to a further breaking of the hydrogen bonding between C–O and the water, and micelles are formed proportionally to the temperature value [64].

The micellization of PEO in water solution produces an increase of density of CH₂ within a fixed volume, whereas the reorientation of PEO chain generated by the applied SMF induces its demicellization and the consequent decrease of CH₂ density within the corresponding volume.

A decrease in intensity of CH₂ vibration bands in the FTIR spectra is expected, corresponding to PEO demicellization, because the oscillator strength of a given spectral transition depends on a number of factors including population or occupancy. Hence, the modeling of PEO reorientation induced by exposure to SMF is in agreement with the decrease in intensity of CH₂ stretching and bending vibrations observed after exposure, that can be explained by the decrease of population of CH₂ within a fixed volume, well represented in Figure 3 (derived from the simulation of [62]).

In this scenario, the increase of formation of the intermolecular hydrogen bonding in PEO aqueous solution,

observed after exposure to SMF, can find its explanation as well, because the reorientation of PEO chain under SMF can increase the surface contact of the hydrophilic head with aqueous solvent.

FTIR spectra of exposed samples were acquired 3 h after the end of exposure to SMF, showing that reversibility in the molecular vibrations of PEO occurs when the SMF is removed.

4. Conclusion

Exposure to a SMF at 200 mT of samples of PEO in aqueous solution induced its demicellization as evidenced by FTIR spectroscopy analysis.

Indeed, a significant decrease in intensity of symmetric and asymmetric stretching CH_2 vibration bands around 2925 and 2850 cm^{-1} , respectively, was observed after 2 h and 4 h of exposure. The decrease of CH_2 group was confirmed by the decrease of the scissoring vibration bands around 1465 cm^{-1} . This result can be attributed to a demicellization of PEO in water due to the reorientation of PEO chain induced by the applied SMF.

An increase of methylene wagging vibration at 1349 cm^{-1} was observed after exposure to SMF, that can be related to a decrease of PEO crystallization process during exposure, in agreement with previous results.

The C–O–C stretching band around 1080 cm^{-1} was observed to increase significantly after 4 h of exposure to SMF; and this result can be attributed to the increase of intermolecular hydrogen bonding occurred in PEO aqueous solution, in agreement with the demicellization process.

These experimental results are in agreement with molecular dynamics simulations for the polyethylene chain performed by other authors.

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