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Acoustic Levitator Power Device: Study of Ethylene-Glycol Water Mixtures

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Abstract. Acoustic levitator power device is formed by two vertically and opposed high output acoustic transducers working at 22 kHz frequency and produces sound pressure levels of 160 dB. The acoustic waves are monitored from an oscilloscope using a signal amplifier. The ability to perform contactless measurements, avoidance of undesired contamination from the container, are some of advantages of this apparatus. Acoustic levitation can be also used for sample preparation of high concentrated mixtures starting from solutions. In the present paper, an acoustic levitator power device is employed to collect data on levitated water mixtures of Ethylene Glycol (EG) which are then analysed by Infra-Red spectroscopy. The study allows to follow the drying process versus time and to obtain a gel-like compound characterized by an extended chemical crosslinking.

1. Introduction

Sample preparation and sample experimental investigation can take advantage by the use of contactless techniques. In particular levitation techniques eliminate container interactions and reduce contamination allowing to study samples with a high degree of control. By eliminating contact with container walls, sources of extraneous nucleation are kept away and it becomes possible to cool liquids to a large extent below their equilibrium melting point; moreover, due to the lower percentage of heterogeneous nucleation sites, levitated super-cooled liquids are expected to be more stable [1-4]. From a general point of view, levitation techniques enable to beget an opposing force to the gravity to keep the sample suspended. Several levitation techniques are commonly employed, such as optical, electro-magnetic, electrostatic, gas-film, aerodynamic and acoustic levitation [1]. Optical levitation is generated by an optical trap that is formed by a focused laser beam with an objective lens of high numerical aperture. A dielectric particle will experience a force due to the transfer of momentum from the incident photons. Electro-magnetic levitation, mostly suitable for electrically conductive materials [1], is generated by a radio-frequency field (≈ 150 kHz), produced by a coil, which induces Foucault currents in the sample. Foucault currents interact with the magnetic field of the coil causing a force that counteracts gravity. Electro-static levitation is applicable for electrically charged samples which are levitated in an electrostatic field generated between two electrodes. One disadvantage is connected with the experimental setup complexity which hinder its use in combination with many other techniques. Gas-film levitation enables the levitation of an object against gravitational force by floating on a thin gas film through a porous membrane; this technique allows to levitate a large quantity of sample. The sample-membrane closeness hampers the use in association with many techniques. Aerodynamic levitation, has proved to be a powerful and versatile technique for studying



highly reactive liquids. The basic idea is to circulate levitation gas through a nozzle onto the sample from below in order to counteract gravity and lift it above the nozzle. It has the outstanding advantage of supporting any type of material ranging from insulators through semiconductors to metals. Acoustic levitation, is applicable to suspend small objects through an acoustic radiation force. In particular an acoustic standing wave is generated between a transducer and a reflector. The samples are levitated against gravity by the pressure forces and tend towards stable equilibrium positions close to the acoustic nodal points. Acoustic levitation has the advantage of not requiring any specific physical properties of the sample, such as a specific electrical charge, a certain refractive index or transparency.

Acoustic levitation seems to be a very promising technique for the study of hydrogen-bonded systems of biophysical interest, such as aqueous solutions [5-7], disaccharides [8-16], proteins [17-18] and polymers [19-24]. Experimental tests have been fruitfully performed for these classes of systems where it is fundamental to address the experimental investigations starting from highly diluted to highly concentrated mixtures; however in the present work only the results obtained for EG aqueous solutions are reported.

2. Acoustic Levitator power device

2.1. Levitation forces

In an acoustic levitator, an acoustic wave which is generated by a piezoelectric crystal is reflected creating a standing wave when the distance between transducer and reflector is an integral multiple of the half wavelength. Pressure nodes and anti-nodes appear at fixed points separated by a distance of $\lambda/2$. Small liquid samples can be levitated in the vicinity of the pressure nodes. The levitation forces have influence on the droplet shape. While the axial forces are mainly responsible for compensating the gravitational force, the radial forces hold the sample in the pressure node. The axial and radial levitation forces lead to a deformation of the sample.

2.2. Experimental set-up

The employed experimental set up, includes a single-axis acoustic levitator consisting of a vibrating source (i.e. transducer) generating ultrasound and a concave surface reflector. This device is designed and manufactured by Materials Development, Inc (MDI) in Illinois (US) and a full description could be founded elsewhere [4]. The produced acoustic waves were monitored from using an oscilloscope. In order to reduce instabilities in the levitated sample acoustic absorbing foam disks were glued onto the face of the transducer. Liquids samples with sizes ranging between 1 and 3 mm were introduced in the acoustic field using a micropipette injection. A video camera was used to record images and video and to help the sample positioning. EG of 99,8% purity, manufactured by Sigma-Aldrich and double-distilled water were used in this study [5-24]. The solutions were prepared by mass, using an analytical balance with $\pm 0.01\text{mg}$ accuracy. The surface tension measurements were carried out using a standard thermostated stalagmometer, which was calibrated with distilled water ($\sigma = 72.8 \text{ mN/m}$ at $T = 20^\circ\text{C}$). A constant temperature water bath was used to control the temperature of the solutions to an accuracy of $\pm 0.1^\circ\text{C}$. Measurements for each solution were repeated four times. Ancillary density measurements were performed by standard pycnometer technique. A FTIR Vertex 70 v by Bruker Optics spectrometer was employed to collect absorption spectra. Each spectrum is composed by 128 interferograms which furnished a spectral resolution of 4 cm^{-1} . Spectra corrections for atmospheric water background, baseline and area normalization were performed. OPUS/Mentor software interface was used to process data.

2.3. Results and discussion

The surface tension of a liquid mixture is an important property, which plays an important role in affecting the mass and heat transfer at the interface. In the chemical field applications, it determines the product quality (i.e. coatings, paints, detergents, cosmetics and agrochemicals) and affects different production processes such as catalysis, absorption, distillation and extraction. Measured

surface tensions of pure EG as a function of temperature (figure 1) and of water mixtures at $T=25^\circ\text{C}$ (figure 2) show that the surface tension of pure EG decreases linearly with the temperature and in a quadratic way with concentration.

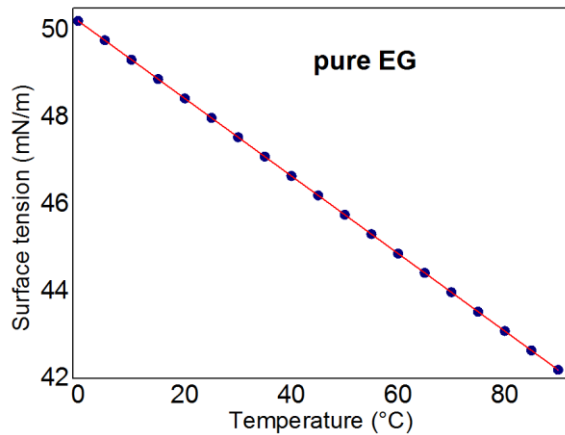


Figure 1. Surface Tension of pure EG as a function of temperature.

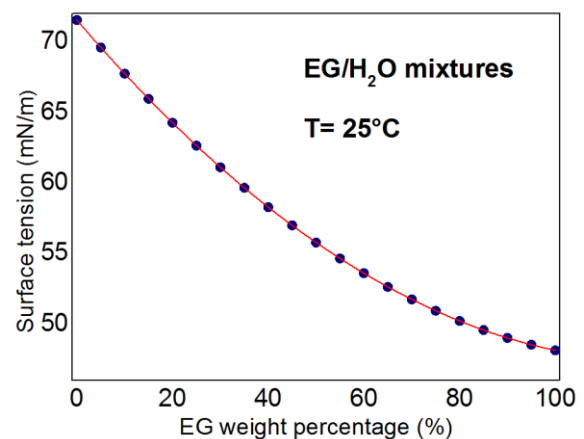


Figure 2. Surface Tension of EG/H₂O mixtures at room temperature ($T=25^\circ\text{C}$) as a function of concentration.

It may be suggested that the presence of extensive intermolecular H-bonding between EG molecules and EG and water molecules could be responsible for decreasing surface tension with increasing temperature and EG concentration [5-24]. In figure 3 the OH stretching contribution area of non levitated aqueous solutions at $T=20^\circ\text{C}$ are reported. As it can be seen the intensity of the OH stretching contribution decreases with the increasing of the EG concentration.

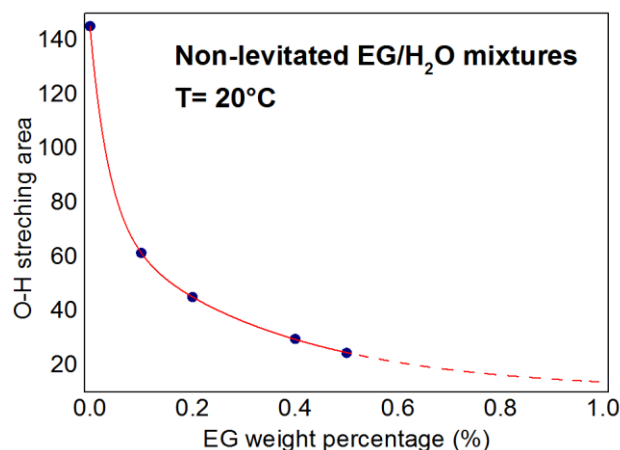


Figure 3. O-H stretching area of absorbance multiple spectra of non-levitated EG aqueous solutions at $T=20^\circ\text{C}$.

In figure 4 the InfraRed absorbance spectra of levitated EG, in aqueous solutions at $T=20^\circ\text{C}$ for the initial concentration value of $\phi_w = 0.50$ as a function of time are reported, i.e. at 15m, 30m, 45m and 60m.

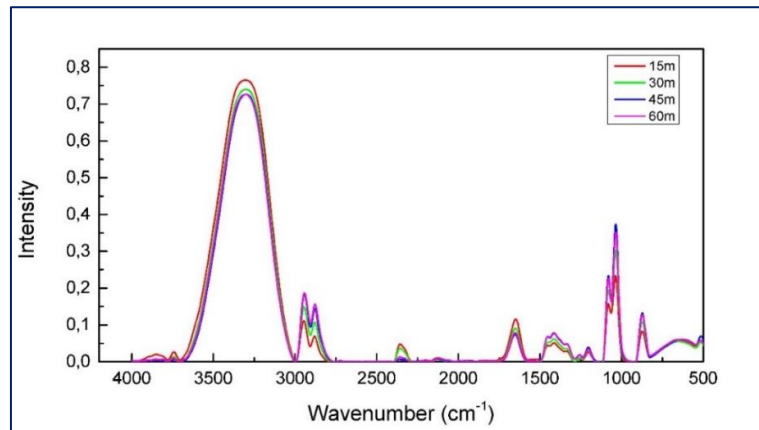


Figure 4. IR absorbance spectra of levitated EG aqueous solutions at $T=20^{\circ}\text{C}$ for the concentration values $\phi_w = 0.50$ as a function of time, i.e. 15m, 30m, 45m, 60m and 80m. This last is not reported because it is superimposed to that obtained at 60m.

In figure 5 the OH stretching contribution area of levitated aqueous solutions at $T=20^{\circ}\text{C}$ are reported as a function of time. As it can be seen from figure 5, the intensity of the OH stretching contribution decreases with the increasing of the levitation time, reaching a plateau value after 60 minutes. From the comparison between the data reported in figures 3 and 5 one can infer that the concentration value is 0.647 after 15m, 0.787 after 30m, 0.918 after 45m and 0,953 after 60m. In the insert the extrapolated values of the concentration as a function of time are reported.

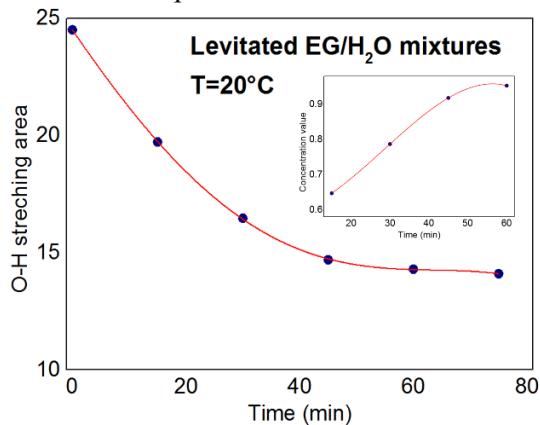


Figure 5. O-H stretching area of IR absorbance spectra of levitated EG aqueous solutions at $T=20^{\circ}\text{C}$ for the concentration values $\phi_w = 0.50$ as a function of time. In the insert the extrapolated values of the concentration as a function of time are reported.

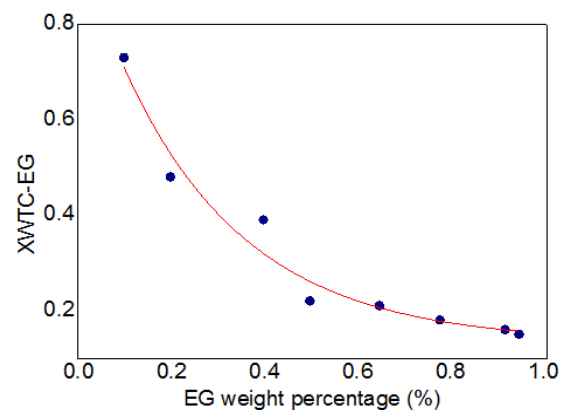


Figure 6. $XWTC_{EG}$ versus concentration ϕ , taking as reference spectrum the one at $\phi=0.10$.

2.3.1. Wavelet cross correlation analysis. WT analysis [25-33] allows the convolution of a signal $f(t)$ onto continuous functions $\Psi_{s,d}(t)$ derived from the translations and dilations of a mother wavelet $\Psi(t)$: $\Psi_{s,d}(t) = \frac{1}{\sqrt{s}} \Psi\left(\frac{t-d}{s}\right)$, s and d being real parameters and where is $s > 0$. Sets of these functions satisfy the admissibility condition, i.e. $\Psi(t)$ continuous functions with a compact support, are called wavelets [25-33]. The convolution of $f(t)$ with $\Psi_{s,d}(t)$, at scale s and delay d , is the WT of the signal $W^f(s, d)$:

$$W^f(s, d) = \int_{-\infty}^{+\infty} f(t) \frac{1}{\sqrt{s}} \Psi^*\left(\frac{t-d}{s}\right) dt \quad (1)$$

Given two data sets $f(t)$ and $g(t)$, with WT $W^f(s, d)$ and $W^g(s, d)$ respectively, the cross-wavelet (XWT) spectrum is defined as: $W^{fg}(s, d) = W^f(s, d)W^g(s, d)^*$, where the operator $*$ denotes the complex conjugate. The cross-correlation coefficients can be represented as:

$$W^f(s, d) = |W^f(s, d)|\exp(\phi^f(s, d)) \quad (2)$$

$|W^f(s, d)|$ being the wavelet amplitude and $\phi^f(s, d)$ the absolute phase:

$$\phi^{fg}(s, d) = \phi^f(s, d) - \phi^g(s, d) = \tan^{-1} \left(\frac{\text{Im}(\langle s^{-1}W^{fg}(s, d) \rangle)}{\text{Re}(\langle s^{-1}W^{fg}(s, d) \rangle)} \right) \quad (3)$$

Figure 6 shows the behavior of XWTCEG coefficients versus concentration. The XWT parameters, evaluated between each couple of the registered data both as a function of concentration and time, taking as reference spectrum the one at a concentration value of 0.10 show the same behavior registered in figure 3 [25-33]. This result confirms the validity of the approach for extracting the concentration value.

2.4. Conclusions

In the present paper, acoustic levitation technique has been used in combination with InfraRed spectroscopy to collect data on levitated water mixtures of EG in a wide concentration range; the study also includes ancillary surface tension and density data determinations. The obtained experimental findings together with their analysis allow to extract information on the concentration values of the levitated systems. Such work shows that hydrogen-bonded systems can be effectively investigated by acoustic levitation; further results obtained with such a technique will be published in a future work.

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