



# Ultrasound stimulus inducing change in hydrogen bonded crosslinking of aqueous polyvinyl alcohols



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## ABSTRACT

The effect of ultrasound (US) stimulation on the shear viscosity of aqueous polyvinyl alcohol (PVA) solution was studied when the solution was exposed to US at 23, 43, 96, and 141 kHz. The US stimulus showed a marked decrease of the shear viscosity of the solution in the order of 43 > 96 > 23 > 141 kHz, respectively, under US power dissipation of 8.5, 8.9, 8.9, and 8.8 W. Subsequently, when US exposure was stopped, the shear viscosity of PVA reverted to its original value. The US stimulation was analyzed with the US power transmitted through the PVA aqueous media. Furthermore, FT-IR spectra measured at different durations of US exposure, suggest that hydrogen bonds in the PVA segments were broken by the US exposure. We conclude that structural changes of the hydrogen bonded crosslinks of PVA were induced to include water molecules for the re-forming of crosslinks of aqueous PVA.

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## 1. Introduction

Intelligent materials of many kinds have been developed with properties that can be changed considerably by an external stimulus such as light, heat, or electricity. Extension of these technologies to several applications has been reported [1], because such materials can respond to external signals and can induce a distinct reaction to the outer environment. The well-known ultrasound (US) technique has been applied in various fields such as engineering and medicine. The US transmitted through any substance, influences several properties of materials. Low-frequency US, which shows effectiveness with common applications, is used mainly to dissolve substances or to homogenize mixtures. In addition, US has earned interest for use in various operations for degasification and particle size reduction [2,3]. For food engineering [4,5], sonochemical effects of high-frequency US have been applied to degradation of organic material [6]. Using US triggers, our previous works have demonstrated that US can serve as an external stimulus in copolymer microgels of *N*-isopropylacrylamide (NI-PAM) and acrylic acid (AA) using methylenebisacrylamide as a crosslinker [7]. The US exposure to spherically polymeric microgels changed the hydrodynamic diameter in water medium and also the volume phase-transition temperature of the microgels. In addition, under the US environment, a significant change of the shear viscosity was observed in Al<sub>2</sub>O<sub>3</sub>/polyacrylic acid (PAA) slurry, NI-

PAM copolymers with PAA, carrageenans and water soluble polymers in each aqueous medium [8–11]. These were attributable to the breaking of hydrogen bonding crosslinks of their polymers under US exposure in aqueous medium.

Regarding the decrease of shear viscosity from US exposure, US absorption might have induced polymer change, especially in hydrogen-bonded crosslinks. To date, absorption coefficients and velocities for different non-ionic polymers solutions such as poly(ethylene glycol(PEG)) and PAA have been reported for high-frequency US of 2–130 MHz [12–17]. However, reports have described no results for lower frequency. It remains unclear, whether a substantial decrease of the shear viscosity was induced by US exposure to aqueous medium or not, because US could affect the aqueous polymeric systems which contain hydrogen bonding. The present study examined the viscosity properties of polyvinyl alcohol (PVA) after exposure to US with different frequencies. In fact, PVA is one of the most widely water soluble polymers used in the industry and also widely study, because their properties can be approach in many fields such as emulsifier, stabilizer for colloid suspensions, coating in the textile, and as an adhesive. Its mechanical properties are strongly linked to the molecular weight and the degree of hydrolysis, the reason of its behavior is due to the hydrogen bond interactions [18–22]. Therefore, US effects observed in this study are expected to be meaningful for industrial applications. For this study, PVA of four molecular weights were chosen to analyze US effects on the shear viscosity of the aqueous solution using US stimulus of 23, 43, 96, and 141 kHz. The US effects on the PVA aqueous solution were observed using FT-IR spectroscopy and observations of shear viscosity.

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## 2. Experimental

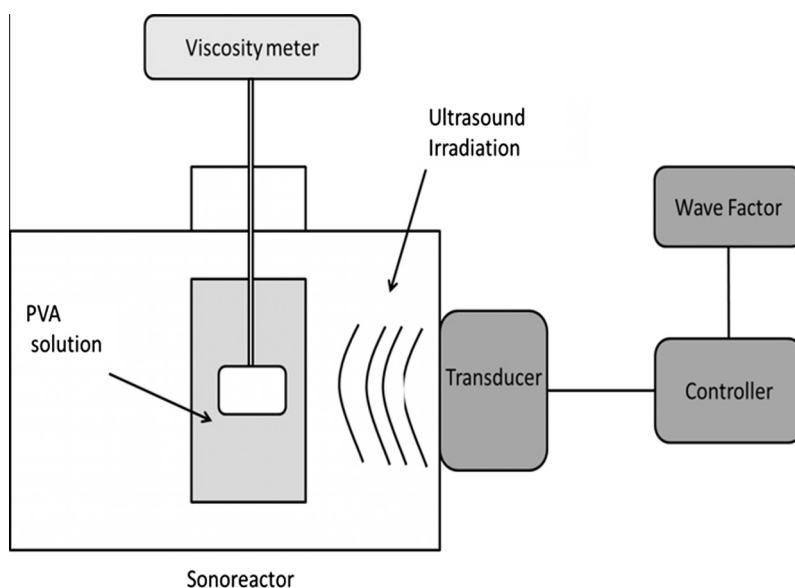
### 2.1. Materials and sample solution preparation

The following detailed information is related to all PVAs used in experiments. The four PVAs were with catalog values of different degrees of polymerization of 13000–21000, 31000–50000, 8200–85000, and 75000, respectively, for PVA13, PVA31, PVA217, and PVA117. PVA13, PVA31, and PVA217 had similar hydrolysis degrees in the range 86–88%. PVA117 had a hydrolysis degree of 98–99%. PVA13 and PVA31 were purchased from Sigma–Aldrich; PVA217 and PVA117 were supplied by Kuraray Co. Ltd. (Japan). Water used to prepare PVA solutions was distilled and purified using an ion exchange column. In the cases of PVA217 and PVA117, because their high molecular weights, these solutions were heated at 60 °C to be totally dissolved and the obtained transparent solutions were slowly cooled to room temperature and kept at this temperature for one night to settle eliminate air bubbles. Each aqueous PVA solution was prepared with 15 wt.% concentration.

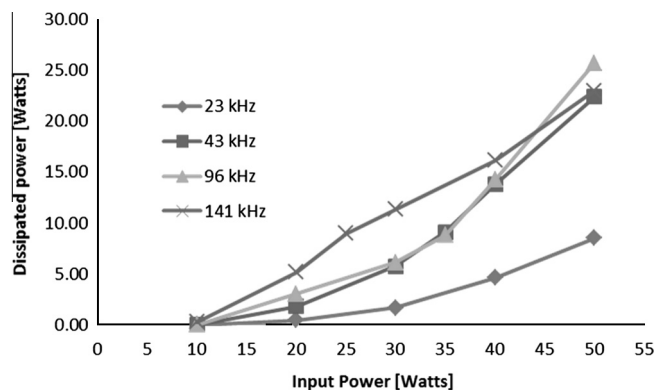
### 2.2. Ultrasound equipment and its evaluation

A sonoreactor device (HSR-305R; Honda Electronics Co. Ltd., Japan) was used. Scheme 1 shows a side view of the sonoreactor. A stainless steel water bath ( $8.5 \times 13.5 \times 13$  (cm<sup>3</sup>)) had a 3-cm-diameter transducer on the side wall. The US frequencies were 23, 43, 96, and 141 kHz with different variable intensities of 10–50 W, which were controlled with a wave factory (WF1943B multifunction synthesizer; NF, Japan). A calorimetric study was carried out to calculate the power dissipated in the water bath [23–25]. The temperature ( $T$ ) was recorded against time ( $t$ ) at 300 s intervals using a thermocouple placed in the middle of the water bath. From the  $T$  versus  $t$  data, the temperature rise at zero time,  $\Delta T/\Delta t$ , can be estimated by constructing a tangent to the curve or by curve-fitting the data to a polynomial in  $t$ . The ultrasonic power dissipated entering to the system was determined by substituting the value of  $\Delta T/\Delta t$  into Eq. (1).

$$D_p = mC_p \frac{\Delta T}{\Delta t} \quad (1)$$



**Scheme 1.** US experimental setting for the measurement of the shear viscosity in polymer solution.



**Fig. 1.** Dissipated power for all of the US frequencies used.

where  $C_p$  is the heat capacity of water ( $4200 \text{ J kg}^{-1} \text{ K}^{-1}$ );  $m$  represents the mass of the solvent used (kg). Fig. 1 shows the dissipated and input US power at different frequencies of 23, 43, 96, and 141 kHz. These data showed that the input power could be adjusted by controlling the dissipated power between 10–50 W for almost zero to 6–25 W. Results showed that the power dissipated in the US water bath was 8.5, 9, 8.7, and 8.8 W for respective power values of 50, 35, 35, and 25, respectively, for 23, 43, 96, and 141 kHz. We used a US power dissipated in the range of 8.5–9 W for comparison of US effects of different frequencies. In addition, the US profiles were monitored by US probe instrument (Olympus, Co.) connected to an oscilloscope (TDS3012, Tektronix Co.). The resultant relation of US intensity with time could compare in each US frequency and confirmed to be similar magnitude in the US, which was exposed to the PVA solution.

### 2.3. Shear viscosity measurement of PVA aqueous solutions

The scheme 1 shows the measurement setup, includes a Brookfield rotating viscometer (Tokyo Keiki Inc.), which facilitates shear viscosity measurements in the water bath after the aqueous PVA solution was exposed to US. The shear viscosities of the aqueous solution obtained before and after the US exposure were compared

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