Original Article

Intensification of Fenton Process by Ultrasonic Waves in Humic Acid Degradation from Aqueous Solutions

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Abstract

Aims: The present study aimed to evaluate the degradation efficiency of humic acid (HA) as a persistent pollutant from the aqueous solution by the Fenton oxidation process intensified with ultrasonic (US) waves. **Materials and Methods:** A pilot plant equipped with a magnetic stirrer and bar and US device was constructed. The influent with a prespecified concentration of HA was transferred into the reactor and sampled with time intervals. The effect of influencing parameters such as solution pH, reaction time, concentration of H_2O_2 and Fe, US power, and initial HA concentration on degradation efficiency was systematically investigated. **Results:** The results showed that by increasing the reaction time from 5 to 10 min, the degradation efficiency was significantly enhanced (10%), while the degradation was not apparently improved by progressing the reaction time to 55 min. At an optimal solution of pH of 3, the induction of US increased the degradation efficiency of HA by about 25%. Overall, the increase in the concentration of H_2O_2 and Fe led to degradation efficiency improvement, 12% and 15.9%, respectively. **Conclusion:** Based on the obtained results, the intensified-Fenton process by the US can be proposed as a fact and relative process for the degradation of HA.

Keywords: Dissolved organic carbon, Fenton process, humic acid, hydrogen peroxide, ultrasonic waves

Introduction

With the rapid development of industry and economy, pollution and lack of water resources have caused serious problems for the sustainable development of human society. Therefore, many efforts have been made to develop efficient technologies to reduce water pollution.[1] Humic acid (HA) is an important natural organic matter (NOM) compound that is widely present in natural surface water, groundwater (drinking water), municipal wastewater, and landfill leachate.[2,3] Meanwhile, the presence of HA in drinking water is of great importance, because HA has adverse effects on water quality by creating an unpleasant color and taste in water.[2,4] More importantly, HA can react with active chlorine in water treatment plants and cause the formation of more than 600 types of carcinogenic and toxic byproducts of disinfection, including trihalomethanes and haloacetic acids.[5,6] HA also causes an increase in the consumption of disinfectants in water purification, disruption in the coagulation process, reduction in the efficiency of membrane processes, and regrowth of microorganisms in the process of drinking water sanitation.[7] In addition, due to the large amounts of carboxyl, phenolic hydroxyl, and carbonyl groups, HA has the ability to complex strongly with metal ions, thus increasing the mobility of metal species in the aqueous environment. As a result, the removal of HA is very important to protect the safety of drinking water.[5,6]

Among the methods of removing these substances from aqueous environments, we can refer to the process of chemical coagulation and sedimentation, oxidation, surface absorption, ion exchange, and filtration by various membranes.[8] Advanced

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oxidation processes (AOPs), based on the generation of radicals with high oxidation power, are a promising solution for the removal of emerging pollutants of concern and the inactivation of pathogenic agents.[9] Among AOPs, Fenton and Photo-Fenton are preferably used in aqueous media because the hydroxyl radical reacts with a diffusion-controlled rate constant with most organic solvents.[1,8] As a photocatalyst, TiO_2 has attracted much attention due to its excellent chemical inertness, nontoxicity, strong oxidation ability, high oxidation efficiency, low cost, and environmental friendliness.[10] Studies have shown that in this process, hydroxyl radicals are produced from the reaction between iron salts and hydrogen peroxide; the reaction is shown in equation 1. Furthermore, the catalytic decomposition of hydrogen peroxide continues with the production of hydroxyl radicals according to equations 2 and 3.

$$Fe_2^{+} + H_2O_2 \rightarrow Fe_3^{+} + OH^{\bullet} + OH^{-} \quad (1)$$

$$Fe_3^{+} + H_2O_2 \rightarrow Fe_2^{+} + HO_2 + H^{+} \quad (2)$$

$$HO^{\bullet} + H_2O_2 \rightarrow HO_2^{\bullet} + H^{+} \quad (3)$$

In this way, the radical chain oxidation starts with the hydroxyl radical, which has no selectivity toward organic compounds.[11] Fenton process due to advantages such as high oxidation power, fast oxidation, reduction of energy consumption due to the catalytic property of iron, simplicity of operation, ability to transport materials, nontoxicity and easy purification of iron ions, and lower production of toxic and dangerous by-products, compared to other advanced oxidation methods, it is considered one of the most effective processes for removing organic pollutants from aqueous solutions.[12,13] Ultrasonic (US) wave combined with heterogeneous AOPs (ultrasound/metal ions, ultrasound/metal oxides, and ultrasound/photocatalysis) and homogeneous AOPs (ultrasound/ozone, ultrasound/H_2O_2, and ultrasound/persulfate) was used for the degradation and also mineralization of organic pollutants.[14] One of the main disadvantages of Fenton's reagent is the precipitation of large amounts of ferric ion sludge, which requires the continuous addition of iron ions to the reaction medium to continue the reaction. These disadvantages can be solved using the photo-Fenton reagent, which is a cyclic process that regenerates Fe^{2+} ions.[15] Another disadvantage of this process is the high price of $H_2O_2^{\bullet}$.[16]

Studies have shown that the use of US waves destroys the molecular structure of HA and increases its biodegradability; hence, the US process is one of the new methods in the water purification processes that have been considered.[17] Considering that this method is a physicochemical process and uses US waves and Fenton, it does not have the possibility of producing side compounds, and it is one of the healthiest removal methods compared to other chemical methods in terms of health.[18] The efficiency of the Fenton process in the removal of organic pollutants from water depends on various factors such as pH, hydrogen peroxide concentration, ferrous ion concentration, and the initial concentration of organic matter.[19]

In this study, the effect of the above factors on the performance of the Fenton process intensified with the US in the removal of HA was investigated. Two goals were achieved in this work; the first goal was to introduce the technology of combining the Fenton process with US waves, and the second goal was to effectively remove HA using this method. In the following, the effect of different factors on the removal of HA is discussed in this study.

Materials and Methods

A pilot has been used to conduct the experiment, which consists of a magnetic stirrer, magnet, US device, and a pilot chamber. Figure 1 shows the schematic of the used pilot. The pilot structure is made of plexiglass with the dimensions of 10 cm × 10 cm × 20 cm (L × W × H) with a working volume of 1 L. The stock solution of HA (1000 mg/L) was prepared with dissolving of 1 g of HA sodium salt (CAS number 68131-04-4, Sigma-Aldrich, St. Louis, Missouri, United States) in 1 L of deionized water, and other working solutions were prepared with the dilution of stock solution. To ensure complete mixing of the contents inside the reactor, a magnetic stirrer was used, and to induce US waves, the tip of the transmitter was placed 3 cm below the water surface. Furthermore, the reactor was placed in a water bath for temperature control, and the pH adjustment was conducted with H_2SO_4 and NaOH (0.1 M) (Merck, Germany). The US device (Bandelin Sonopuls HD 3200 homogenizer, Germany) used has a frequency of 20 kHz, a power of 50–150 W, and a working voltage of 230 V (±10%).

In this study, to investigate the degradation experiments, the synthetic solutions of HA were prepared after adjusting their pH in the range of 4–9. At different reaction times, the reactor was sampled, and the amount of absorption was measured by spectrophotometer (DR5000 Spectrophotometer, Hach Company, 2005).

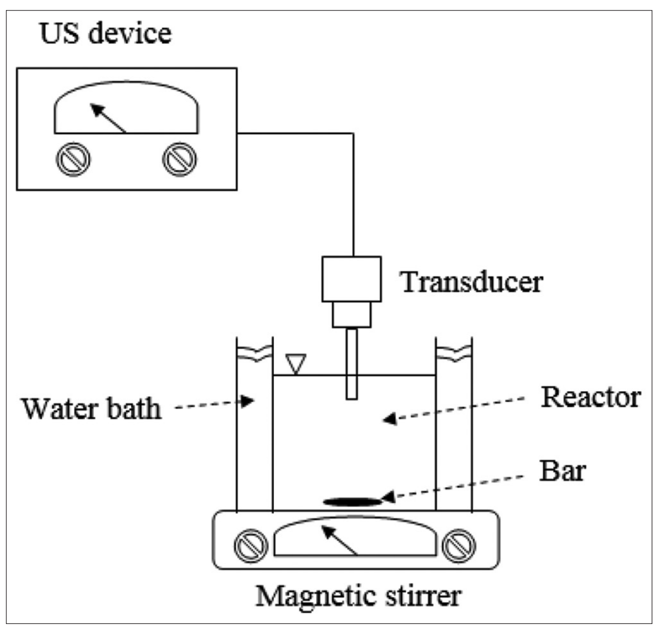

Figure 1: Schematic of the used pilot. US: Ultrasonic

Results

To optimize the Fenton process by US waves, the effect of the main operation of parameters, including solution pH, reaction time, dose of H_2O_2 and Fe^{2+}, initial HA concentration, and intensity of US waves, was systematically investigated, and the obtained result is shown in Figures 2-7.

Discussion

Effect of pH

The pH of the solution significantly affects the efficiency of the Fenton oxidation process, as it controls the dose of Fe^{2+} and the production of OH in the solution.[20] In this study, the effect of solution pH on the HA removal efficiency by the Sono-Fenton process was studied in the 3–9 range. As seen in Figure 2, the highest removal efficiency is about 46% at pH = 3. Furthermore, with increasing pH, the removal efficiency decreases, and at pH of 9, the removal efficiency reaches the lowest value, equal to 3.34%.

As can be seen in Figure 2, at pH higher than 3, the HA removal efficiency decreased significantly due to the formation of $Fe(OH)_2$ and $Fe(OH)_3$ deposits, which reduced the production of OH radicals and subsequently reduces their catalytic activity. In addition, at pH higher than 3, H_2O_2 also decomposes into oxygen and water.[21] In alkaline pH, the HA removal efficiency was very low, which is due to the OH scavenging effect. Furthermore, at high alkalinity levels, the hydroxyl radical decomposes into an oxide ion (O), which reduces the removal efficiency.[22] In the study of Pignatello *et al*., they showed that at acidic pH, the reaction between hydrogen peroxide and ferrous ion occurs, which causes the decomposition of many organic pollutants, using the AOPs in the decomposition of organic substances by the Fenton process.[23]

Effect of reaction time

The reaction time is an important factor in the AOPs. For estimation of HA removal efficiency by Sono-Fenton as a function of reaction time, the experiments were performed at initial HA concentration: 20 mg/L, H_2O_2 dose: 50 mg/L, Fe^{2+} dose: 30 mg/L, and solution pH: 3. The variations of HA removal efficiencies as the dependence on reaction time are shown in Figure 3.

As shown in Figure 3, one peculiar feature of the rate of removal in all experimental categories is the nonuniformity of removal with respect to time. The rate of removal is much faster in the initial 10 min, and a significant fraction of total removal occurs in this period only. This indicates that higher reaction times lead to a greater reaction between HA and $^{\cdot}OH$ radicals, which results in a greater HA removal. After that, the rate of removal is constant and did not change significantly. This behavior is due to the decrease of the HA concentration during reaction time and the reduction of the $^{\cdot}OH$ radicals collision with the HA. These results are similar to the results obtained by Chakma and Moholkar,[24] who reported an increase in the removal of bisphenol A (BPA) rate corresponding to the increase in reaction time.

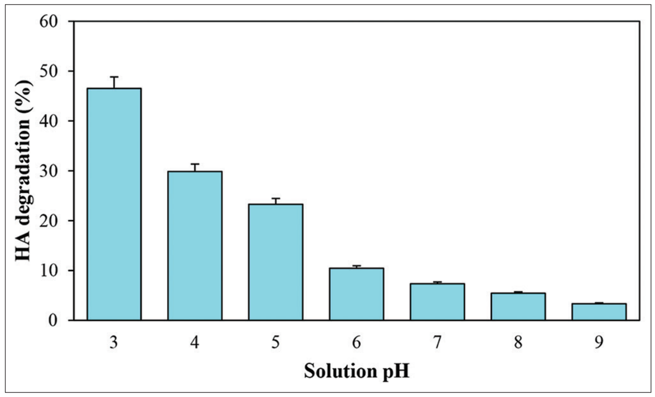

Figure 2: Effect of solution pH on humic acid (HA) degradation (HA concentration: 20 mg/L, H_2O_2 concentration: 50 mg/L, Fe^{2+} dose: 30 mg/L, and 30-min contact time). HA: Humic acid

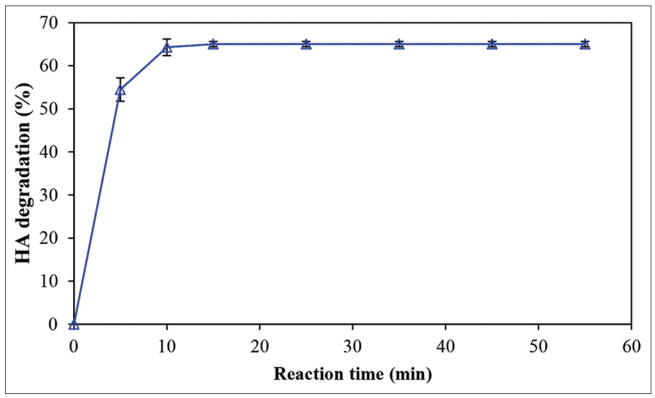

Figure 3: Effect of reaction time on humic acid (HA) degradation (HA concentration: 20 mg/L, H_2O_2 dose: 50 mg/L, Fe^{2+} dose: 30 mg/L, and solution pH: 3). HA: Humic acid

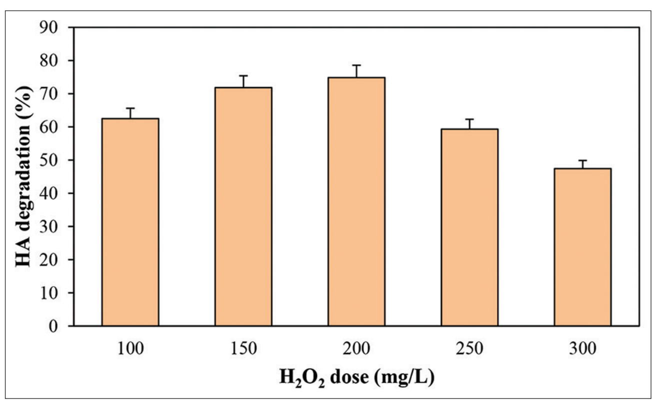

Figure 4: Effect of H_2O_2 dose on humic acid (HA) degradation (HA concentration: 20 mg/L, Fe^{2+} dose: 30 mg/L, solution pH: 3, and reaction time: 15 min). HA: Humic acid

Effect of H_2O_2 dose

The $^{\cdot}OH$ radicals are a potent oxidizing agent that reacts with organic and inorganic substances. A series of HA removal experiments were carried out to investigate the effects of H_2O_2

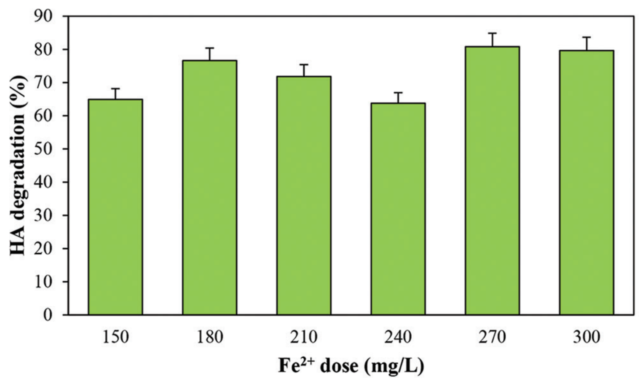

Figure 5: Effect of Fe^{2+} dose on humic acid (HA) degradation (HA concentration: 20 mg/L, H_2O_2 dose: 100 mg/L, solution pH: 3, and reaction time: 15 min). HA: Humic acid

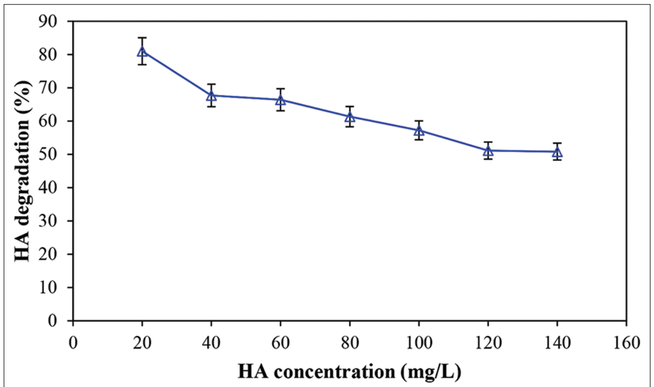

Figure 6: Effect of initial humic acid (HA) concentration on HA degradation (H_2O_2 dose: 100 mg/L, Fe^{2+} dose: 270 mg/L, solution pH: 3, and reaction time: 15 min). HA: Humic acid

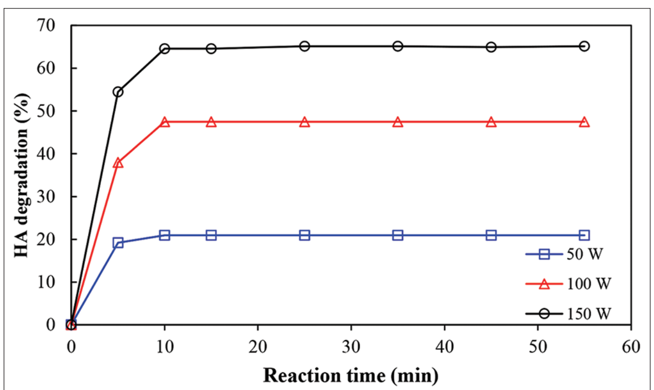

Figure 7: The role of the induction of ultrasonic waves in improving the efficiency of the process. HA: Humic acid

dose on HA removal at initial HA concentration: 20 mg/L, Fe^{2+}: 30 mg/L, solution pH: 3, and reaction time: 15 min, and the results obtained are shown in Figure 4.

As displayed in Figure 4, by increasing the dose of hydrogen peroxide from 100 to 300 mg/L, the HA removal efficiency has decreased. The obtained results can be described as follows: hydrogen peroxide plays an important role in the Fenton oxidation process, as it is the source of $^{\bullet}OH$ radicals. However, an excess amount of hydrogen peroxide not only reduces the treatment efficiency but also raises the cost of treatment, as it is the main expense.[20] Application of high H_2O_2 dose cannot significantly improve the HA removal efficiency, since excess H_2O_2 acts as free radical scavengers and would consume $^{\bullet}OH$ radicals as shown in equation 4.[25]

$$H_2O_2 + {}^{\bullet}OH \rightarrow H_2O + HO_2^{\bullet} \quad (4)$$

In the study of Wu *et al*. under the title of "Removal of HA from landfill leachate," using Fenton and chemical coagulation, they have been shown that the concentration of hydrogen peroxide higher than 320 mM reduces the efficiency of HA removal.[26] They attributed this to the quenching of hydroxyl radicals caused by the high concentration of hydrogen peroxide.[26] Murray and Parsons, in research titled "Removal of NOM from drinking water with the help of Fenton and Photo-Fenton process," reported that at a fixed concentration of ferrous ion, the efficiency of removing organic substances increases with the increase of hydrogen peroxide up to a certain concentration.[27]

Effect of different ferrous ions dose

The optimization of the Fe^{2+} dose is crucial to reduce the amount of the chemical treatment sludge and to lower the treatment cost since the Fenton process is based on the production of $^{\bullet}OH$ radicals by decomposing H_2O_2 in the presence of Fe^{2+} as the catalyst (equation 5).[20]

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^{\bullet} \quad (5)$$

To understand the role of Fe^{2+} dose on the Sono-Fenton process, the various doses of Fe^2 were used at initial HA concentration: 20 mg/L, H_2O_2 dose: 100 mg/L, solution pH: 3, and reaction time: 15 min. The effect of Fe^{2+} dose on HA removal by sono-Fenton is illustrated in Figure 5.

As seen in Figure 5, with the increase in Fe^{2+} dose, the HA removal efficiency increased, and the highest removal efficiency was observed in a dose of 270 mg/L. The increasing trend is due to that higher ferrous dosage can form more $^{\bullet}OH$ radicals through the Fenton reaction, thus leading to a higher rate of HA removal (equation 2).[28] A significant increase in HA removal efficiency was not observed at higher doses of 270 mg/L; the reason for this is that a high Fe^{2+} dose causes $^{\bullet}OH$ radicals to undergo self-extinguishment by Fe^{2+} ions and reduces the efficiency of HA removal (equation 6).[29]

$$Fe^{2+} + 4{}^{\bullet}OH \rightarrow 2\ Fe\ (OH)_2 \quad (6)$$

In the study of Wu *et al*., the concentration of 0–240 mMol of Ferro ion on the removal of HA in a fixed concentration of 160 mMol of hydrogen peroxide reported its optimal value with a removal efficiency of 79%.[26] Kitis *et al*. investigated the effects of applying hydrogen peroxide at concentrations ranging from 50 to 1000 mg/L. The study focused on scenarios where iron ions were absent. The findings revealed that under these conditions, only a 7% reduction in the initial concentration of dissolved organic matter was observed.

In the absence of iron ions, only 7% of the initial concentration of carbon has reduced dissolved organic matter.[30]

Effect of initial humic acid concentration

The effects of initial concentration on HA removal efficiency by the Sono-Fenton process at H_2O_2 dose: 100 mg/L, Fe^{2+} dose: 270 mg/L, solution pH: 3, and reaction time: 15 min are shown in Figure 6.

As seen, with increasing the initial HA concentration, its removal efficiency decreased so that the highest and lowest removal efficiency was observed for the concentration of 20 mg/L and 140 mg/L, respectively. The increase of initial HA concentration would decrease the probability of reaction between HA molecules and the $^{\bullet}OH$ radicals. In addition, the unspecific oxidation of all the molecules present in the reaction, especially the major intermediates with the $^{\bullet}OH$ radicals, may lead to side reactions and excessive consumption of the $^{\bullet}OH$ radicals.[31] These results are similar to the results obtained by Geng *et al.*,[10] who reported an increase in the HA removal corresponding to the reduction in initial HA concentration.

Effect of ultrasonic

Ultrasound power is known as an effective parameter in chemical oxidation processes. In this study, the Fenton process intensified with US waves, the effect of the power of ultrasound waves on the removal of HA, at a frequency of 20 kHz, a power of 50–150 W, and a working voltage (±10%) of 230 V was investigated, and the results are shown in Figure 7.

As seen in Figure 7, the induction of US waves plays an important role in improving the efficiency of the process. In such a way that with the induction of US waves, the removal efficiency of the process increases by about 20%–25%. This is due to the production of very active free radicals with high oxidizing power such as $^{\bullet}HO^{\bullet}$, $^{\bullet}OH$, and $^{\bullet}H$, which are created during the propagation of US waves due to cavitation. The production of intense local heat up to 5000° Kelvin and high hydrodynamic pressure up to about 180 MPa causes the removal and decomposition of organic compounds.[32] On the other hand, iron ions with US radiation can amplify US waves and increase the speed of HA removal. In addition, Fe^{2+} molecules produced can react with H_2O_2 and produce a hydroxyl radical that attacks HA molecules.[32]

In investigating the effect of sonophotocatalyst and Fenton on the removal of HA from water, Geng *et al*. showed that the US effect in the Fenton process caused the production of more reactive radicals and helped the removal efficiency of HA.[10] The study of Torres *et al*., titled "BPA mineralization by integrated ultrasound-UV-iron (II) treatment," showed that this process is the most cost-effective method known to remove BPA from wastewater.[33] Huang *et al*., in a study titled "Heterogeneous sono-Fenton catalytic removal of BPA by Fe_3O_4 magnetic nanoparticles under neutral condition," found that the removal efficiency of BPA was more than 95%.[34]

Conclusion

In this study, the effect of pH parameters, reaction time, initial concentration of HA, and concentration of hydrogen peroxide and ferric ion on the removal of HA from aqueous solution by the Fenton process with the help of US waves was investigated. The study's findings highlight the significant role of US waves in enhancing the process efficiency. Specifically, the removal efficiency increased by approximately 20%–25% due to the influence of these waves. Furthermore, under optimal conditions (H_2O_2 dose: 100 mg/L, Fe^{2+} dose: 270 mg/L, and solution pH: 3), HA in aqueous solution with an initial concentration of 20 mg/L showed high removal in 15 min. In general, the results of this study showed that the process intensified with US waves is able to reduce the concentration of HA in water environments to a high extent.

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Ethic code

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Conflicts of interest

There are no conflicts of interest.

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