

## USING IRON-CONTAINING METAL OXIDE AS CATALYST FOR HETEROGENEOUS FENTON PROCESS IN TEXTILE INDUSTRY WASTEWATER

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### Keywords

Heterogeneous Fenton  
Textile wastewater  
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### Abstract

With the increase in industrialization, the unconscious use of surface and groundwater has led to the rapid pollution of water, which is the main source of life for all living things. As a result, the need for clean water has brought the global water crisis to the agenda. The textile industry is one of the largest producers of wastewater in the world. Textile industry wastewater contains high amounts of non-biodegradable organic compounds, high concentrations of dyestuffs, salt, detergent and soap. Therefore, it is of great importance to remove organic pollutants in this wastewater. Since traditional methods are insufficient to remove organic compounds in wastewater, advanced treatment methods are required. Advanced oxidation processes (AOPs) are one of the alternative treatment methods preferred in recent years. In this study, color removal from textile industry wastewater was researched by the heterogeneous Fenton process, which is an advanced oxidation process. The parameters such as catalyst dosage, pH, hydrogen peroxide concentration, temperature, reaction time and mixing speed that effect heterogeneous Fenton processes were investigated. Under optimum experimental conditions, the color removal efficiency was achieved as 84.4%.

## TEKSTİL ENDÜSTRİSİ ATIKSUYUNDA HETEROJEN FENTON PROSESİ İÇİN KATALİZÖR OLARAK DEMİR İÇEREN METAL OKSİT KULLANIMI

### Anahtar Kelimeler

Heterojen Fenton  
Tekstil atıksuyu  
Renk giderimi

### Öz

Endüstrileşmenin artışıyla beraber yüzey ve yeraltı sularının bilinçsiz bir şekilde kullanımı tüm canlılar için temel yaşam kaynağı olan suyun gün geçtikçe hızlı bir şekilde kirlenmesine yol açmıştır. Bunun sonucunda temiz su ihtiyacı da küresel su krizini gündeme getirmiştir. Tekstil endüstrisi dünyadaki en büyük atıksu üreticilerinden biridir. Tekstil endüstrisi atıksuları, yüksek miktarlarda biyolojik olarak parçalanamayan organik bileşikler, yüksek konsantrasyonlarda boyar maddeler, tuz, deterjan ve sabun içerir. Bu nedenle atıksuyun içerisindeki organik kirleticilerin uzaklaştırılması büyük önem taşımaktadır. Atıksudaki organik bileşiklerin uzaklaştırılmasında geleneksel yöntemler yetersiz olduğundan, gelişmiş arıtma yöntemlerine ihtiyaç duyulmaktadır. İleri oksidasyon prosesleri (İOP) son yıllarda tercih edilen alternatif arıtım yöntemlerinden biridir. Bu çalışmada, tekstil endüstrisi atıksuyundan renk giderimi, ileri oksidasyon proseslerinden biri olan heterojen Fenton prosesi ile araştırılmıştır. Heterojen Fenton proseslerini etki eden katalizör dozajı, pH, hidrojen peroksit konsantrasyonu, sıcaklık, reaksiyon süresi ve karıştırma hızı gibi parametreler incelenmiştir. Optimum deneysel koşullar altında renk giderme verimi % 84.4 olarak elde edilmiştir.

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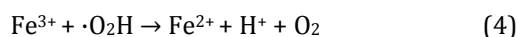
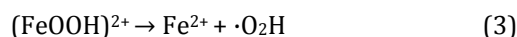
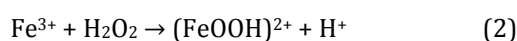
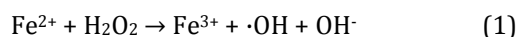
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## 1. Introduction

The textile industry is one of the industries with high water consumption in the world and the amount of wastewater generated after production is quite high. Industrial wastewater from dyeing and finishing processes contains a significant amount of dyestuff (Feng et al., 2010). The dyestuffs in wastewater are difficult to biodegrade, carcinogenic, toxic, highly resistant to traditional treatment methods and have a stable structure. In addition, the dyestuffs in the water change the pH value of the water even at very low concentrations and increase the chemical oxygen demand. Hence, discharging these wastewaters into the receiving environment without treatment has a detrimental effect on the health of live and causes serious environmental problems (Soltani et al., 2013; Verma et al., 2012).

In recent years, advanced oxidation processes (AOPs) using ozone, titanium dioxide (TiO<sub>2</sub>), ultraviolet (UV) light, and Fenton's reagent (H<sub>2</sub>O<sub>2</sub> and ferrous ion) are widely used because they transform toxic and non-biodegradable organic pollutants into harmless end products. Among them, the homogeneous Fenton process is one of the most practical advanced oxidation technologies available, as it creates hydroxyl radicals that are strongly oxidizing (Aşçı, 2013). Iron ions act as catalysts and hydroxyl radicals ( $\cdot\text{OH}$ ) are produced from the reaction of iron ions with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) in an acidic condition. Hydroxyl radicals are not selective, they react with all organic substances and decompose into CO<sub>2</sub> and H<sub>2</sub>O as final product (Göde et al., 2019; Ribeiro et al., 2016). The oxidation reactions generally follow as below Eq. (1)-(4) (Sani et al., 2020).



In heterogeneous Fenton processes, iron ions in the classical Fenton process are replaced by catalysts containing active catalytic sites. The oxidation reactions take place on the catalyst surface. The most important advantage of the catalysts used in heterogeneous Fenton reactions is the recovery and reusability of the catalyst used. In addition, catalysts prevent the formation of catalytic sludge waste by minimizing the leakage of iron ions into wastewater (Buthiyappan and Raman, 2019; Domingues et al., 2019; Xia et al., 2017). In recent years, metal oxides, which have properties such as low toxicity, low cost and chemical stability as catalysts in the Heterogeneous Fenton process, have been of great

interest. Many metal oxides are known to be semiconductors. Semiconductors; they have two different energy bands, one containing an electron and the other an empty energy level. Different of conductors, there is a gap band between the conductivity band and the valence band. They can oxidize all kinds of organic compounds due to the gaps with high oxidation potential (Zhang et al., 2016; Ruales-Lonfat et al., 2015).

In this study, it is aimed to remove color from textile industry wastewater. Heterogeneous Fenton process was applied by using iron-containing metal oxide as catalyst. The effects of parameters such as catalyst dosage, pH, hydrogen peroxide concentration, temperature, reaction time and mixing speed on oxidation process were investigated.

## 2. Materials and Method

### 2.1. Materials

The wastewater used in this study was supplied from the balancing pool without being subjected to any pretreatment from a textile company in Edirne/Turkey. Sodium hydroxide (NaOH) (Sigma Aldrich) and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) (Merck) chemicals were used to adjust the pH values of the wastewater samples. In the preparation of the catalyst, chemicals such as iron (III) nitrate nonahydrate (Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O) (Sigma Aldrich), manganese (IV) oxide (MnO<sub>2</sub>) (Merck), and ammonium hydroxide (NH<sub>4</sub>OH) (Sigma Aldrich) were used. Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) (30% w/w) used in heterogeneous Fenton processes was provided from Sigma Aldrich.

### 2.2. Catalyst

Experimental studies were carried out with MnO<sub>2</sub> catalyst containing 8% iron ion by weight. The catalyst was prepared according to the co-precipitation method. First of all, Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O and MnO<sub>2</sub> were dissolved in 100 ml of distilled water. NH<sub>4</sub>OH (26%) was added dropwise to the solution in a heated magnetic stirrer. The pH value was adjusted to 9 and the solution was mixed at 65°C for 2 hours at 300 rpm. The precipitate formed was separated and kept in the oven for 24 hours at 105°C and then at 600°C for 2 hours in the ash oven (Kaya and Ascı, 2019; Ayas et al., 2016).

### 2.3. Heterogeneous Fenton experimental procedure

The pH value of the wastewater sample was adjusted to the values determined with a 2M H<sub>2</sub>SO<sub>4</sub> solution. Catalyst was added to the pH adjusted samples by weighing certain amounts. Hydrogen peroxide was then added and the Fenton reaction started. The prepared

samples were kept in the shaking water bath at constant temperature for the duration of the reaction. The pH of the samples taken from the water bath was adjusted to the basic conditions using a 2M NaOH solution. The samples were filtered and the solution portion was separated for color analysis. The experimental procedure of the heterogeneous Fenton process was indicated in Fig. 1.

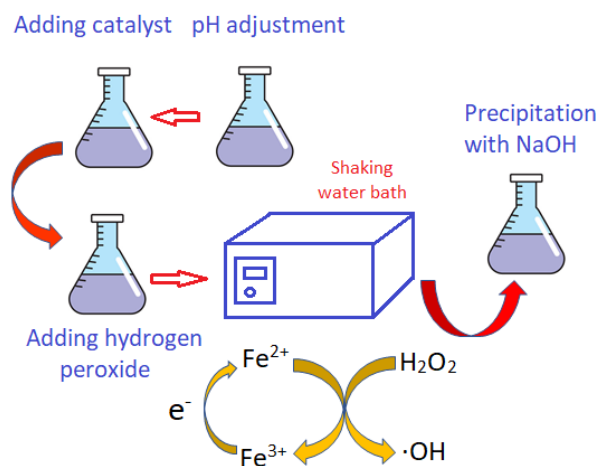


Figure 1. Heterogeneous Fenton experimental procedure.

#### 2.4. Color analysis

The wavelength that gives the maximum absorbance was determined by scanning the wavelength in the Hach Lange DR 3900 brand spectrophotometer. The color measurements were performed at this wavelength ( $\lambda_{max}=656$  nm) and the color removal efficiency was calculated using the equation remarked below.

$$\text{Color removal (\%)} = \left( \frac{C_0 - C_t}{C_0} \right) \times 100 \quad (5)$$

The  $C_0$  value mentioned in the equation refers to the initial absorbance value, and the  $C_t$  value to the absorbance value after heterogeneous Fenton process.

### 3. Results and Discussion

#### 3.1. Catalyst results

Energy Dispersion Spectrometer (EDS) analysis was performed at Central Research Laboratory Application and Research Centre of Eskişehir Osmangazi University to determine the element percentages of synthesized catalyst. The surface area analyze was applied by Brunauer, Emmet and Teller (BET) method at at Technology Application and Research Center of Afyon Kocatepe University. The analysis results were presented in Table 1.

Table 1

Element percentages and surface area properties in the catalyst.

Fe <sup>3+</sup> /MnO <sub>2</sub>		
EDS elemental analysis results (%)	Fe	8.89
	O	1.51
	Mn	89.60
BET analysis results	Surface area (m <sup>2</sup> /g)	30.91
	Total pore volume (cm <sup>3</sup> /g)	0.016
	Pore size (nm)	2.011

#### 3.2. Influence of catalyst dosage

In heterogeneous Fenton processes, oxidation reactions take place on the surface of the catalyst and therefore the dosage of catalyst is an important parameter in terms of hydroxyl radical generation. The experimental studies were carried out at the catalyst dosages of 1, 2, 4, 6 and 8 g/L to investigate the effects of catalyst dosage on heterogeneous Fenton process from textile industrial wastewater. Hydrogen peroxide concentration, pH, temperature and reaction time were kept constant at 200 ppm, 2, 30°C and 120 minutes, respectively. The color removal efficiencies varying with time were indicated in Fig. 2.

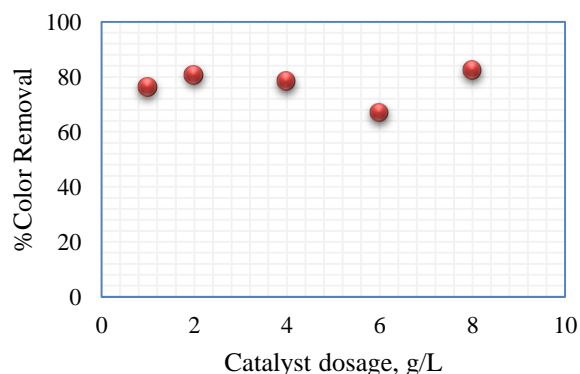
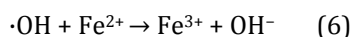


Figure 2. Influence of catalyst dosage on the color removal.

According to the results, it was observed that the color removal efficiency increased due to the release of hydroxyl radicals with high oxidation potential with the increase in the catalyst dosage. As the catalyst dosage was increased from 1 g/L to 2 g/L, the color removal efficiency increased from 76.3% to 80.6%. When the catalyst dosage was doubled (4 g/L), the color removal efficiency of 78.4% was achieved. The optimum catalyst dosage was determined as 2 g/L since the color removal efficiency decreased with increasing catalyst dosage. This situation can be explained by the increase of active sites on the catalyst surface, the acceleration of ·OH radical production and thus the increase of the color removal efficiency (Wang et al., 2016; Idel-aouad et al.,

2011). The increase in the dosage of catalyst increases the hydroxyl radical formation up to a point. The color removal efficiency decreases when working in high catalyst dosage due to hydroxyl radicals are scavenged by excess iron ions in the environment as Eq. (6) (Ghasemi et al., 2020; Kantar et al., 2019).



### 3.3. Influence of pH

It is an important parameter in heterogeneous Fenton process as pH effects reaction stability. Experiments to determine the pH effect on the color removal were acquired at constant catalyst dosage (2 g/L), hydrogen concentration (200 ppm), temperature (30°C) and reaction time (120 min). pH values were varied between 1.5, 2, 3, 4 and 5. The experimental results were reported in Fig. 3.

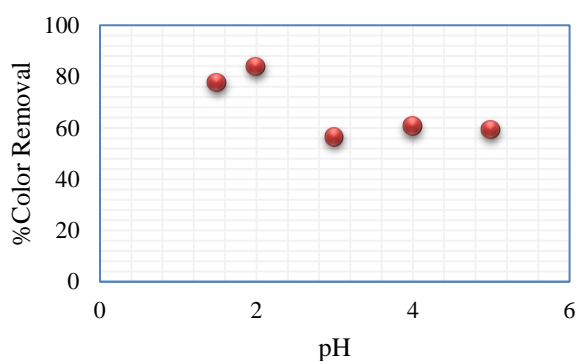
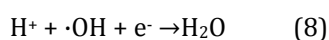
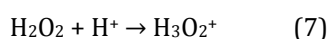


Figure 3. Influence of pH on the color removal.

In the heterogeneous Fenton process, the optimum pH value was determined as 2. The color removal efficiency at the optimum pH value was reached 83.9%. When Fig. 3 is examined, it is seen that the color removal efficiency increases up to the pH 2 value and decreases after this value. The reason for this decrease is due to the decrease in iron ion activity in the catalyst. As the iron ion is more active in acidic conditions, more hydroxyl radicals are produced. However, although low pH values positively effect Fenton reactions, a decrease in dye removal occurs due to the scavenging of hydroxyl radicals by  $\text{H}^+$  ions in an extremely acidic reaction environment as Eq. (7) and Eq. (8) (Guo et al., 2014; Devi et al., 2009; Qiao et al., 2005). In addition, at low pH values, the reaction efficiency is severely effected as complex species of  $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$  and  $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$ , which react slowly with hydrogen peroxide, are formed (Buthiyappan et al., 2016).



At high pH values, hydrogen peroxide decomposes into  $\text{H}_2\text{O}$  and  $\text{O}_2$  and complexes such as iron (III) hydroxide are formed. Therefore, the production of hydroxyl

radicals decreases and the reaction efficiency decreases accordingly (Lin et al., 2014). Malakootian et al. (2013) and Chen (2006) reported the optimum pH value as 2 in their similar studies.

### 3.4. Influence of $\text{H}_2\text{O}_2$ concentration

Hydroxyl radicals are formed by catalytic decomposition of hydrogen peroxide in heterogeneous Fenton process. Determination of optimum hydrogen peroxide concentration in terms of oxidation efficiency is significant. In this context, experimental studies were conducted at optimum catalyst dosage (2 g/L) and optimum pH (2). Hydrogen peroxide concentration has been studied at different values between 50-400 ppm at constant temperature (30°C) and reaction time (120 min).

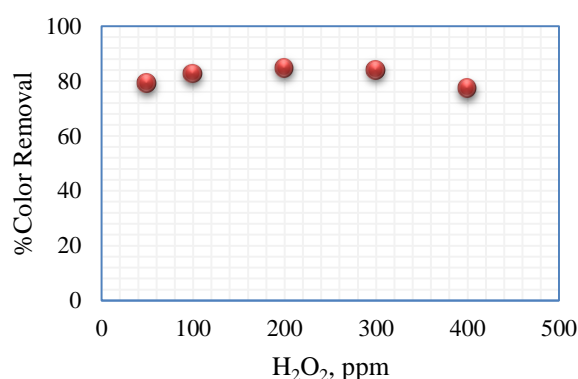


Figure 4. Influence of  $\text{H}_2\text{O}_2$  concentration on the color removal.

As seen in Figure 4, when the hydrogen peroxide concentration increased from 50 ppm to 200 ppm, the color removal efficiency increased. The maximum color removal efficiency of 200 ppm was achieved as 84.7%. Since the color removal efficiency decreased in values after 200 ppm hydrogen peroxide concentration, the optimum concentration was determined as 200 ppm. Since sufficient hydroxyl radicals are not produced at low hydrogen peroxide concentrations, lower removal is provided. When the hydrogen peroxide concentration in the environment increases, the reaction efficiency increases with the increase of hydroxyl radical. However, excessive use of hydrogen peroxide causes it to react with hydroxyl radicals as Eq. (9). With this reaction, hydroperoxyl radicals ( $\text{HO}_2\cdot$ ) are formed, which have lower oxidation potential (1.7 eV) than the hydroxyl radical (2.8 eV). Since hydroperoxyl radicals have lower activity, they do not contribute to oxidative degradation reactions (Mokbi et al., 2019; Rehman et al., 2018; Ukpaka, 2018; Kwan and Voelker, 2003).



### 3.5. Influence of temperature

The temperature is an important parameter effecting the catalytic oxidation rate in heterogeneous Fenton processes. Experimental studies were carried out at the optimum conditions determined to investigate the temperature effect in the heterogeneous Fenton method. The temperature values have been changed as 25, 30, 35, 40, 45 and 50°C under the conditions of pH value 2, catalyst dosage 2 g/L, H<sub>2</sub>O<sub>2</sub> concentration 200 ppm and 120 minutes reaction time, and the experimental results obtained were shown in Fig. 5.

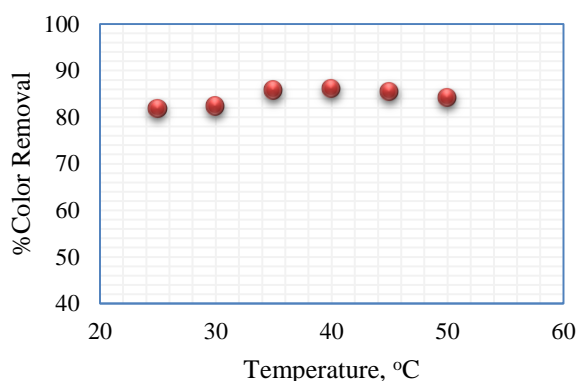


Figure 5. Influence of temperature on the color removal.

In heterogeneous Fenton processes, the reaction rate of between hydrogen peroxide and iron ions increases with increasing temperature. As a result, more hydroxyl radicals are formed in a shorter time and the color removal efficiency increases. However, at temperatures above 50°C, hydrogen peroxide decomposes into water and oxygen and the reaction efficiency decreases (Naseem et al., 2019; Gan and Li, 2013). According to Fig. 5, while the color removal efficiency was 81.8% at 25°C, it was reached 86.1% at the 40°C. Then the color removal efficiency was observed to decrease at temperatures above 40°C. Therefore, the optimum reaction temperature has been accepted as 40°C.

### 3.6. Influence of reaction time

One of the important advantages of the heterogeneous Fenton process is that it provides effective color removal at lower reaction time compared to other treatment methods (Nidheesh et al., 2013). The effect of reaction time on color removal efficiency was investigated by different values in the range of 5-240 minutes. The other parameters were kept constant, such as catalyst dosage (2 g/L), pH (2), hydrogen peroxide concentration (200 ppm) and temperature (40°C).

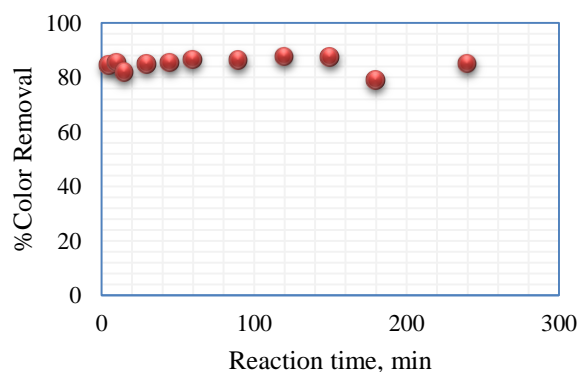


Figure 6. Influence of reaction time on the color removal.

As shown in Figure 6, the color removal efficiency has increased as the reaction time has increased from the 5th minute to the 120th minute, and its maximum value has been reached as 87.6%. As can be seen here, even in the first 5 minutes, a color removal efficiency of 84.4% was obtained. As a result, although 87.6% efficiency is obtained in 120 minutes, considering the cost, it can be said that the optimum time is actually 5 minutes. But, the 120 min of contact time, is needed to reach a satisfactory equilibrium. Therefore, the effect of all parameters on the color removal, excluding the mixing speed, was made constant for 120 minutes. Deng et al. (2012) determined the reaction time as 30 minutes in their work which the degradation of Acid Orange II with Fe<sub>3</sub>O<sub>4</sub>-multi-walled carbon nanotube catalyst in the heterogeneous Fenton process. Iranifam et al. (2011) performed Fenton process experiments in 20 minutes reaction time for the degradation of CI Basic Yellow 28.

### 3.7. Influence of mixing speed

In the heterogeneous Fenton process, three different mixing speeds (40, 100 and 160 rpm) were applied to determine the effect of mixing speed on color removal. When mixing speed increases, mass resistance decreases in heterogeneous Fenton processes and homogeneous distribution of Fenton reactants is achieved. The optimum mixing speed was determined as 160 rpm and the color removal efficiency was attained as 86.5%.

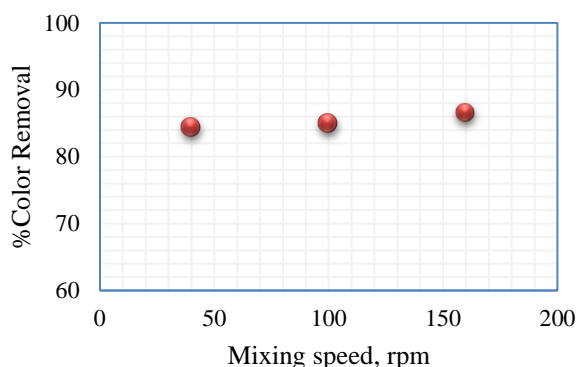


Figure 7. Influence of mixing speed on the color removal.

#### 4. Conclusion

Heterogeneous Fenton process on color removal from textile industry wastewater has been investigated. For this purpose,  $MnO_2$  catalyst containing 8% (w/w) iron ion was synthesized for using as a catalyst. The parameters effecting the oxidation reaction such as catalyst dosage, pH, hydrogen peroxide, temperature, reaction time and mixing speed were researched. Under optimum experimental conditions (catalyst dosage 2 g/L, pH 2, hydrogen peroxide concentration 200 ppm, temperature 40°C, reaction time 5 minutes and 160 rpm) 84.4% color removal efficiency was achieved. Heterogeneous Fenton process is a suitable method for treatment textile industry wastewater since it has high catalytic activity, long-term stability and high removal efficiency.

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#### Author Contributions

Merve DURGUT contributed to experimental studies and literature search; Şefika KAYA contributed to experimental studies, literature search, analysis and interpretation of results, preparation of manuscript; Yeliz AŞÇI contributed to interpretation of results, preparation of manuscript.

#### Conflict of Interest

There is no conflict of interest.

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