

# Advanced Oxidation Treatment of Chemical Delinting Wastewater Using a Fenton-like Reagent

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**Abstract** Delinting units or cotton seed production plants generate effluents heavily loaded with persistent organic matter derived from cottonseed linters. To protect the environment, it is essential to develop treatment techniques that are effective, economical, and environmentally friendly. Advanced Oxidation Processes (AOPs) using the Fenton reagent have proven capable of efficiently removing recalcitrant organic pollutants with minimal ecological impact. The objective of this study is to treat wastewater generated by the chemical delinting unit using an advanced oxidation process based on the Fenton reagent, in a context where environmental standards are becoming increasingly stringent. The adopted methodology first involved characterizing the wastewater. Then, the Fenton reagent was prepared from anhydrous ferric chloride ( $\text{FeCl}_3$ ) and hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). Finally, batch treatment tests were conducted. To evaluate the treatment performance, several physicochemical parameters of the wastewater were analyzed, including pH, Chemical Oxygen Demand (COD), Biochemical Oxygen Demand ( $\text{BOD}_5$ ), turbidity, color, electrical conductivity, total phosphorus, total nitrogen, and suspended solids content. The results of the treatment tests demonstrated a remarkable performance of the process, with removal efficiencies of 98.71% for suspended solids, 94.36% for color, 90.83% for total nitrogen, 87.22% for total phosphorus, 99.93% for COD, and 100% for  $\text{BOD}_5$ . The Fenton reagent proved to be highly effective, allowing significant depollution of the contaminated water while ensuring compliance with the Senegalese discharge standard NS 05-061. These encouraging results provide a solid basis for considering large-scale application of this process for the treatment of effluents from chemical delinting units.

**Keywords:** Advanced Oxidation, Fenton Reagent, Wastewater Treatment, Delinting

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## 1. Introduction

Although water is indispensable to life, nearly 80% of wastewater is still discharged into aquatic environments without prior treatment, with industry accounting for a major share of pollutant emissions. This situation has triggered widespread concern worldwide due to the rapid increase in environmental pollution associated with industrial development and population growth. Intensive industrialization, which emerged during the last century, has led to the appearance of emerging and refractory pollutants in environmental matrices such as air, water, and soil [1,2]. These so-called bio-recalcitrant substances can contaminate living organisms [3,4]. In fact, some of these compounds are carcinogenic or mutagenic, while others act as endocrine disruptors, interfering with the hormonal systems of living beings. Certain industrial

effluents contain hormones and hormonal derivatives, phenolic compounds, antibiotics, organochlorines, and cosmetic residues, among others. These substances are known to cause numerous disturbances to aquatic fauna and pose serious risks to human health [5,6]. Most of these emerging contaminants are resistant to conventional wastewater treatment technologies, and their increasing presence in the environment has become a major concern for environmental management agencies [7,8]. To mitigate environmental contamination by these pollutants, several effective and eco-friendly treatment strategies have been developed. Among these, Advanced Oxidation Processes (AOPs) have gained prominence due to their ability to degrade persistent organic pollutants. The chemical delinting industry, which removes residual fibers from cottonseeds, generates effluents rich in persistent and non-biodegradable organic matter. If discharged untreated, these effluents can lead to severe water and soil pollution, threatening ecosystems and human health [9]. In this

context, Advanced Oxidation Processes (AOPs) represent an efficient and sustainable solution for the treatment of industrial wastewater. Among them, the Fenton process, which combines hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) with ferrous ions ( $\text{Fe}^{2+}$ ), is particularly well recognized for its ability to generate highly reactive hydroxyl radicals ( $\text{OH}^\bullet$ ). These radicals rapidly and effectively degrade complex organic pollutants into simpler, less harmful compounds. The objective of this study is to treat the wastewater produced by a chemical delinting unit using an Advanced Oxidation Process (AOP) based on the Fenton reagent, within a context of increasingly stringent environmental regulations.

## 2. Materials and Methods

### 2.1. Materials

Wastewater samples were collected from the residual basin of a chemical delinting unit. A multiparameter probe was used to measure pH, electrical conductivity, and total dissolved solids (TDS), while a DR3900 Hach UV-visible spectrophotometer was employed to determine COD, color, and suspended solids (SS).

### 2.2. Synthesis of the Fenton Reagent

The Fenton reagent was prepared by dissolving 25 g of anhydrous ferric chloride ( $\text{FeCl}_3$ ) in 500 mL of HCl solution ( $\text{pH} = 2.11$ ). The solution was stirred for 10 minutes to ensure complete dissolution, then 10.6 mL of  $\text{H}_2\text{O}_2$  was added. Stirring continued for 30 minutes to promote the formation of hydroxyl radicals ( $\text{OH}^\bullet$ ) for efficient oxidation of organic pollutants.

### 2.3. Batch Treatment Procedure

Batch experiments were performed using 400 mL of wastewater in separate beakers. Varying volumes of Fenton reagent (0.5 with stirring, 1 with stirring and 0.5 mL of Fenton reagent without stirring) were added to optimize treatment efficiency. Each mixture was stirred for 30 minutes to ensure homogeneous distribution and optimal interaction with pollutants. After oxidation, samples were neutralized and left to settle, allowing solid residues to precipitate. This step ensures the clarification of treated water prior to physicochemical analysis.

### 2.4. Treatment Efficiency Calculation

Treatment efficiency (%) for a given parameter was calculated as:  $(\%) = (P_i - P_f)/P_i$ . Where  $P_i$  and  $P_f$  are the initial and final values of the physicochemical or biological parameter, respectively.

## 3. Results and Discussion

### 3.1. Characterization of the Wastewater

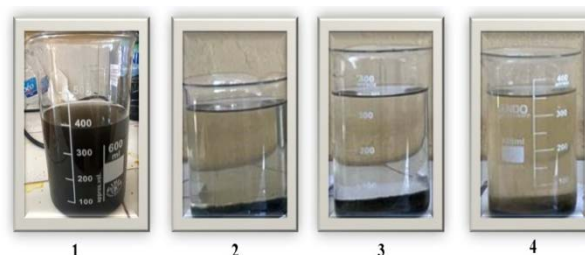
Table 1 presents the physicochemical and microbiological characteristics of the untreated effluent

compared with the limit values specified by the Senegalese standard NS 05-061. Most parameters - particularly Total suspended solids (TSS), color, total nitrogen, Chemical Oxygen Demand (COD), and Biochemical Oxygen Demand ( $\text{BOD}_5$ ) - exceed the regulatory limits, indicating a high level of organic pollution. The initial pH, close to neutrality, complies with the standard and results from the neutralization step performed after the delinting process, prior to storage. The relatively low electrical conductivity and total dissolved solids reflect a weak mineralization, confirming the organic nature of the pollution. The elevated TSS concentration indicates a significant presence of suspended particles mainly derived from cotton linters, while the intense coloration is attributed to organic matter decomposition. Similarly, the high total nitrogen content originates primarily from organic nitrogen compounds contained in the linters. The very high COD and  $\text{BOD}_5$  values demonstrate a substantial organic load comprising both biodegradable and non-biodegradable fractions. A COD/ $\text{BOD}_5$  ratio greater than 3 suggests that physicochemical treatment, such as an Advanced Oxidation Process (AOP), is more appropriate than biological methods for efficient pollutant removal.

**Table 1. Physicochemical characteristics of the untreated wastewater**

| Physicochemical and biochemical parameter | Untreated wastewater | Standard NS 05 061 |
|-------------------------------------------|----------------------|--------------------|
| pH                                        | 6.81                 | 6 – 9              |
| E.C ( $\mu\text{S}/\text{cm}$ )           | 371.1                | 1400               |
| TSS ( $\text{mg}/\text{L}$ )              | 194.6                | 1200               |
| MEST ( $\text{mg}/\text{L}$ )             | 542                  | 50                 |
| Color (echelle Co-Pt)                     | 2057                 | -                  |
| Total nitrogen ( $\text{mg}/\text{L}$ )   | 43.5                 | 20                 |
| Total phosphorus ( $\text{mg}/\text{L}$ ) | 1.8                  | 10                 |
| DCO ( $\text{mg}/\text{L}$ )              | 1122                 | 200                |
| DBO ( $\text{mg}/\text{L}$ )              | 128.2                | 80                 |

### 3.2. Batch Treatment Technique



1= raw water, 2 = 0,5ml of Fenton-like reagent and 400mL raw water; 3 = 1 mL of Fenton-like reagent and 400mL raw water; 4= 0,5 mL of Fenton-like reagent and 400mL raw water without stirring

**Figure 1.** Simulation of the treatment of 400 mL of wastewater using 0.5, 1.0, and 1.5 mL of Fenton reagent.

The treatment results are presented in Figure 1. The analysis of physicochemical and microbiological parameters was performed to evaluate the efficiency of the applied process on the liquid effluents. Key parameters—including pH, electrical conductivity, color, suspended solids (SS), phosphorus, nitrogen, Chemical Oxygen Demand (COD), and Biochemical Oxygen Demand ( $\text{BOD}_5$ )—were compared between untreated and treated

samples. The findings highlight the optimal operating conditions of the process and confirm its effectiveness in treating effluents generated by the chemical delinting unit. Moreover, these results provide valuable insights for process optimization and potential large-scale implementation of the Fenton-based treatment system.

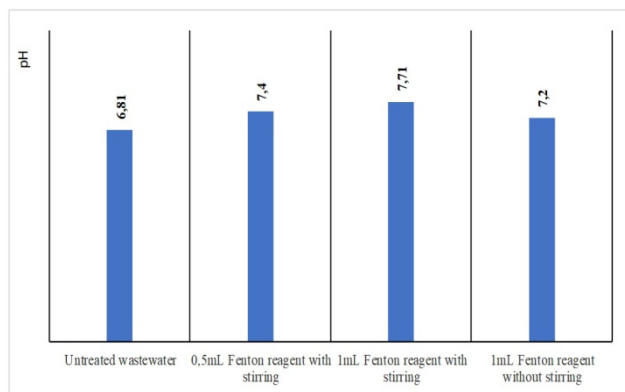
**Table 2. Laboratory Batch Treatment Analysis Results**

| Physicochemical and biochemical parameter | Untreated wastewater | Fenton Reagent Volumes (mL) |       |       | Standard NS05 061 |
|-------------------------------------------|----------------------|-----------------------------|-------|-------|-------------------|
|                                           |                      | 0,5                         | 1     | 1     |                   |
| Ph                                        | 6,81                 | 7.4                         | 7.71  | 7.2   | 6 – 9             |
| E.C (µS/cm)                               | 371.1                | 630.9                       | 722.5 | 752.6 | 1400              |
| TSS (mg/L)                                | 194.6                | 297.7                       | 379.2 | 387.4 | 1200              |
| MEST (mg/L)                               | 542                  | 9                           | 7     | 8     | 50                |
| Color (échelle Co-Pt)                     | 2057                 | 211                         | 116   | 109   | -                 |
| Total nitrogen (mg/L)                     | 43.5                 | 4.67                        | 3.99  | 2.49  | 20                |
| Total phosphorus (mg/L)                   | 1.8                  | 0.21                        | 0.23  | 0.07  | 10                |
| DCO (mg/L)                                | 1122                 | 31.2                        | 0.742 | 0.183 | 200               |
| DBO (mg/L)                                | 128.2                | 0                           | 0     | 0     | 80                |

The physicochemical and microbiological parameters of the wastewater before and after treatment are presented in Table 2. This comparison allows an assessment of the effectiveness of the applied treatment method. Parameters such as suspended solids (SS), color, total nitrogen, total phosphorus, Chemical Oxygen Demand (COD), and Biochemical Oxygen Demand (BOD<sub>5</sub>) were significantly reduced below the permissible limits, indicating the high efficiency of the treatment process.

### 3.3. Hydrgen Potential (pH)

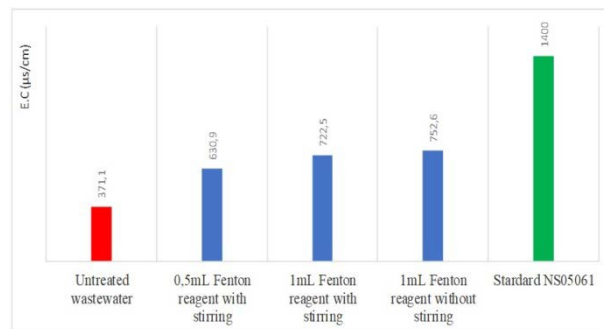
Figure 2 shows the pH values of the treated wastewater in relation to the acceptable range defined by the Senegalese standard NS 05-061 (6–9). A slight increase in pH was observed after treatment, as it was adjusted to facilitate settling and clarification. Following treatment, the pH values comply with the Senegalese discharge standards.



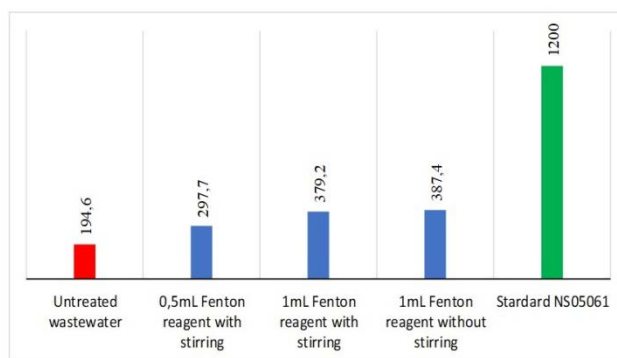
**Figure 2.** Variation of the hydrogen potential (pH) before and after treatment compared to the standard

### 3.4. Electrical Conductivity and Total Dissolved Solids (TDS)

Figure 3 and Figure 4 present the electrical conductivity and total dissolved solids (TDS), respectively, which are associated with the presence of mineral salts. The wastewater exhibited low values of electrical conductivity and TDS (371.1 µS/cm and 194.6 mg/L, respectively), indicating a low degree of mineralization in the effluent.



**Figure 3.** Variation of electrical conductivity before and after treatment

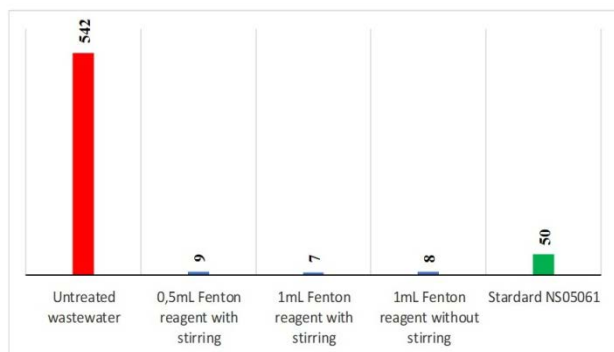


**Figure 4.** Variation of total dissolved solids (TDS) before and after treatment

After treatment, both electrical conductivity and TDS increased. This rise is attributed to the higher ionic content in the treated water, resulting from the Fenton reagent ions (chloride, Fe<sup>3+</sup>), ionic by-products formed during the reaction of hydrogen peroxide with organic compounds, and additional ions introduced during the neutralization step with NaOH.

### 3.5. Total Suspended Solids (TSS)

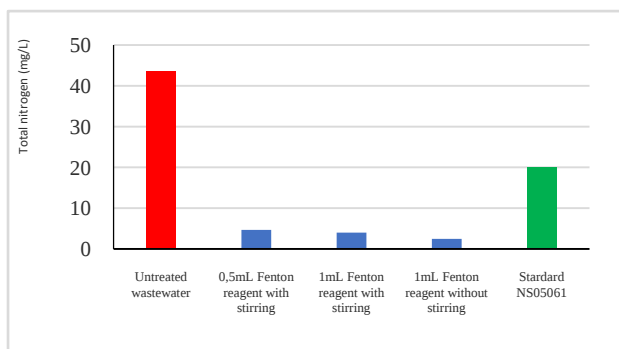
The total suspended solids (TSS) content provides a measure of the treatment efficiency. The wastewater from the delinting unit was heavily loaded with TSS, with an initial value of 542 mg/L (Figure 5), far exceeding the Senegalese standard of 50 mg/L, highlighting the need for treatment. Post-treatment measurements of TSS using 0.5 mL and 1 mL of Fenton reagent with stirring, and 1 mL without stirring, yielded values of 9, 7, and 8 mg/L, respectively. All of these values comply with the Senegalese discharge standard. Treatment with 1 mL of reagent under stirring proved to be the most effective, achieving a 98.7% reduction in TSS. This decrease is primarily due to chemical oxidation of the organic matter followed by settling of the reaction products



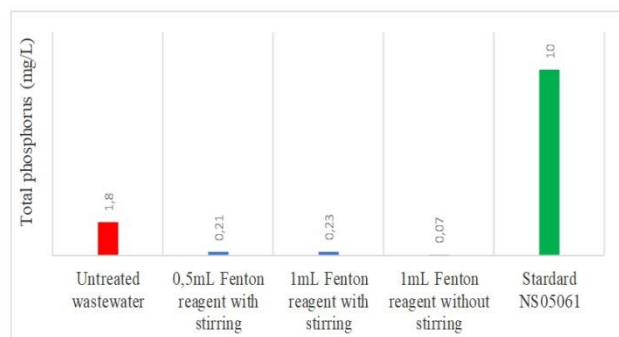
**Figure 5.** Total Suspended Solids (TSS) values before and after treatment tests compared with the NS05-061 standard

### 3.6. Total Nitrogen and Phosphorus

Figure 6 and Figure 7 illustrate the changes in total nitrogen and total phosphorus concentrations, respectively, before and after treatment. The initial total nitrogen concentration, which was high at 43.5 mg/L and exceeded the standard, was reduced to 3.99 mg/L after treatment. In contrast, the total phosphorus concentration in the untreated wastewater was already within the permissible limit (1.8 mg/L) and decreased further to 0.23 mg/L following treatment. These results indicate a significant removal of nutrients, which could otherwise contribute to eutrophication in the receiving environments.



**Figure 6.** Variation of Total Nitrogen Concentrations Before and After Treatment

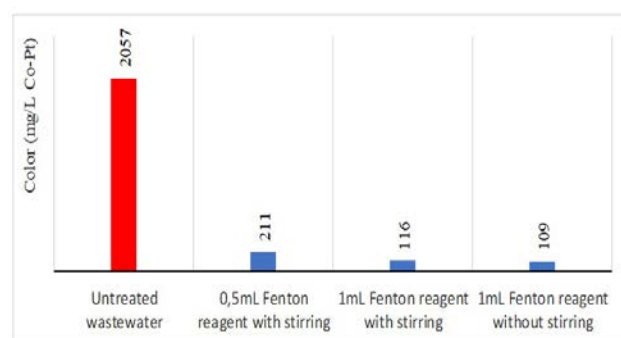


**Figure 7.** Variation of total phosphorus concentrations before and after treatment

### 3.7. Water Color) Before and After Treatment

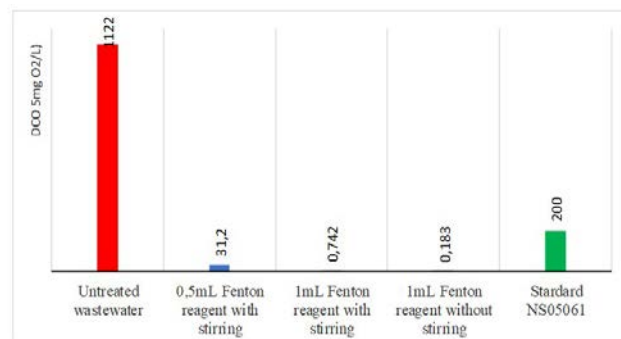
The wastewater exhibited a high color intensity, with a

value of 2057 Co-Pt (Figure 8). This strong coloration is primarily due to the presence of organic matter from cottonseed fibers. The Fenton treatment oxidizes this organic matter, followed by the settling of oxidation by-products, resulting in a reduction of colored organic compounds. Using 1 mL of Fenton reagent in 400 mL of wastewater achieved a 94.36% reduction in color. This result is consistent with the findings of Benoit et al. [10], who studied the reuse of granitic laterite in the treatment of textile industry wastewater. In their seven experimental trials, the color removal efficiency exceeded 90%, with a minimum reduction of 95.1%. This substantial decrease in color demonstrates the high effectiveness of the Fenton reagent in removing organic pollutants from wastewater.



**Figure 8.** Variation of water color concentrations) before and after treatment

### 3.8. Chemical Oxygen Demand (COD)



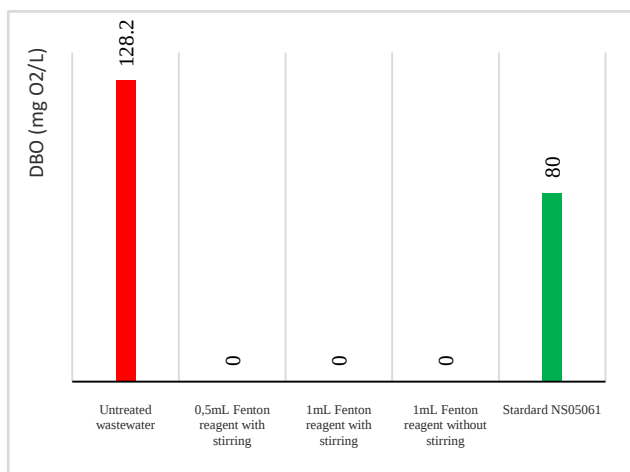
**Figure 9.** Variation of COD before and after treatment compared to the standard

Chemical Oxygen Demand (COD) measures the amount of oxygen required to oxidize all organic and inorganic substances in a sample. It is widely used to assess the pollution load of wastewater. Figure 9 presents the COD variations before and after treatment. The untreated wastewater exhibited an extremely high COD of 1122 mg/L O<sub>2</sub>, well above the permissible limit of 200 mg/L O<sub>2</sub>. Similarly high COD values, ranging from 1600 to 3200 mg/L O<sub>2</sub>, have been reported in textile industry effluents [11]. The high COD in the wastewater is attributed to the presence of oxidizable compounds used at various stages of the process. Treatment with the Fenton reagent allowed the determination of the mineralization rate, calculated as follows: The mineralization rate quantifies the percentage of organic matter in wastewater that has been oxidized to simpler inorganic compounds during treatment. Using 1 mL of Fenton reagent, 400 mL of wastewater was

99.9% mineralized, demonstrating the high efficiency of the process in oxidizing organic pollutants.

### 3.9. Biochemical Oxygen Demand (BOD<sub>5</sub>)

Biochemical Oxygen Demand (BOD<sub>5</sub>) measures the amount of oxygen required by bacteria to biologically degrade all oxidizable organic matter over a period of five days. It is commonly used to assess biological degradability in wastewater. Figure 10 shows the BOD<sub>5</sub> variations before and after treatment compared to the NS05-061 standard. The untreated effluent exhibited a high BOD<sub>5</sub> of 128.2 mg/L O<sub>2</sub>, which was completely removed by the Fenton reagent. The resulting BOD<sub>5</sub> reduction reached 100%, demonstrating the reagent's effectiveness in eliminating biodegradable organic pollutants. This result aligns with the findings of Adjou [12], who used titaniferous sand as a sorbent in column experiments. In Adjou's study, Fenton treatment achieved 99% degradation of methylene blue (BM), and 87% and 57% degradation for 2,4-dichlorophenoxyacetic acid (2,4-D) and methyl orange (MO), respectively. Overall, the applied Fenton treatment proved highly effective in reducing multiple water quality parameters, ensuring compliance with the Senegalese discharge standard.



**Figure 10.** Variation of BOD<sub>5</sub> before and after treatment compared to the standard

### 3.10. Summary of Treatment Efficiency

The Fenton advanced oxidation process effectively reduced all key parameters to below Senegalese discharge standards (NS05-061):

- ✓ COD: 99.9% reduction
- ✓ BOD<sub>5</sub>: 100% reduction
- ✓ Color: 94.36% reduction
- ✓ TSS: 98.7% reduction
- ✓ Total nitrogen: >90.8% reduction
- ✓ Total phosphorus: 87% reduction

While electrical conductivity and TDS increased due to ionic contributions from the reagent, these values remain moderate and manageable.

## 4. Conclusion

The Fenton process proved to be a highly effective, simple, and feasible treatment method for chemical delinting wastewater, achieving near-complete removal of organic pollutants, nutrients, suspended solids, and color, in compliance with national standards. Despite its effectiveness, high operational costs limit widespread adoption. Future work will explore:

- ✓ Catalytic potential of laterite as an alternative to ferric salts in AOPs, to reduce treatment costs.
- ✓ Agronomic valorization of treatment sludge, which contains high organic matter from cotton linters and could enhance soil fertility.

**Disclosure statement:** *Conflict of Interest:* The authors declare that there are no conflicts of interest.

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