



Photocatalytic Degradation of Emerging Contaminants Using *Sargassum*-derived Silver Nanoparticle and Alpha-cyclodextrin Inclusion Complex

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ABSTRACT

This paper outlines an eco-friendly biosynthesis of silver nanoparticle (SAA) from the marine brown algae *Sargassum*, sourced from the Koodankulam shoreline, as verified by XRD analysis. The substance is subsequently treated with an organic compound known as alpha cyclodextrin to yield a firm inclusion complex (S1). UV and IR are employed to characterize the synthesized nanoparticle. The significant peak of absorbance at 415 nm confirms the synthesis of silver nanoparticle produced by the algae *Sargassum*. The functional components in SAA have been characterized using IR spectroscopy. IR technology, ultraviolet (UV) spectroscopy, and ¹H NMR spectroscopy techniques are employed to analyze the synthesized alpha-cyclodextrin inclusion complex (S1). Fluorescence analyses conducted under daylight demonstrated that extracts from various solvents derived from granular *Sargassum* samples exhibited a spectrum of colours. The synthesized silver nanoparticle and its alpha-cyclodextrin inclusion complex are principally examined for photocatalytic applications against the contaminants (Dyes).

Keywords: *Sargassum*, silver nanoparticle, Photocatalytic, Alpha cyclodextrin, inclusion complex.

INTRODUCTION

Green nano-biology involves the synthesis of silver nanoparticles from diverse biological sources¹⁻⁴, exhibiting varying morphologies and dimensions, with prospective industrial uses⁵. With the advent of green nanoparticle production, there has been an increasing desire for environmentally acceptable

methods of metal-nanoparticle synthesis that do not utilize harmful chemicals⁶⁻¹⁰. Nanoparticles have been regarded as significant throughout the past decades and are being employed in many applications, including water purification, catalytic activity, biological and chemical sensors, memory systems and wireless electronic logic^{5,11-12}. The predominant method for synthesizing green nanoparticles



involves utilizing aqueous extracts from a variety of organisms, including fungi¹³, an alga¹⁴, and the plants¹⁵⁻¹⁸. *Sargassum* species, a marine macroalga classified within the category Phaeophyceae, is extensively found in tropical and arid oceans. It is part of the aquatic family Sargassaceae and the order Fucales. A broad spectrum of bioactive characteristics of *Sargassum* has been documented¹⁹. Furthermore, the *Sargassum* species, including, *Sargassum tenerrimum*²⁰, *Sargassum polycystum*, and *Sargassum glaucescens*²¹ *Sargassum latifolium*²² have been investigated and demonstrated notable antibacterial and antifungal properties. Alpha-cyclodextrins are cyclical oligosaccharides composed of six 1,4-linked d-glucose units. The glucose chair conformation results in a hollow truncated cone structure, imparting a hydrophobic nature to the inner cavity of the cyclodextrin, in contrast to its hydrophilic external surface. Alpha cyclodextrin exhibit functional features including moderate water solubility, tastelessness, Odor lessness, thermal stability (up to 200 °C), and stability in both alkaline and acidic solutions²³. Cyclodextrin molecules possess a distinctive structure, allowing for the entrapment of a guest molecule. Cyclodextrin can create inclusion complexes with a diverse array of liquid, solid and gaseous substances. The complex formation is multidimensional, geometrically constrained interaction between the cavity of cyclodextrin and a foreign molecule²⁴. Cyclodextrins are appropriate for use in food preservation and flavouring, medication, the cosmetics industry, agricultural, and chemical sectors.

This study synthesizes silver nanoparticle using the algae *Sargassum*. An appealing alpha cyclodextrin inclusion complex is formed by combining the synthesized silver nanoparticle with the compound alpha cyclodextrin. Subsequently, their photocatalytic breakdown of the pollutants is analysed and quantified.

MATERIALS AND METHODS

Compilation of specimen

The algae known as *Sargassum* have been taken from the shoreline area of Koodankulam. It is further purified with seawater to eliminate contaminants such as grains of sand and organisms known as epiphytes. To eliminate contaminants,

particularly pathogenic creatures, it is thoroughly purified using double-distilled water. The specimen was permitted to desiccate prior to being pulverized into a smooth powder.

Fabrication of Silver Nanoparticles from *Sargassum*

A sustainable production of silver nanoparticle was conducted in a glass container contains 70 mL of a 1 mM aqueous AgNO₃ solution and 30 mL of a micro-alga extract. The mixture was positioned in the darkness and agitated for an entire day. Consistent ocular assessments of silver nanoparticle synthesis indicated that a light-yellow solution transformed into a deep brown after 20 minutes, signifying the formation of silver nanoparticle. Sequential centrifugation and lyophilization were employed to enhance the developed nanoparticle.

Fluorescence Examination

A range of chemical solvents are employed to investigate the behaviour of a pulverised *Dictyota dichotoma* algae sample. The hue variations in the



Fig. 1: Fabrication of silver nanoparticle from the algae *Sargassum*

pulverised substance are examined under both ultraviolet and visible light.

Reagents Required

Synthesized silver nanoparticle (SAA) from the algae *Sargassum*, alpha cyclodextrin (Sigma-Aldrich) and double distilled water are used for the preparation of Alpha cyclodextrin inclusion complex.

Instruments Required

Systronics Double Beam Spectrophotometer -2203 (Ultraviolet Visible Spectroscopy)
JASCO Spectrofluorometer FP-8200 (Fluorescence Spectroscopy)

Preparation of solid and liquid inclusion complex

For the preparation of solid inclusion complex about 0.301g of alpha cyclodextrin in water is mixed with 0.121g of synthesized silver nanoparticle (SAA) in water and kept on stirring for 96 hrs. The prepared alpha cyclodextrin inclusion complex with SAA (Silver nanoparticle) is obtained as a precipitate, which is then oven dried and utilized for characterization and application studies.

Similarly, for liquid inclusion complex about 0.063g of alpha cyclodextrin in water is mixed with 0.008g of SAA in 10 ml of water in a clean beaker. The concentration of the alpha cyclodextrin taken can also be vary for the production of liquid inclusion complex. This is then characterized by IR and proton NMR spectroscopy.

Catalytic activity

The catalytic activity of the synthesized silver nanoparticle (SAA) and the alpha cyclodextrin inclusion complex (S_1) is studied using the contaminants Congo red and methylene blue. The decomposition study involving light from the sun irradiation is conducted outdoors in direct sunlight during midday (summer time) from 12.30 to 2.30 p.m. on clear, hot days. The investigations utilised synthesised quantities of silver nanoparticle (SAA) and alpha cyclodextrin inclusion complex (S_1). In the general experiment, six distinct beakers are filled with 20 ml of Congo red dye solution and 0.002 g of SAA. Similarly, another six beakers are filled with 20 ml of methylene blue dye solution and 0.002 g of SAA, which are then placed in a sonicator under direct sunlight to investigate photocatalytic degradation.

The degradation of environmental contaminants in relation to the synthesised nanoparticle SAA is observed at 30-minute intervals (0, 30, 60, 90, 150, 180). The identical protocol should be adhered to conduct the photocatalytic degradation investigation of the alpha cyclodextrin inclusion complex S_1 .

RESULT AND DISCUSSION

XRD of SAA (synthesized silver nanoparticle)

Figure 2 illustrates the X-ray diffraction patterns of SAA. The crystalline quality of the synthesised nano SAA is indicated by a strong peak. The crystalline dimension of the biosynthesised *Sargassum* nanoparticle was determined using the Debye-Scherrer equation. $2.7 \times 10^{-9} \text{m}$ was the mean crystal size of the *Sargassum* silver metal nanoparticle.

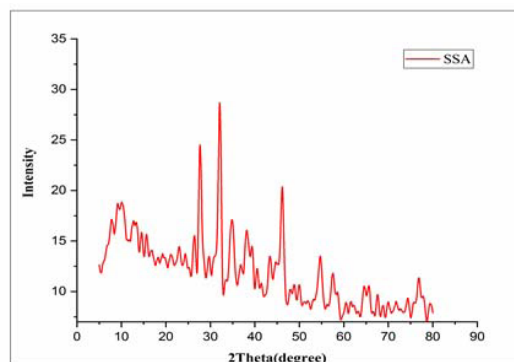


Fig. 2. XRD profile of SAA

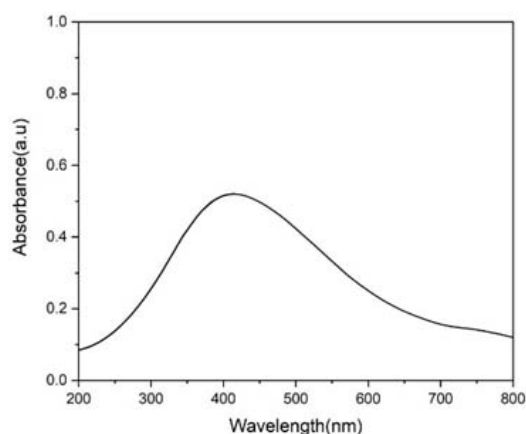


Fig. 3. Ultraviolet-Visible image of SAA-nanoparticle derived from *Sargassum*

Table 1: Ultraviolet Visible absorption spectra of SAA: Alpha cyclodextrin inclusion complex

Conc. of Alpha cyclodextrin	λ_{max}	Absorbance (A)
0	415	0.317
0.002	423	0.319
0.004	430	0.332
0.006	435	0.327
0.008	440	0.344
0.01	445	0.362

Table 2: Fluorescent Emission spectra of SAA: Alpha cyclodextrin inclusion complex

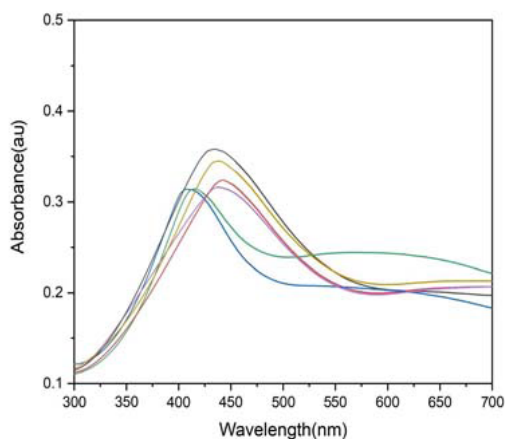
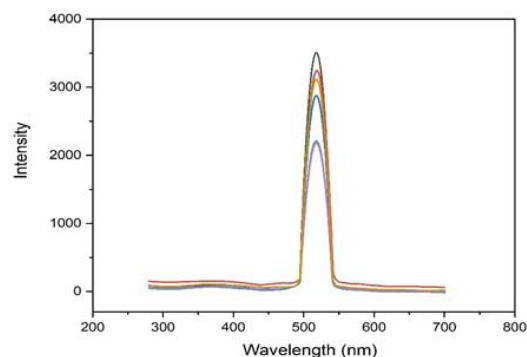
Concentration of a-CD	λ_{max}	Intensity (I)
0	510	2112.5
0.002	511.7	2200
0.004	513.3	2873
0.006	514.1	3075
0.008	515.5	3250
0.01	516	3500

Table 3: Proton Shifts for the Alpha-Cyclodextrin Inclusion Complex with SAA and Alpha-Cyclodextrin

	H1	H2	H3	H4	H5	H6
Alpha cyclodextrin	4.978	-	3.906	-	3.753	3.462
Inclusion complex of SAA with Alpha cyclodextrin	4.970	-	3.918	-	3.749	3.475
d	0.008	-	0.012	-	0.004	0.013

Table 4: Fluorescence Characteristics of Sargassum

Solvents	Visible light	Ultraviolet light	
		Longer wavelength	Shorter wavelength
C ₂ H ₅ OH	Green	Dark orange	Pale Green
H ₂ O	Dark brown	Fluorescent green	Dark Green
C ₆ H ₁₄	Brown	Orange	Green
CHCl ₃	Green	Yellow	Pale Green

**Fig. 4. Ultraviolet visible absorption spectra of SAA: Alpha cyclodextrin inclusion complex****Fig. 5. Fluorescent Patterns of emission from the SAA: Alpha cyclodextrin inclusion complex**

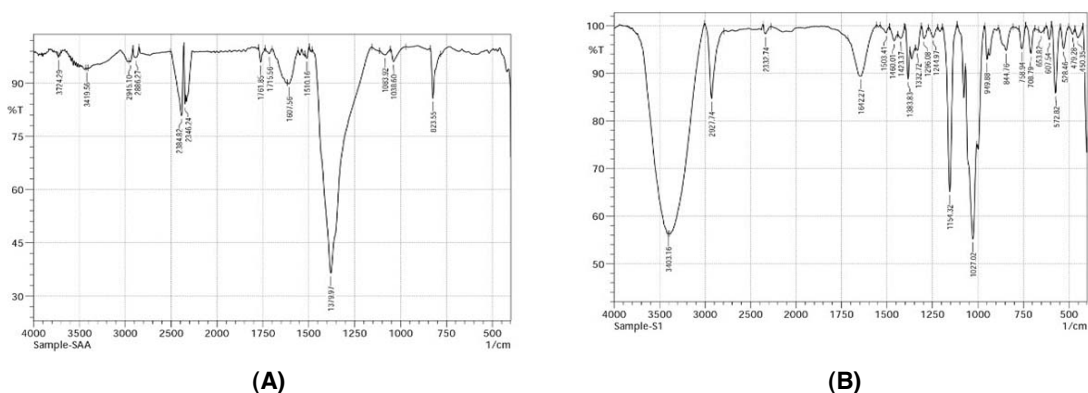


Fig. 6. IR spectra of the produced silver nanoparticle and its Alpha cyclodextrin inclusion complex (a) SAA and (b) S1

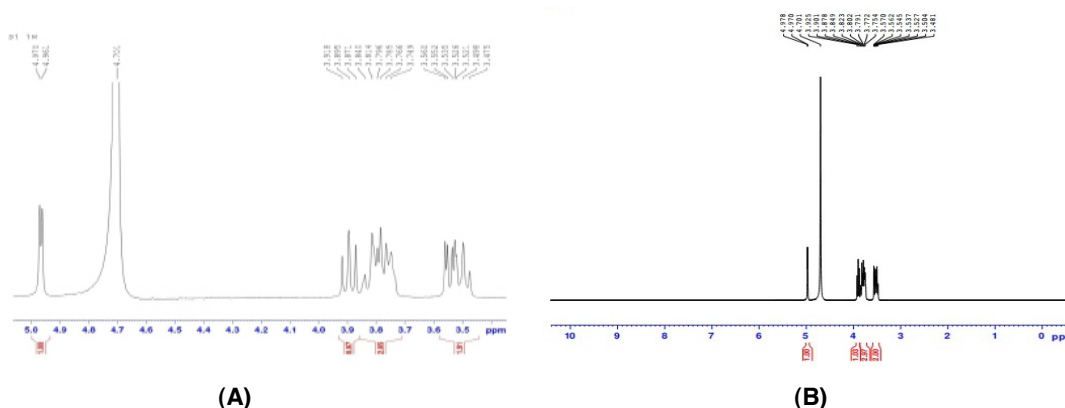


Fig. 7. Proton NMR of the produced Alpha cyclodextrin inclusion complex with SAA and Alpha cyclodextrin (a) S1 (b) Alpha cyclodextrin

Ultraviolet visible spectroscopy of SAA

The initial indication of silver nanoparticle (SAA) generation was observed through visual inspection of the reaction media. The introduction of algae extracts of *Sargassum* to the AgNO_3 solution resulted in a gradual colour change from pale yellow to brown, indicating the synthesis of silver nanoparticle SAA. The characteristic brown coloration resulted from the stimulation of surface plasmon resonance (SPR) in silver nanoparticles²⁵.

The synthesis of silver nanoparticle SAA was corroborated by the conspicuous absorption peak at 415 nm in the ultraviolet visible spectrum as shown in (Figure. 3), indicative of the surface plasmon resonance of silver nanoparticle.

Ultraviolet visible absorption study of SAA: Alpha cyclodextrin inclusion complex

The spectrum of absorption for SAA at various doses of Alpha cyclodextrin are portrayed in Figure 4 and Table 1. When a blue shift (hypochromic) or red shift (bathochromic) occurs, it is considered that the SAA and Alpha cyclodextrin connect. A wavelength shift from λ_{max} 415 nm to λ_{max} 445 nm occurs in this case as a consequence of a Bathochromic (red) shift that is noticeable as absorbance increases. The results shown above prove that an inclusion complex is formed when the generated silver nanoparticle (SAA) gets wrapped up in the Alpha cyclodextrin cavity.

Fluorescent emission study of SAA: Alpha cyclodextrin inclusion complex

The inclusion complex of SAA and Alpha cyclodextrin is discernible in the emission spectra (Figure 5 and Table 2). From I_{\max} 510 nm to I_{\max} 516 nm, the intensity in this case improves as the wavelength increases, which is the red (Bathochromic) shift. The foregoing finding confirms that the silver nanoparticle that is formed gets entrained in the Alpha cyclodextrin cavity and forms an inclusion complex.

IR Spectroscopy of SAA and S_1

The IR spectra of SAA and S_1 is depicted in figure 6(a) and 6(b), revealing the O-H stretching of hydroxyl bonds at 3419.56 cm^{-1} in SAA, which shifts to 3403.16 cm^{-1} in S_1 . The signal at 2945.10 cm^{-1} in nano SAA is displaced to 2927.74 cm^{-1} in sample

S_1 , indicating the existence of C-H bond stretching. The C=C stretching vibration of SAA at 1607.56 cm^{-1} is displaced to 1647.27 cm^{-1} . The C-O stretching at 1083.97 cm^{-1} is displaced to 1154.32 cm^{-1} . The aforementioned alterations in the FT-IR spectra of the synthesized silver nanoparticle (SAA) and its inclusion complex (S_1) demonstrate that the host Alpha cyclodextrin cavity accommodates the guest molecule SAA.

Proton NMR spectroscopy of S_1

The picture (Figure 7a & 7b) and Table 3 illustrate the proton NMR of the synthesized alpha-cyclodextrin inclusion complex (S_1) and the corresponding proton shifts, respectively. The up-field shift of H1 from 4.978 to 4.970 indicates a modification in its local electronic environment, presumably resulting from the incorporation of a

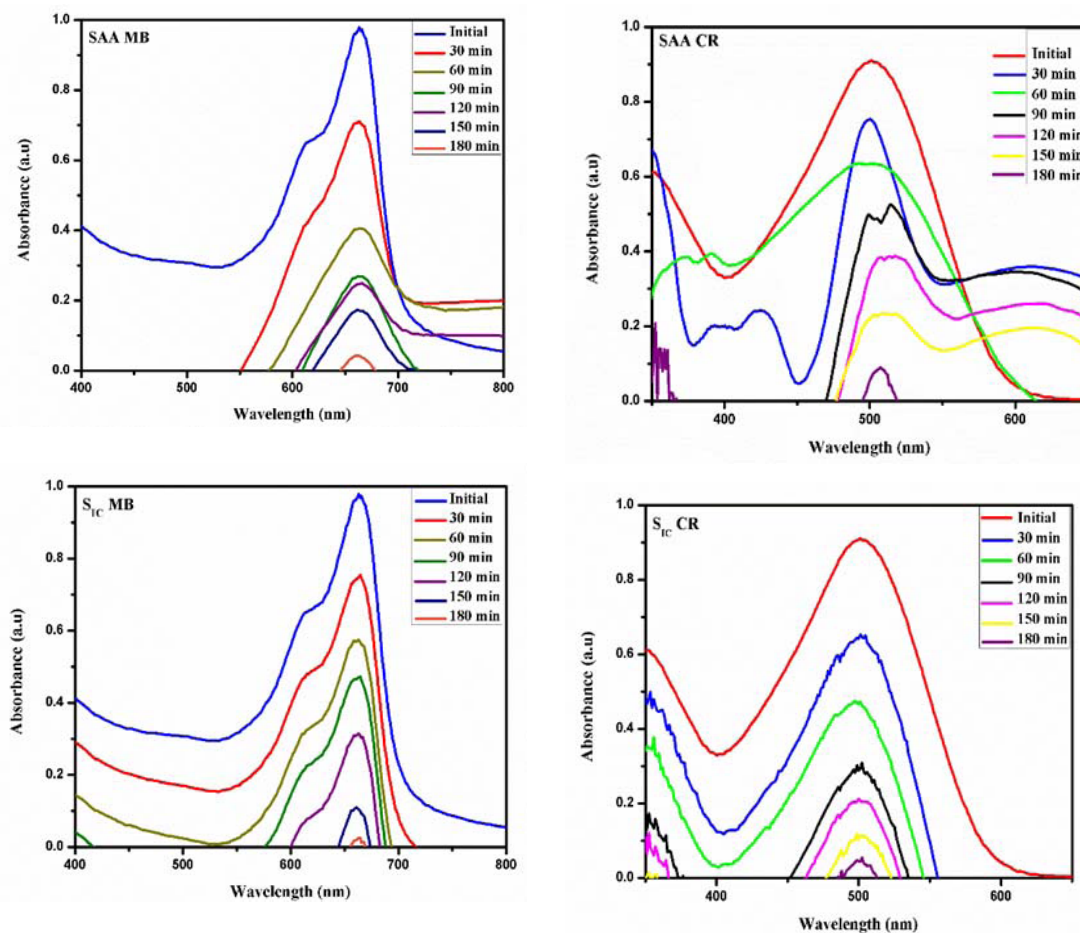


Fig. 8. Catalytic activity of Anionic pollutants (CR) and Cationic pollutants (MB) by SAA and S_1 (a) SAA MB (b) SAA CR (c) S_1 MB (d) S_1 CR

guest molecule into the α -cyclodextrin cavity. The downfield change of H3 from 3.906 to 3.918 ppm clearly indicates the creation of an inclusion complex, as it signifies direct interactions between H3 and the guest molecule within the α -cyclodextrin cavity. The upfield change of H5 from 3.753 to 3.749 ppm corroborates that the guest molecule is situated within the cyclodextrin cavity, modifying the shielding environment of H5. The downfield shift of H6 from 3.462 to 3.475 signifies that the H6 proton is located at the principal hydroxyl side of Alpha cyclodextrin, resulting in a broader cavity opening.

Fluorescence Examination

Table 4 shows the Fluorescence characteristics of *Sargassum*. The C_2H_5OH and $CHCl_3$ extracts of the sample appear green when exposed to visible light, whereas H_2O and C_6H_{14} extracts are brown and dark brown, respectively. Longer wavelengths of ultraviolet light render C_2H_5OH and C_6H_{14} extracts dark orange and orange, respectively. The H_2O extract of algae has a bright green hue, while $CHCl_3$ displays a yellow colouration. $CHCl_3$ and C_2H_5OH extracts of algae yield a pale green hue at shorter wavelengths, while H_2O and C_6H_{14} display dark green and green hues, respectively, at shorter wavelengths.

Catalytic activity of SAA and S_1

The synthesised nano-scale SAA and its inclusion complex S_1 extract, serving as catalysing agents, were employed to decompose the prevalent organic pollutants methylene blue (MB) and Congo red (CR). Figure 8 depicts the catalytic efficacy of SAA and S_1 with both pollutants. The catalytic data for SAA indicates that after a full 180 minutes of exposure to sun, the reduction of cationic pollutant (MB) and anionic pollutant (CR) was recorded at 91% and 87%, respectively, while S_1 achieved reductions of 97% for cationic pollutant (MB) and 91% for anionic pollutant (CR). A thorough analysis of the data suggests that a cationic pollutant

(MB), degrades more efficiently than an anionic pollutant (CR). This disparity may be attributed to the molecular structure of the cationic contaminant, which features an oxygen group and a positive charge.

CONCLUSION

The *Sargassum*, an alga, was retrieved from the Koodankulam coast for the purpose of this study. An eco-friendly method of preparing silver nanoparticle from the seaweed extract *Sargassum* is the focus of this investigation. Alpha cyclodextrin and a synthetic silver nanoparticle, SAA, were used to create an inclusion complex. Infrared, ultraviolet, 1H nuclear magnetic resonance, XRD, absorption and fluorescence emission spectroscopy, and other analytical tools verified that silver nanoparticle were successfully produced from the seaweed and its inclusion complex. The results also showed that the nanocatalyst SAA and its inclusion complex S_1 had excellent catalytic activity for breaking down cation and anion pollutants when exposed to sunlight. Cationic pollutants (MB) photodegrade at a faster rate than anionic pollutants (CR) in SAA and S_1 , according to a more thorough analysis of the data. The chosen contaminants are easily broken down by the synthetic nano and its inclusion complex. Researchers found that SAA, a nanomaterial made from algae, and its inclusion complex S_1 have environmental applications that could benefit the planet.

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Conflict of Interest

The authors declare that there are no conflicts of interest related to this study.

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